2-(4-Chlorophenyl)-4-(2-hydroxyethyl)-5-methyl-2,4-dihydro-3H-1,2,4-triazol-3-one

S. Thamotharan, V. Parthasarathi, Vinay Sunagar, Bharati Badami and Anthony Linden
In the title compound, \( \text{C}_11\text{H}_{12}\text{ClN}_3\text{O}_2 \), the dihedral angle between the phenyl and triazole rings is 30.63 (9)°. An intermolecular O—H···O hydrogen bond is formed between the hydroxy group and the carbonyl group of the triazole moiety of a neighbouring molecule. This interaction links the molecules into chains, which run parallel to the c axis. C—H···O intermolecular hydrogen bonds are also observed.

Comment

Extensive studies have been carried out on substituted 1,2,4-triazole derivatives (Cornelissen et al., 1992; Kunkeler et al., 1996; Chinnakali et al., 1999; Fun et al., 1999; Kumaran et al., 1999). Research findings indicate that the 1,2,4-triazole moiety is associated with diverse pharmacological activities, such as analgesic, anti-asthmatic, diuretic, antifungal, antibacterial, pesticidal and anti-inflammatory activities (Bennur et al., 1976; Heubach et al., 1980; Sharma & Babel, 1982; Mohamed et al., 1993). In view of this, the crystal structure determination of the title compound, (I), has been carried out in order to elucidate the stereochemistry and the molecular conformation.

The bond lengths and angles in (I) are comparable with those reported for related structures (Chen et al., 1998; Wang et al., 1998). Unweighted least-squares planes calculations show that the phenyl group is oriented at an angle of 30.63 (9)° with respect to the plane of the triazole ring. The hydroxyethyl group projects roughly perpendicular to the triazole ring [\( \text{C}3—\text{N}4—\text{C}12—\text{C}13 = 80.7 (2)° \)]. The exocyclic angle \( \text{N}2—\text{C}3—\text{O}3 \) [128.58 (17)°] is significantly larger than the normal value of 120°, and this may be due to the short contact between atoms H11 of the phenyl ring and O3 (2.54 Å). The hydroxy group forms an intermolecular hydrogen bond with the carbonyl O atom of an adjacent molecule. This interaction links the molecules into chains, which run parallel to the c axis and have a graph-set motif of \( \text{C}(7) \) (Bernstein et al., 1995). Several C—H···O intermolecular hydrogen bonds are also observed in (I) (Table 2).

Experimental

The title compound was prepared by heating 3-(4-chlorophenyl)-5-methyl-2-oxo-5,3,4-oxadiazole with ethanolamine. The solid obtained, (I), was crystallized from ethanol (m.p. 388–403 K).
Crystal data

C_{12}H_{12}ClN_{3}O_{2}

M_r = 253.69

Monoclinic, P2_{1}/c

a = 12.6968 (4) Å

b = 11.1886 (3) Å

α = 7.9872 (2) Å

β = 95.446 (1)°

V = 1129.54 (5) Å³

Z = 4

D_{c} = 1.492 Mg m^{-3}

Mo Kα radiation

Cell parameters from 13 330 reflections

θ = 2.0-27.5°

μ = 0.33 mm^{-1}

T = 160 (2) K

Table, pale yellow

0.28 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer

φ and ω scans with φ offsets

Absorption correction: multi-scan

(Blessing, 1995)

θ_m = 0.895, θ_max = 0.24 e Å^{-3}

2597 independent reflections

23 311 measured reflections

2177 reflections with I > 2σ(I)

R_{int} = 0.081

θ_{max} = 27.5°

k = -16 → 16

l = -10 → 10

Refinement

Refinement on F^2

H atoms treated by a mixture of independent and constrained refinement

wR(F^2) = 0.043

w = 1/[σ^2(F^2) + (0.0683P)^2]

where P = (F^2 + 2F_c^2)/3

Δρ_{max} = 0.24 e Å^{-3}

Δρ_{min} = -0.37 e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

<table>
<thead>
<tr>
<th>Symmetry code</th>
<th>N1-C5</th>
<th>N4-C5</th>
<th>N1-C6</th>
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<tr>
<td>(i)</td>
<td>1.296 (2)</td>
<td>1.377 (2)</td>
<td>1.377 (2)</td>
</tr>
<tr>
<td>(ii)</td>
<td>1.401 (2)</td>
<td>1.383 (2)</td>
<td>1.383 (2)</td>
</tr>
<tr>
<td>(iii)</td>
<td>1.370 (2)</td>
<td>1.377 (2)</td>
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</table>

Table 2

Hydrogen-bonding geometry (Å, °).

<table>
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<tr>
<th>D—H—A</th>
<th>D—H</th>
<th>H—A</th>
<th>D—A</th>
<th>D—H—A</th>
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<tbody>
<tr>
<td>O14···H14—O3</td>
<td>0.86 (3)</td>
<td>1.91 (3)</td>
<td>2.7498 (19)</td>
<td>163 (3)</td>
</tr>
<tr>
<td>C12—H12···O14</td>
<td>0.98 (2)</td>
<td>2.57 (2)</td>
<td>3.528 (2)</td>
<td>163</td>
</tr>
<tr>
<td>C12—H12···O14</td>
<td>0.98 (2)</td>
<td>2.57 (2)</td>
<td>3.544 (2)</td>
<td>163</td>
</tr>
<tr>
<td>C15—H15···O14</td>
<td>0.98 (2)</td>
<td>2.94 (2)</td>
<td>3.463 (2)</td>
<td>174</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) x, y, z; (ii) x, y, z; (iii) x, y, z; (iv) x, y, z.

The position of the hydroxy H atom was determined from a difference Fourier map and refined freely along with its isotropic displacement parameter. The methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å), with U_{eq}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the parent C—C bond. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.95–0.99 Å) and constrained to ride on their parent atoms with U_{eq}(H) = 1.2U_{eq}(C).

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO—SMN (Otwinowski & Minor, 1997); data reduction: SCALEPACK (Otwinowski & Minor, 1997); program(s) used to solve structure: SIR92 (Altomare et al., 1994); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Version 1.07); Furrugia, 1997); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2002).

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References


