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3,5-Bis(2-pyridyl)-4-*p*-chlorophenyl-4*H*-1,2,4-triazole

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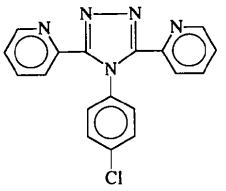
Abstract

The title compound, $C_{18}H_{12}ClN_5$, was prepared by the reaction of 4,4′-dichlorophenylphosphazoanilide and *N*,*N*′-dipyridoylhydrazine. The X-ray analysis revealed that the pyridyl groups, the substituted benzene ring and the 1,2,4-triazole ring do not share a common plane.

Comment

Bridging systems based on the 1,2,4-triazole ring are very interesting because of their similarity to the 1,3-imidazole bridging found in the copper–zinc protein superoxide dismutase (Feiters, 1990). We have synthesized a new compound, 3,5-bis(2-pyridyl)-4-*p*-chlorophenyl-1,2,4-triazole, (I), which can act as a doubly bidentate chelating ligand, and we report here its crystal structure analysis.



(I)

The title structure consists of two pyridine rings, one 1,2,4-triazole ring and one substituted benzene ring. All four rings do not share a common plane. The dihedral angle between the substituted-benzene and the 1,2,4-triazole rings is 88.37 (5)°, the two rings are almost perpendicular. The pyridyl groups are twisted with respect to the 1,2,4-triazole ring. The two N atoms, N(1) and N(5), are on opposite sides of the triazole ring and close to the benzene ring. The dihedral angles between the pyridyl groups [C(1)–C(5) and N(1), and C(8)–C(12) and N(5)] and the 1,2,4-triazole ring are 28.74 (4) and 26.95 (4)°, respectively, and those between the pyridyl groups and the substituted benzene ring are 91.30 (4) and 97.16 (4)°, respectively. The dihedral angle between the two pyridyl groups is 54.26 (5)°.

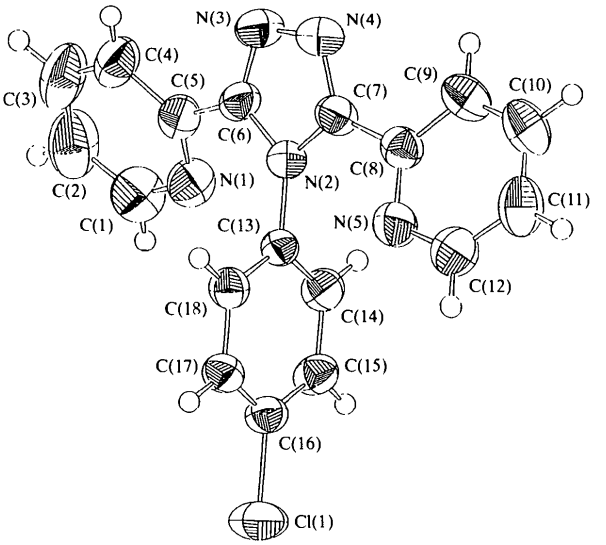


Fig. 1. The molecular structure of the title compound with the numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

Experimental

The title compound was obtained by the reaction of equal amounts of 4,4′-dichlorophenylphosphazoanilide and *N*,*N*′-dipyridoylhydrazine in *o*-dichlorobenzene for 3 h at 463–473 K (Grimmel *et al.*, 1946; Klingsberg, 1958). Recrystallization was from acetone.

Crystal data

$C_{18}H_{12}ClN_5$
M_r = 333.78
Triclinic
$P\overline{1}$
a = 8.949 (2) Å
b = 11.593 (2) Å
c = 8.6175 (9) Å
α = 97.89 (1)°
β = 110.715 (9)°
γ = 100.44 (2)°
V = 802.5 (3) Å^3
Z = 2
D_x = 1.381 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 14.4–14.9°
μ = 0.24 mm^{-1}
T = 296.2 K
Block
0.30 × 0.25 × 0.13 mm
White

Data collection

Rigaku AFC-7*S* diffractometer
ω–2θ scans
Absorption correction:
ψ scans (North *et al.*, 1968)
T_{min} = 0.949, T_{max} = 0.998
2228 reflections with $I > 1.5\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.49°
h = 0 → 11
k = −15 → 14
l = −11 → 10

3910 measured reflections
3906 independent reflections

3 standard reflections every 150 reflections
intensity decay: 2.43%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.052$
$S = 1.282$
2228 reflections
218 parameters
H atoms included but not refined
$w = 1/[\sigma^2(F_o) + 0.00063|F_o|^2]$
$(\Delta/\sigma)_{max} = 0.006$

$\Delta\rho_{max} = 0.150$ e Å^{-3}
$\Delta\rho_{min} = -0.180$ e Å^{-3}
Extinction correction: Zachariasen (1967) type 2 Gaussian isotropic
Extinction coefficient: 0.039 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Cl(1)—C(16)	1.737 (2)	N(3)—N(4)	1.378 (3)
N(2)—C(6)	1.372 (2)	N(3)—C(6)	1.311 (3)
N(2)—C(7)	1.371 (3)	N(4)—C(7)	1.316 (3)
N(2)—C(13)	1.444 (2)		
C(6)—N(2)—C(7)	104.7 (2)	N(3)—C(6)—C(5)	123.6 (2)
N(4)—N(3)—C(6)	107.9 (2)	N(2)—C(7)—N(4)	110.1 (2)
N(3)—N(4)—C(7)	107.3 (2)	N(2)—C(7)—C(8)	127.1 (2)
N(2)—C(6)—N(3)	109.9 (2)	N(4)—C(7)—C(8)	122.7 (2)
N(2)—C(6)—C(5)	126.4 (2)		

Data collection: *Rigaku/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1995*a*). Cell refinement: *Rigaku/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995*b*). Program(s) used to solve structure: *DIRDIF*92 (Beuskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *SHELXTL* (Sheldrick, 1994). Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1519). Services for accessing these data are described at the back of the journal.

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Anticancer Agents. III. 4,4′-(Hexane-1,6-diyl)bis(piperazine-2,6-dione)

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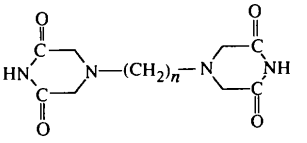
Abstract

In the crystals of $C_{14}H_{22}N_4O_4$, the molecule has a crystallographic inversion centre and the methylene chain is fully extended.

Comment

Compound (I) with $n = 2$ (ICRF-154) has been shown to have anticancer activity against a variety of cancer cells (Creighton, 1971; Cai *et al.*, 1989).



(I)

In a previous study, it was found that bifunctionalized compounds with six methylenes in the bridging group were usually more effective against cancer cells and less toxic than those with bridging groups of other lengths; examples include hexamethylenebis(acetamide) (Reuben *et al.*, 1976) and a suberic acid bis(methylamide) series (Breslow *et al.*, 1991). In order to discover new and more effective anticancer agents, we synthesized the title compound, which is an analogue of ICRF-154 with $n = 6$. Its activity against human erythroleukemia K562 cells was found to be greater than that of ICRF-154. This result suggests that six methylene groups may provide a suitable spacer distance between functional groups to match receptors or binding groups on the target.