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6a,7-Dihydro-2,6-dimethyl-7-phenyl-6*H*-chromeno[4,3-*d*]-1,2,4-triazolo[1,5-*a*]-pyrimidin-6a-ol†

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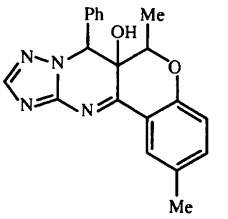
Abstract

In the title compound, $C_{20}H_{18}N_4O_2$, the dihydropyrimidine and dihydropyran rings adopt a distorted sofa conformation. The phenyl substituent at C7 and the hydroxy group at C6a adopt axial positions, whereas the methyl group at C6 is equatorial.

Comment

Derivatives of 1,2,4-triazolo[1,5-*a*]pyrimidine possess high biological activity, for example, as cardiovascular agents (Tsuda *et al.*, 1986). The chemical and physiological characteristics of these compounds are directly related to their conformations. However, relevant data on the structures of dihydro-1,2,4-triazolo[1,5-*a*]pyrimidines condensed with pyran rings, (I), or other heterocycles have not previously been available.



(I)

Torsion angles N7a—C10a—N11—C11a and C1—C1a—C11a—N11 are 15.4 (4) and 9.4 (4)°, respectively. This indicates some loss of planarity of the conjugated system of the molecule, although the C1a—C11a, N11—C11a and C10a—N11 bond lengths [1.453 (4), 1.294 (3) and 1.381 (3) Å, respectively] are evidence of the conjugation between the benzene ring, azomethine group and 1,2,4-triazole ring (Fig. 1).

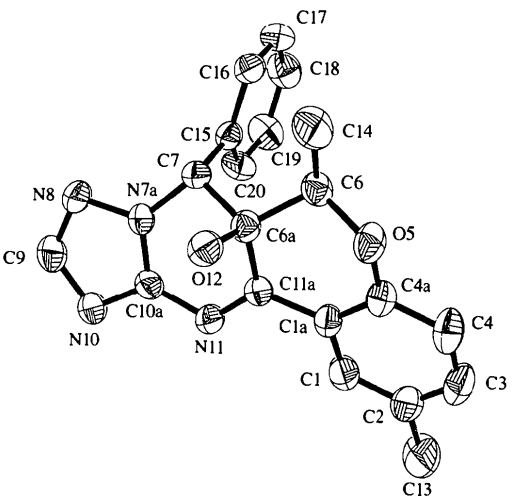


Fig. 1. A view of (I) with displacement ellipsoids drawn at the 50% probability level. The H atoms are omitted for clarity.

The conformations of both the pyrimidine and pyran rings can be described as distorted sofas. The puckering coordinates (Zefirov, Palyulin & Dashevskaya, 1990) are $S = 0.63$, $\theta = 50.9$ and $\varphi = 18.3$ for the pyrimidine, and $S = 0.72$, $\theta = 44.7$ and $\varphi = 10.2$ for the pyran moiety, where S is the puckering degree, and θ and φ are polar angles which describe the ring conformation type (values of these angles for ideal conformations are: sofa $\theta = 45$, $\varphi = 0$; half chair $\theta = 45$, $\varphi = 30$). Comparison of the title compound with *cis*-3-bromo-2,3-dihydro-2-phenyl-4*H*-1-benzopyran-4-one and (2*R*,3*R*)-3,5,7-trihydroxy-2-(2*R**,3*R**)-3-(4-hydroxy-3-methoxyphenyl)-2-hydroxymethyl-(1,4-benzodioxan-6-yl)-4-chromanone

† Alternative name: 6a,7-dihydro-2,6-dimethyl-7-phenyl-6*H*-[1,2,4]-triazolo[2′,3′:1,2]pyrimidino[5,4-*c*]chromen-6a-ol.

shows that the dihydropyranone ring adopts a distorted half-chair conformation and annelation of the ring concerned leads to the change in its conformation (Cantrell & Hockstein, 1982; Lotter & Wagner, 1983). An alkyl substituent in the second position has an equatorial orientation (Geddes, Sheldrick & Akrigg, 1980; Chambers & Marfat, 1994). Comparison of (I) with 5,7-diphenyl-6-oxy-6,7-dihydropyrazolo[1,5-*a*]pyrimidine, the puckering coordinates for which are $S = 0.61$, $\theta = 46.2$ and $\varphi = 28.9$, indicates that annelation of the dihydropyrimidine ring results in a decrease in its twisting (Desenko *et al.*, 1993).

The crystal is composed of a racemic mixture of the (6*R*,6a*S*,7*R*) and (6*S*,6a*R*,7*S*) isomers. The phenyl and hydroxyl substituents on C7 and C6a are both in axial positions [the C10a—N7a—C7—C15 and O12—C6a—C11a—C1a torsion angles are 95.7 (3) and −104.2 (3)°, respectively]. The methyl group is equatorial; the C4a—O5—C6—C14 and C11a—C6a—C6—C14 torsion angles are −178.7 (2) and −168.1 (2)°, respectively.

In the crystal, the molecules of (I) form chains due to intermolecular hydrogen bonds O12—H12O···N10^{i}, characterized by H···N 1.92 (4) Å and O—H···N 174 (4)° [symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$].

Experimental

Compound (I) was obtained from 2,9-dimethyl-3-phenyl-3,7-dihydro-2*H*-1,2,4-triazolo[1′,5′-*a*′]pyrimido[4,5-*d*]benzo-[*b*]pyran by dissolving the latter in DMSO and allowing slow aerial oxidation to occur over a period of two weeks. Spectral characteristics of (I) are as follows: UV spectrum (isopropanole, Specord M40 spectrometer): *Λ*max (*ε*/1000): 313 (20.7), 376 (20.8); IR spectrum (KBr tablets, Specord 75 IR spectrometer): *ν*(C═N) 1618 cm^{-1}; PMR spectrum (DMSO-d_6, Varian VXR-300 spectrometer): *δ* (p.p.m.): 6.8–7.9 (8H, *m*), 7.94 (1H, *s*, 6-H), 7.18 (1H, *s*, OH), 5.59 (1H, *s*, 3-H), 3.68 (1H, *q*, *J* = 6.4 Hz, 2-H), 2.32 (3H, *s*, 13-CH_3), 1.41 (3H, *d*, *J* = 6.4 Hz, 14-CH_3). Single crystals were grown by diffusion of benzene into a solution of (I) in DMSO.

Crystal data

$C_{20}H_{18}N_4O_2$
$M_r = 346.38$
Monoclinic
$P2_1/n$
$a = 10.770\,(2)$ Å
$b = 10.045\,(2)$ Å
$c = 16.499\,(4)$ Å
$\beta = 105.07\,(2)°$
$V = 1723.6\,(6)$ Å^3
$Z = 4$
$D_x = 1.335$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 11–12°$
$\mu = 0.089$ mm^{-1}
$T = 293\,(2)$ K
Plate
0.40 × 0.20 × 0.10 mm
Yellow

Data collection

Siemens *P3/PC* diffractometer
θ–2θ scans
Absorption correction: none
2618 measured reflections
2467 independent reflections
1685 reflections with $I > 2\sigma(I)$

$R_{int} = 0.023$
$\theta_{max} = 25.04°$
$h = 0 \rightarrow 12$
$k = -11 \rightarrow 0$
$l = -19 \rightarrow 18$
2 standard reflections
every 98 reflections
intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.201$
$S = 1.152$
2365 reflections
241 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.7176P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.037$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Refinement on F^2 was carried out on all reflections except for 66 with very negative F^2 or having $|(F_o - F_c)| > 4\sigma(F_o)$ and therefore flagged by the user for potential systematic errors. Standard uncertainties on C—C bond distances are 0.004 Å.

Data collection: *P3/PC Diffractometer Program* (Siemens, 1989). Cell refinement: *P3/PC Diffractometer Program*. Data reduction: *SHELXTL* (Sheldrick, 1994) . Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1134). Services for accessing these data are described at the back of the journal.

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