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4-(*p*-Bromophenyl)-3,5-bis(2-pyridyl)-4*H*-1,2,4-triazole

WEI CHEN,[a] Z. X. WANG,[b] F. F. JIAN,[b] Z. P. BAI[b] AND X. Z. YOU[b]

[a]*Department of Chemistry, University of Malaya, Lembah Pantai, 50603 Kuala Lumpur, Malaysia, and* [b]*Coordination Chemistry Institute and State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: chenwei@kimia.um.edu.my*

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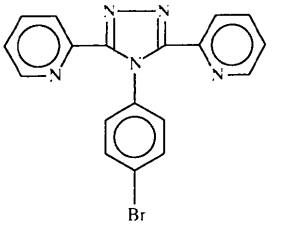
Abstract

The molecule of the title compound, $C_{18}H_{12}BrN_5$, is basically planar except that the phenyl and pyridyl rings are tilted in a propeller manner with respect to the central five-membered 1,2,4-triazole ring.

Comment

1,2,4-Triazoles are very useful ligands in coordination chemistry. A series of coordination compounds containing substituted 1,2,4-triazole ligands which can form five-membered chelate rings with metal ions have been studied (Bencini *et al.*, 1985, 1987; van Koningsbruggen *et al.*, 1995). We have recently synthesized the title compound, (I), which can act as a doubly-bidentate chelating ligand and its crystal structure is reported herein.



(I)

The dihedral angles that the two pyridyl rings make with the 1,2,4-triazole ring are 46.8 (2) and 28.1 (2)°, while that of the phenyl ring is 53.8 (2)°. The molecular planes perpendicular to the *b* axis are about 3.5 Å apart [N3···N5($\frac{3}{2}$ − *x*, $\frac{1}{2}$ + *y*, *z*) 3.510 (6) Å].

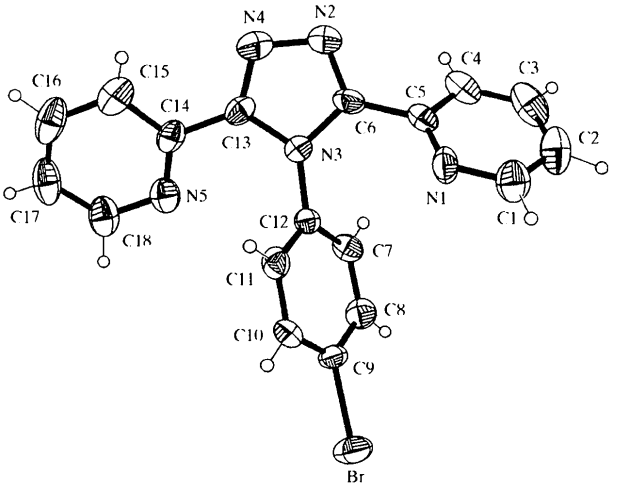


Fig. 1. *ORTEP*II (Johnson, 1976) plot at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Experimental

The title compound was obtained by the reaction of equivalent amounts of 4,4′-dibromophenylphosphazoanilide and *N*,*N*′-dipyridoylhydrazine in *o*-dichlorobenzene at 463–473 K for 3 h (Grimmel *et al.*, 1946; Klingsberg, 1958). Single crystals suitable for X-ray diffraction were recrystallized from ethanol.

Crystal data

$C_{18}H_{12}BrN_5$
M_r = 378.24

Mo $K\alpha$ radiation
λ = 0.71073 Å

Orthorhombic
Pbca
a = 18.4645 (9) Å
b = 8.0647 (6) Å
c = 21.9392 (9) Å
V = 3267.0 (3) Å^3
Z = 8
D_x = 1.538 Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
θ = 12–13°
μ = 2.525 mm^{-1}
T = 300 (2) K
Needle
0.36 × 0.18 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.435, T_{max} = 0.685
4569 measured reflections
3199 independent reflections

1645 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.97°
$h = 0 \rightarrow 22$
$k = -9 \rightarrow 7$
$l = -27 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: <1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.121
S = 0.925
3199 reflections
266 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.753 e Å^{-3}
$\Delta\rho_{min}$ = −0.823 e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0029 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *CAD*-4 *VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX/PC*. Data reduction: *NRCVAX* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1205). Services for accessing these data are described at the back of the journal.

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4-Benzoyl-4-(2-cyanoethyl)heptanedinitrile at 143 K

MICHAEL BOLTE AND MARIA SCHOLTYSSIK

Institut für Organische Chemie der Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany.
E-mail: bolte@chemie.uni-frankfurt.de

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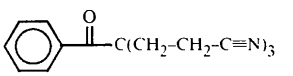
Abstract

The title compound, $C_{17}H_{17}N_3O$, crystallizes with two molecules in the asymmetric unit which differ significantly only in the conformation of one torsion angle. All bonds between the methylenic C atoms display antiperiplanar conformations, except one, which is synclinal. The carbonyl bond is twisted by 32° out of the plane of the phenyl ring and is synperiplanar to one of the methylenic C atoms.

Comment

The title compound, (I), crystallizes with two molecules (Figs. 1*a* and 1*b*) in the asymmetric unit.



(I)

Bond lengths and angles are in the usual ranges (*International Tables for Crystallography*, Vol. C); the mean values of the C_{sp^3}—C_{sp} and the C_{sp}—N bonds, for example, are 1.462 (4) and 1.141 (4) Å, respectively.

A least-squares fit of all non-H atoms, excluding C23 and N23 (r.m.s. deviation 0.103 Å), shows