

5-(2-Chlorobenzyl)-2-(2-hydroxyethyl)-6-methylpyridazin-3(2H)-one

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Key indicators

Single-crystal X-ray study

$T = 173\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.036

wR factor = 0.078

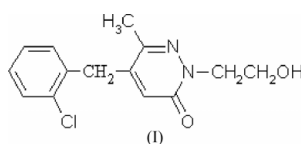
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{14}\text{H}_{15}\text{ClN}_2\text{O}_2$, is of pharmacological interest. It contains an oxypyridazine heterocycle, carrying a methyl group in the position *para* to the oxo group, a hydroxyethyl group at one of the N atoms and a 2-chlorobenzyl residue in the position *meta* to the oxo group. The dihedral angle between the two rings is $85.71(5)^\circ$. In the crystal structure, the molecules form hydrogen-bonded centrosymmetric dimers.

Comment

Many pyridazine compounds have been reported to be anticonvulsive agents (Rubat *et al.*, 1990; Foussard-Blanc & Lacroix, 1991). Furthermore, Gehrlein *et al.* (1981) have described the antihypertensive effects of novel hydroxyethylpyridazine compounds. In continuation of this line of investigation, we have synthesized compound (I); it will be subjected to further pharmacological investigations, especially tests of its anticancer activity. Compound (I) is stable at room temperature, and its structure has been determined by IR, MS and NMR (^1H and ^{13}C) spectroscopy. Since these techniques did not provide sufficient information about the conformation of the reaction product, we have carried out an X-ray structure analysis. The bond lengths and angles of (I) are normal (Table 1). The dihedral angle between the two rings is $85.71(5)^\circ$. In the crystal structure, molecules of (I) form hydrogen-bonded centrosymmetric dimers (Table 2).



The structure of a very similar compound, (II), in which the N atom carries an H atom instead of a 2-hydroxyethyl group, has been determined by Moreau *et al.* (1995). However, the conformations of the two compounds are completely different, as can be seen by the torsion angle $\text{C}12-\text{C}11-\text{C}7-\text{C}1$, which is $84.6(2)^\circ$ in the title compound and 149.6° in (II). The reason for this difference may be due to the different packing patterns. Whereas the title compound crystallizes as centrosymmetric hydrogen-bonded dimers, compound (II) contains hydrogen-bonded zigzag chains.

Experimental

A mixture of levulinic acid and *o*-chlorobenzaldehyde, containing HCl at room temperature, gave phenylmethyllevulinic acid, which was treated with hydrazine in boiling ethanol to give 5-(*o*-chlorobenzylidene)-6-methylpyridazin-3-one. Addition of 2-bromoethanol,

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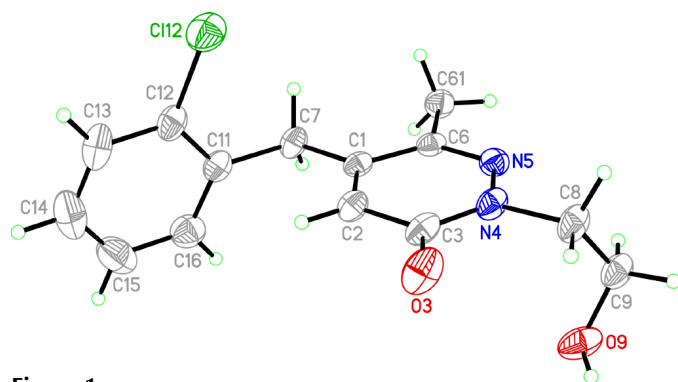


Figure 1
Perspective view of (I), with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

in the presence of potassium carbonate in boiling tetrahydrofuran, yielded 5-(2-chlorobenzyl)-2-(2-hydroxyethyl)-6-methylpyridazin-3(2H)-one. Single crystals were obtained by recrystallization from ethanol.

Crystal data

$C_{14}H_{15}ClN_2O_2$
 $M_r = 278.73$
 Monoclinic, $P2_1/c$
 $a = 10.6426$ (13) Å
 $b = 7.3564$ (8) Å
 $c = 18.467$ (2) Å
 $\beta = 101.409$ (9)°
 $V = 1417.2$ (3) Å³
 $Z = 4$

$D_x = 1.306$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5126 reflections
 $\theta = 3.5$ – 25.1 °
 $\mu = 0.27$ mm⁻¹
 $T = 173$ (2) K
 Block, colourless
 $0.29 \times 0.15 \times 0.14$ mm

Data collection

Stoe IPDS-II two-circle diffractometer
 ω scans
 Absorption correction: multi-scan (MULABS; Spek, 1990; Blessing, 1995)
 $T_{\min} = 0.936$, $T_{\max} = 0.970$
 9173 measured reflections

2526 independent reflections
 1680 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.063$
 $\theta_{\text{max}} = 25.2$ °
 $h = -12 \rightarrow 12$
 $k = -8 \rightarrow 8$
 $l = -20 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.078$
 $S = 0.88$
 2526 reflections
 177 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0382P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C3—N4	1.370 (3)	N5—C6	1.304 (2)
N4—N5	1.367 (2)	C9—O9	1.421 (3)
N4—C8	1.472 (2)	C12—C112	1.748 (2)
N5—N4—C3	125.06 (15)	C3—N4—C8	122.36 (16)
N5—N4—C8	112.57 (16)	C6—N5—N4	118.51 (16)

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O9-H9 \cdots O3^i$	0.81 (3)	2.00 (3)	2.751 (2)	154 (3)

Symmetry code: (i) $1 - x, 2 - y, 1 - z$.

All H atoms were located in a difference Fourier synthesis and were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$], using a riding model with C—H(aromatic) = 0.95 Å, C—H(methyl) = 0.98 Å and C—H(methylene) = 0.99 Å. The methyl group was allowed to rotate but not to tip. The hydroxy H atom was refined isotropically without constraints.

Data collection: X-AREA (Stoe & Cie, 2001); cell refinement: X-AREA; data reduction: X-AREA; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP in SHELXTL-Plus (Sheldrick, 1991); software used to prepare material for publication: SHELXL97.

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