C4—N9—C1′—O4′	−118.3 (3)	C2′—C3′—C4′—O4′	5.7 (3)
C8—N9—C1′—O4′	62.9 (4)	O3′—C3′—C4′—C5′	126.4 (3)
C4—N9—C1′—C2′	125.3 (3)	C2′—C3′—C4′—C5′	−114.0 (3)
C8—N9—C1′—C2′	−53.6 (4)	N9—C1′—O4′—C4′	−160.1 (2)
O4′—C1′—C2′—C3′	38.8 (3)	C2′—C1′—O4′—C4′	−36.3 (3)
N9—C1′—C2′—C3′	158.1 (2)	C5′—C4′—O4′—C1′	142.2 (3)
C1′—C2′—C3′—O3′	89.8 (3)	C3′—C4′—O4′—C1′	19.2 (3)
C1′—C2′—C3′—C4′	−26.5 (3)	O4′—C4′—C5′—O5′	178.3 (2)
O3′—C3′—C4′—O4′	−113.9 (3)	C3′—C4′—C5′—O5′	−62.9 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O100	0.86	2.04	3.044 (3)	144
N6—H6*B*···O3′^{i}	0.86	2.06	2.891 (3)	161
O3′—H3′*B*···N3^{ii}	0.82	2.01	2.773 (4)	154
O5′—H5′*A*···O2^{iii}	0.82	1.90	2.716 (3)	176
O100—H101···O5′^{iv}	0.86 (2)	2.05 (3)	2.866 (3)	159 (3)
O100—H102···O2^{v}	0.86 (2)	1.99 (2)	2.846 (3)	176 (4)

Symmetry codes: (i) $x, y - 1, z$; (ii) $\frac{1}{2} + x, \frac{5}{2} - y, 1 - z$; (iii) $x - \frac{1}{2}, \frac{5}{2} - y, 1 - z$; (iv) $\frac{1}{2} - x, 2 - y, \frac{1}{2} + z$; (v) $-x, y - \frac{1}{2}, \frac{3}{2} - z$.

With the absence of suitable anomalous scatterers within the molecule, the determination of the absolute configuration was not possible from our X-ray data. However, comparison with the configuration of the parent molecule indicates that the proposed conformation is correct.

Data collection: *XSCANS* (Siemens, 1996). Cell refinement: *XSCANS*. Data reduction: *SHELXTL* (Sheldrick, 1997*a*). Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*c*). Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1322). Services for accessing these data are described at the back of the journal.

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5,7-Diphenyl[1,2,4]triazolo[1,5-*a*]pyrimidine at 122 K

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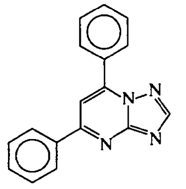
Abstract

The title compound, $C_{17}H_{12}N_4$, belongs to a group of N-bridgehead purine analogue ligands. The nine atoms comprising the triazolopyrimidine core have an r.m.s. deviation of 0.023 Å from the mean plane. The two phenyl groups attached to the core form interplanar angles of 27.38 (6) and 55.76 (3)° with this core plane.

Comment

Many derivatives of bicyclic [*a*]-heteroannulated pyrimidine are known as compounds of interesting coordinating properties for metal ions. Crystal structures have been reported for several metal complexes with ligands of this type (Biagini Cingi *et al.*, 1987, 1983; Haasnoot *et al.*, 1984). Therefore it is surprising that the structures of the free ligands have not been described in the literature (Cambridge Structural Database; Allen *et al.*, 1983). In order to enable the comparison between a free and a coordinated ligand, we undertook the structure determination of 5,7-diphenyl[1,2,4]triazolo[1,5-*a*]pyrimidine, subsequently referred to as dptp.



dptp

The *ORTEP*II (Johnson, 1996) drawing in Fig. 1 illustrates the overall geometry of dptp as found in the crystal. The nine atoms comprising the triazolopyrimidine core have an r.m.s. deviation of 0.023 Å from the least-squares plane defined by these atoms. This ring system is not strictly planar but slightly bent along the N8—C3*A* bond as indicated by the interplanar angle of 3.13 (9)° between the planes calculated for six atoms of the pyrimidine ring and the atoms of the triazole moiety. Fusion of the triazole with the pyrimidine ring decreases the aromaticity of the latter as shown by the elongation of C6—C5 [1.427 (2) Å] and N8—C3*A* [1.380 (2) Å] bonds compared to ~1.377 and ~1.330 Å, respectively, for non-conjugated, aryl-substituted pyrimidine compounds (Sawitzki & Schnering, 1979). The other bond lengths are almost unaffected. This is in line with previous observations for the pyridazine ring in which the aromaticity decreased when it was connected to a triazole ring system (Boulanger *et al.*, 1988).

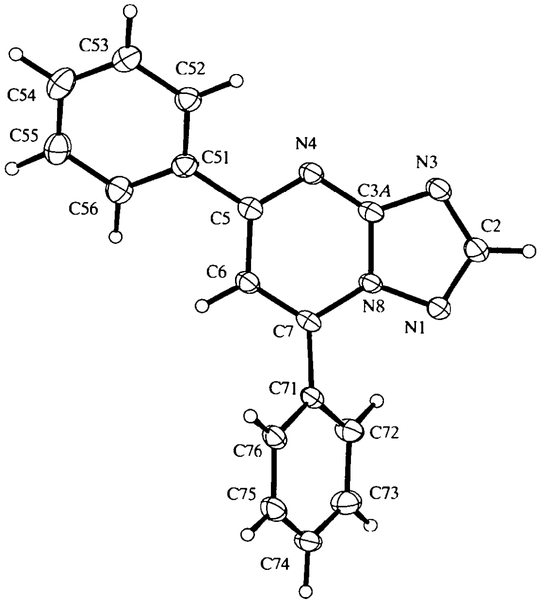


Fig. 1. Molecular structure of the title compound. Displacement ellipsoids enclose 50% probability, the H atoms are drawn as spheres with a fixed radius.

Coordination compounds of Co^{II} and Zn^{II} with dptp have been investigated and the structures are also known for complexes of the related ligand, where the phenyl rings are replaced with methyl. In all the known structures where these ligands are monodentate, the ligating N atom is N3. The bond lengths in dptp when it functions as a ligand towards Co^{II} and Zn^{II} are almost identical to those observed in the present structure (Szłyk *et al.*, 1997). The only significant changes of the geometry of dptp in these complexes are a relative shortening of the N1—C2 and N4—C3*A* bonds and a lengthening of the N3—C2 bond. Some significant differences between free and coordinated dptp are found with respect to the relative orientations of the two phenyl groups. The phenyl ring attached to C5 is almost coplanar to the central core when dptp is ligating to Zn^{II} and Co^{II} with interplanar angles 5.5 and 5.8°, respectively; in the free ligand this angle is 27.38 (6)°. The orientation of the other phenyl group is more similar in free and coordinated dptp. In the present structure, the angle between the least-squares planes calculated for the C71–C76 ring and the central nine-atom core is 55.76 (3)°, not too different from the equivalent angles in the Zn^{II} and Co^{II} complexes: 52.9 and 48.0°, respectively. This orientation of the C7 group effectively minimises the interactions between the *ortho*-H atoms and the central core. Therefore it was not expected that C—H···N hydrogen bonds could be observed in this structure. However, we find that the H atoms attached to C2, C75, and C76 are all engaged in intermolecular C—H···N hydrogen bonds with C···N distances in the range 3.283 (2)–3.459 (2) Å. The H atoms from C75 and C76 are interacting with N3(x, $1 + y$, z) and N1($\frac{1}{2} + x$, $\frac{1}{2} - y$, z), respectively, through linear hydrogen bonds, where H2 takes part in a bifurcated or three centre hydrogen bonds to N3 and N4 from another molecule related by a twofold screw axis parallel to the a axis. That N3 serves as an acceptor for two hydrogen bonds is in line with the observation that it is the ligating atom in the investigated coordination compounds.

To elucidate this aspect further, semiempirical and *ab initio* calculations were performed for the nine-atom central core. The ligand conformation was refined by force-field calculation using the *mm*2 force field and subsequently used for Zindo-S and *ab initio* (3–21G) computations by use of the program *HyperChem* (Hypercube, 1997). Both types of computations verified that the atomic charge and the electrostatic potential have their largest negative values associated with N3.

Experimental

Synthesis was carried out as described previously (Grodzicki *et al.*, 1996) and recrystallized from ethanol–acetone (1:1).

Crystal data

$C_{17}H_{12}N_4$	Cu $K\alpha$ radiation
$M_r = 272.31$	$\lambda = 1.54184$ Å
Orthorhombic	Cell parameters from 20 reflections
$P2_12_12_1$	$\theta = 39$–$47°$
$a = 7.8427$ (12) Å	$\mu = 0.653$ mm^{-1}
$b = 10.2644$ (13) Å	$T = 122$ (2) K
$c = 16.961$ (2) Å	Elongated prism
$V = 1365.4$ (3) Å^3	$0.50 \times 0.20 \times 0.15$ mm
$Z = 4$	Colourless
$D_x = 1.325$ Mg m^{-3}	
D_m not measured	

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2931 measured reflections
2630 independent reflections
2586 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\rm max} = 74.92°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 12$
$l = -21 \rightarrow 21$
6 standard reflections
frequency: 166.7 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.081$
$S = 1.020$
2630 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.2478P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.034$
$\Delta\rho_{\rm max} = 0.162$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.153$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0084 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C2	1.3325 (16)	N8—C3*A*	1.3796 (15)
N1—N8	1.3624 (15)	C6—C7	1.3698 (18)
N4—C5	1.3272 (16)	C6—C5	1.4273 (17)
N4—C3*A*	1.3432 (17)	N3—C3*A*	1.3346 (16)
N8—C7	1.3719 (15)	N3—C2	1.3523 (18)
C2—N1—N8	101.09 (10)	N3—C3*A*—N4	127.95 (11)
C5—N4—C3*A*	116.39 (11)	N3—C3*A*—N8	109.24 (11)
N1—N8—C7	127.37 (10)	N4—C3*A*—N8	122.78 (11)
N1—N8—C3*A*	110.18 (9)	N4—C5—C6	122.65 (11)
C7—N8—C3*A*	122.36 (10)	C6—C7—N8	115.18 (11)
C7—C6—C5	120.60 (11)	N1—C2—N3	117.03 (12)
C3*A*—N3—C2	102.45 (10)		

Only reflections which had $F^2 \geq 3\sigma(F^2)$ were used in the refinement. The refinement included both positional and isotropic displacement parameters for the H atoms; the C—H range = 0.96 (2)–1.02 (2) Å. The value of the Flack (1983) parameter [−0.2 (3); 996 Friedel-related reflections] was unreliable.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *DREADD* (Blessing, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OS1048). Services for accessing these data are described at the back of the journal.

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Dichloro[(*E*)-2-chloro-1-(2-hydroxyprop-2-yl)vinyl](4-methoxyphenyl)tellurium(IV)

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Abstract

The geometry around the Te$^{\rm IV}$ atom in the title compound, $C_{12}H_{15}Cl_3O_2Te$ or $[TeCl_2(C_5H_8ClO)(C_7H_7O)]$, is pseudo-trigonal bipyramidal, with the Cl atoms in the apical positions and the Te lone pair occupying the fifth position in the equatorial plane. By including the intermolecular secondary Te···Cl contact [3.485 (1) Å] *trans* to the Te—C$_{\rm vinyl}$ bond, the structure may be regarded as pseudo-octahedral. The molecules are associated *via* an O—H···Cl hydrogen bond to form centrosymmetric dimeric units, which in turn are arranged in an infinite zigzag chain along the *b* axis through the secondary Te···Cl bond. Distances and angles are: Te—Cl 2.4820 (8) and 2.5600 (8), Te—C

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