

H-atom parameters not refined

$$w = 1/(\sigma^2|F_o| + 0.002|F_o|^2)$$

$$(\Delta/\sigma)_{\max} = 0.008$$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Br(1)	0.56560 (2)	0.32825 (6)	1.04460 (5)	0.0544 (2)
O(12)	0.3795 (1)	0.1116 (4)	0.7692 (4)	0.058 (2)
O(11)	0.3529 (1)	0.3259 (4)	0.7087 (4)	0.058 (1)
C(11)	0.3834 (2)	0.2407 (5)	0.7512 (4)	0.042 (2)
C(12)	0.4273 (2)	0.3118 (5)	0.7801 (5)	0.046 (2)
C(13)	0.4621 (2)	0.2206 (6)	0.8562 (4)	0.048 (2)
N(1)	0.4527 (1)	0.1947 (4)	0.9697 (4)	0.042 (1)
C(14)	0.4557 (2)	0.3040 (5)	1.0434 (5)	0.051 (2)
C(15)	0.4454 (2)	0.2832 (7)	1.1471 (5)	0.062 (2)
C(16)	0.4317 (2)	0.1477 (7)	1.1717 (5)	0.067 (3)
C(17)	0.4290 (2)	0.0384 (7)	1.0953 (5)	0.072 (3)
C(18)	0.4405 (2)	0.0607 (6)	0.9940 (5)	0.054 (2)
O(21)	0.2814 (1)	0.2180 (4)	0.6513 (3)	0.054 (1)
O(22)	0.2574 (1)	0.4203 (4)	0.7114 (4)	0.067 (2)
C(21)	0.2518 (2)	0.3002 (5)	0.6700 (4)	0.041 (2)
C(22)	0.2069 (2)	0.2363 (6)	0.6324 (5)	0.050 (2)
C(23)	0.1732 (2)	0.3149 (6)	0.6808 (5)	0.051 (2)
N(2)	0.1776 (1)	0.2818 (4)	0.8029 (4)	0.042 (1)
C(24)	0.1640 (2)	0.1536 (5)	0.8303 (5)	0.054 (2)
C(25)	0.1663 (2)	0.1194 (7)	0.9425 (6)	0.067 (3)
C(26)	0.1835 (2)	0.2154 (8)	1.0259 (6)	0.072 (3)
C(27)	0.1982 (2)	0.3469 (8)	0.9970 (6)	0.071 (3)
C(28)	0.1947 (2)	0.3795 (6)	0.8835 (5)	0.058 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O(12)—C(11)	1.225 (6)	O(11)—C(11)	1.265 (6)
C(11)—C(12)	1.522 (7)	C(12)—C(13)	1.522 (7)
C(13)—N(1)	1.488 (7)	N(1)—C(14)	1.333 (7)
N(1)—C(18)	1.350 (7)	C(14)—C(15)	1.379 (9)
C(15)—C(16)	1.383 (9)	C(16)—C(17)	1.355 (9)
C(17)—C(18)	1.371 (10)	O(21)—C(21)	1.276 (6)
O(22)—C(21)	1.213 (6)	C(21)—C(22)	1.524 (7)
C(22)—C(23)	1.529 (8)	C(23)—N(2)	1.475 (7)
N(2)—C(24)	1.331 (7)	N(2)—C(28)	1.344 (7)
C(24)—C(25)	1.372 (9)	C(25)—C(26)	1.357 (9)
C(26)—C(27)	1.378 (10)	C(27)—C(28)	1.379 (10)
O(12)—C(11)—O(11)	125.3 (5)	O(12)—C(11)—C(12)	120.4 (4)
O(11)—C(11)—C(12)	114.2 (4)	C(11)—C(12)—C(13)	114.1 (4)
C(12)—C(13)—N(1)	111.3 (5)	C(13)—N(1)—C(14)	119.4 (4)
C(13)—N(1)—C(18)	118.6 (4)	C(14)—N(1)—C(18)	122.0 (5)
N(1)—C(14)—C(15)	120.1 (5)	C(14)—C(15)—C(16)	118.5 (6)
C(15)—C(16)—C(17)	120.1 (7)	C(16)—C(17)—C(18)	120.3 (6)
N(1)—C(18)—C(17)	118.9 (5)	O(21)—C(21)—O(22)	125.0 (5)
O(21)—C(21)—C(22)	114.1 (4)	O(22)—C(21)—C(22)	120.9 (5)
C(21)—C(22)—C(23)	114.1 (4)	C(22)—C(23)—N(2)	111.8 (4)
C(23)—N(2)—C(24)	118.2 (4)	C(23)—N(2)—C(28)	120.5 (5)
C(24)—N(2)—C(28)	121.4 (5)	N(2)—C(24)—C(25)	120.4 (5)
C(24)—C(25)—C(26)	119.7 (6)	C(25)—C(26)—C(27)	119.6 (7)
C(26)—C(27)—C(28)	119.4 (6)	N(2)—C(28)—C(27)	119.5 (6)

A crystal sealed in a Lindemann glass capillary was used to collect the intensity data. An Lp correction was applied. The structure was solved by direct methods and refined anisotropically. The H atoms of ppbet were generated geometrically (C—H = 0.96 \AA) and the acidic proton was located from a difference map and fixed. All H atoms were assigned isotropic displacement parameters and included in the structure-factor calculations. All computations were performed with the *SHELXTL/PC* program package (Sheldrick, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and data collection and processing parameters, and a packing diagram have been deposited with the IUCr (Reference: HL1048). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,4,6-Triphenylpyridine

J. ONDRÁČEK AND J. NOVOTNÝ

Department of Solid State Chemistry,
Institute of Chemical Technology, Technická 5,
166 28 Praha 6, Czech Republic

M. PETRŮ, P. LHOTÁK AND J. KUTHAN

Department of Organic Chemistry,
Institute of Chemical Technology, Technická 5,
166 28 Praha 6, Czech Republic

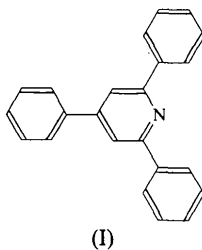
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Abstract

The crystal and molecular structures of 2,4,6-triphenylpyridine ($C_{23}H_{17}N$) obtained by X-ray analysis are compared with those obtained by quantum-chemical calculations based on the AM1 method. Both the theoretical and experimental results indicate that a disrotatory conformation is preferred. The AM1 results of the preferred conformation of a model compound (1,3,5-triphenylbenzene) are also reported.

Comment

2,4,6-Triphenylpyridine (1) was prepared by the published procedure (Kurfürst, Lhoták, Petrů & Kuthan, 1989). Crystals for X-ray analysis were obtained by slow crystallization from a 10:3:1 mixture of ethanol, acetone and chloroform.



The molecule with the atom numbering is shown in Fig. 1 and the crystal packing in Fig. 2. The results of the X-ray analysis are in good agreement with those obtained by quantum-chemical calculations based on the AM1 approach (Dewar, Zoebisch, Healy & Stewart, 1985; Petrů, Lhoták & Kuthan, 1994). The ground-state geometries were calculated for several conformers of 2,4,6-triphenylpyridine and although only small energy differences were observed between these conformers, there is evidence of a preference for a disrotatory arrangement. However, the dihedral angles in the solid state (32–35°) are smaller than those resulting from the quantum-chemical calculations (40°); this is probably due to the parameterization used in the AM1 semi-empirical method. Very similar values for the inter-annular angles and bond lengths were obtained by the theo-

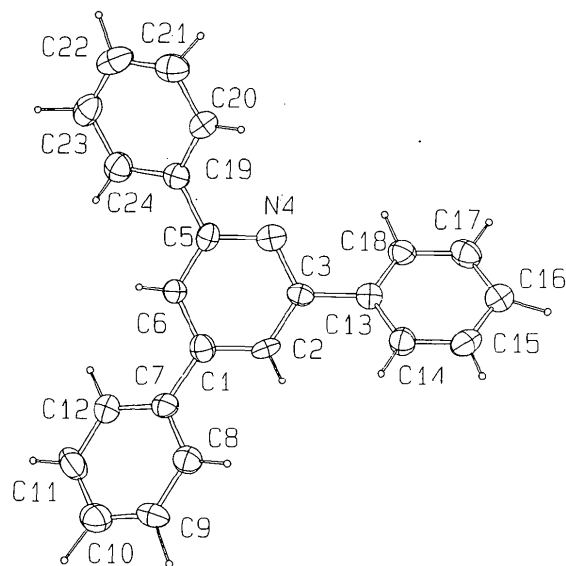


Fig. 1. View of (1) with atom numbering. Displacement ellipsoids are drawn at the 50% level.

retical approach and X-ray study (Table 3); these are proof of the relatively small degree of conjugation of this molecule in the ground state. This is comparable with the results described for biphenyl (Haefelinger & Redelmann, 1985) and 1,3,5-triphenylbenzene (Frag, 1954).

Studies of organic luminophores based on triaryl substituted pyridines were published recently (Kurfürst, Lhoták, Petrů & Kuthan, 1989; Lhoták, Kurfürst & Nádeník, 1992); the explanation and interpretation of their photophysical properties is a topic of great interest.

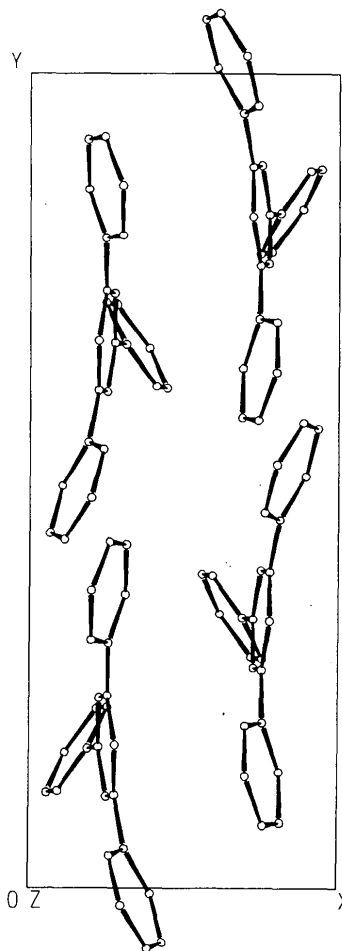


Fig. 2. Packing scheme.

Experimental*Crystal data*

C₂₃H₁₇N
M_r = 307.39
 Orthorhombic
*Pna*2₁
a = 7.487 (1) Å
b = 19.600 (2) Å
c = 11.287 (1) Å

Mo Kα radiation

λ = 0.71073 Å
 Cell parameters from 20 reflections
 θ = 15.95–17.77°
 μ = 0.0663 mm⁻¹
T = 295 K

$V = 1656.4 (3) \text{ \AA}^3$	Parallelepiped
$Z = 4$	$0.35 \times 0.20 \times 0.10 \text{ mm}$
$D_x = 1.233 \text{ Mg m}^{-3}$	White
Data collection	
Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.030$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25^\circ$
Absorption correction: none	$h = 0 \rightarrow 8$
7008 measured reflections	$k = -23 \rightarrow 23$
1533 independent reflections	$l = -13 \rightarrow 13$
744 observed reflections	2 standard reflections
$[I > 1.96\sigma(I)]$	frequency: 120 min
	intensity variation: -1.1%
Refinement	
Refinement on F^2	$w = 1/[\sigma^2(F_o) + 0.0009F_o^2]$
$R = 0.039$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$wR = 0.039$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
$S = 0.35$	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
744 reflections	Atomic scattering factors from <i>SHELX76</i>
285 parameters	(Sheldrick, 1976)
All H-atom parameters refined	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
C1	0.2468 (6)	0.7340 (3)	0.3003 (5)	0.0557 (18)
C2	0.2735 (7)	0.7315 (2)	0.1809 (5)	0.0489 (17)
C3	0.2755 (6)	0.6714 (2)	0.1194 (4)	0.0462 (15)
N4	0.2502 (5)	0.6107 (2)	0.1775 (5)	0.0588 (14)
C5	0.2218 (7)	0.6127 (3)	0.2973 (5)	0.0536 (18)
C6	0.2215 (8)	0.6738 (3)	0.3562 (5)	0.0495 (16)
C7	0.2439 (6)	0.7988 (2)	0.3669 (5)	0.0517 (15)
C8	0.1895 (7)	0.8590 (3)	0.3133 (6)	0.0628 (20)
C9	0.1864 (7)	0.9202 (2)	0.3749 (6)	0.0692 (20)
C10	0.2402 (8)	0.9235 (3)	0.4901 (6)	0.0835 (22)
C11	0.2994 (9)	0.8643 (4)	0.5446 (7)	0.0819 (23)
C12	0.3002 (8)	0.8028 (3)	0.4840 (5)	0.0740 (20)
C13	0.3111 (5)	0.6693 (2)	-0.0102 (5)	0.0488 (14)
C14	0.2458 (8)	0.7199 (3)	-0.0847 (5)	0.0625 (20)
C15	0.2811 (9)	0.7178 (3)	-0.2040 (6)	0.0718 (23)
C16	0.3843 (9)	0.6648 (3)	-0.2532 (7)	0.0731 (22)
C17	0.4430 (8)	0.6157 (3)	-0.1798 (5)	0.0696 (19)
C18	0.4078 (7)	0.6177 (2)	-0.0598 (5)	0.0608 (18)
C19	0.1896 (6)	0.5481 (2)	0.3593 (5)	0.0519 (17)
C20	0.1054 (8)	0.4937 (3)	0.3036 (6)	0.0600 (20)
C21	0.0651 (8)	0.4344 (3)	0.3643 (7)	0.0755 (23)
C22	0.1132 (8)	0.4268 (3)	0.4802 (8)	0.0807 (24)
C23	0.1995 (8)	0.4789 (3)	0.5367 (6)	0.0745 (21)
C24	0.2379 (7)	0.5391 (3)	0.4770 (6)	0.0662 (20)

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

C1—C2	1.363 (6)	C11—C12	1.386 (10)
C1—C6	1.351 (8)	C13—C14	1.389 (7)
C1—C7	1.476 (7)	C13—C18	1.364 (8)
C2—C3	1.367 (6)	C14—C15	1.373 (9)
C3—N4	1.372 (6)	C15—C16	1.409 (9)
C3—C13	1.488 (7)	C16—C17	1.344 (9)
N4—C5	1.369 (8)	C17—C18	1.380 (8)
C5—C6	1.370 (8)	C19—C20	1.389 (8)
C5—C19	1.467 (7)	C19—C24	1.388 (9)
C7—C8	1.387 (7)	C20—C21	1.383 (9)
C7—C12	1.390 (8)	C21—C22	1.365 (12)

C8—C9	1.387 (8)	C22—C23	1.366 (9)
C9—C10	1.363 (9)	C23—C24	1.389 (9)
C10—C11	1.386 (10)		
C6—C1—C7	120.7 (3)	C10—C11—C12	120.7 (7)
C2—C1—C7	122.5 (2)	C7—C12—C11	121.1 (6)
C2—C1—C6	116.8 (3)	C3—C13—C18	121.3 (4)
C1—C2—C3	122.3 (5)	C3—C13—C14	120.8 (4)
C2—C3—C13	121.7 (4)	C14—C13—C18	117.9 (5)
C2—C3—N4	120.2 (4)	C13—C14—C15	120.3 (5)
N4—C3—C13	118.1 (4)	C14—C15—C16	121.0 (6)
C3—N4—C5	118.0 (4)	C15—C16—C17	117.7 (6)
N4—C5—C19	118.2 (5)	C16—C17—C18	121.5 (6)
N4—C5—C6	120.3 (5)	C13—C18—C17	121.7 (5)
C6—C5—C19	121.5 (5)	C5—C19—C24	121.6 (4)
C1—C6—C5	122.5 (5)	C5—C19—C20	121.4 (5)
C1—C7—C8	121.9 (4)	C20—C19—C24	117.0 (5)
C1—C7—C12	120.9 (4)	C19—C20—C21	121.3 (6)
C8—C7—C12	117.1 (5)	C20—C21—C22	120.6 (6)
C7—C8—C9	121.5 (6)	C21—C22—C23	119.4 (7)
C8—C9—C10	121.0 (5)	C22—C23—C24	120.4 (6)
C9—C10—C11	118.6 (6)	C19—C24—C23	121.5 (5)

Table 3. Comparison of AM1 results with those of X-ray analyses

	2,4,6-Triphenylpyridine		1,3,5-Triphenylbenzene	
	AM1	X-ray	AM1	X-ray ^a
C2—C1—C7—C8	40.0	28.9 (7)	41.3	27.0
N4—C3—C13—C18	-39.9	-34.9 (6)	-41.4	-24.0
C6—C5—C19—C24	-40.3	-30.3 (8)	-42.0	-34.0
C1—C7	1.461	1.476 (7)	1.464	
C3—C13	1.475	1.488 (7)	1.462	
C5—C19	1.475	1.467 (7)	1.463	

Reference: (a) Farag (1954).

Data collection, data reduction and cell refinement: Enraf–Nonius CAD-4 and *SDP* software (Frenz, 1985). Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure by block-diagonal least squares in three blocks: *SHELX76* (Sheldrick, 1976). Program used to calculate molecular geometry: *PARST91* (Nardelli, 1991).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1076). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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