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3-(Ethoxycarbonyldihydroxymethyl)-2-oxo-1,2-dihydroquinoxaline

S. Ferfra,[a] N. Ahabchane,[a] E. M. Essassi[a] and M. Pierrot[b]*

[a]Laboratoire de Chimie Organique Hétérocyclique, Faculté des Sciences, Université MohamedV, Rabat, Morocco, and [b]LBS-UMR 6517, Centre Scientifique Saint-Jérôme, 13397 Marseille CEDEX 20, France
Correspondence e-mail: marcel.pierrot@lbs.u-3mrs.fr

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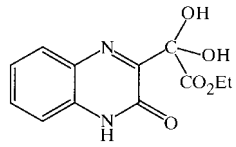
The crystal structure of the title compound, ethyl 2,2-dihydroxy-2-(3-oxo-3,4-dihydroquinoxalin-2-yl)acetate, $C_{12}H_{12}N_2O_5$, indicates a short intramolecular contact between the N1 atom and a hydroxyl group [2.772 (4) Å]. Two intermolecular hydrogen bonds participate in the molecular packing.

Comment

Interest in quinoxaline derivatives has increased greatly during recent years due to their different applications in various areas. Some derivatives are used as colorimetric agents (Campaigne & McLaughlin, 1983), antibacterial agents (Boutte & Lecolier, 1976) and colouring matter. Other derivatives possess various biological activities (Dalgaard *et al.*, 1994; De Clercq, 1998; Habib & El Hawash, 1997; Johnson *et al.*, 1968; Li *et al.*, 1997). In a previous paper (Benchimdi *et al.*, 1993), we reported the reaction of 3-ethoxycarbonylmethyl-2-oxo-1,2-dihydroquinoxaline, (I), with bromide which allowed 3-ethoxycarbonylbromomethyl-2-oxo-1,2-dihydroquinoxaline, (II), to be obtained. The latter proved to be a useful key intermediate, leading to novel quinoxalines functionalized on a carbon of the ester group. Further reaction of (II) in dimethylformamide with sodium azide in water at low temperature provided 3-ethoxycarbonylazidomethyl-2-oxo-1,2-dihydroquinoxaline, (III), and as a result of the substitution of bromide, the unexpected title compound, 3-(ethoxycarbonyldihydroxymethyl)-2-oxo-1,2-dihydroquinoxaline, (IV), the structure of which has been determined by X-ray diffraction. The quinoxaline bicycle N1/C2/C3/N4/C5/C6/C7/C8/C9/C10 is planar (r.m.s. deviation 0.006 Å). The O5 and C11 atoms are located in this plane [at −0.0168 (4) and −0.0962 (5) Å, respectively] which is at an angle of 75.8 (4)° to the ethoxycarbonyl group (plane O4/C11/O3/C12/C13: r.m.s. deviation 0.014 Å). In the crystal structure, the molecules are linked by two intermolecular hydrogen bonds between N1 and O5, and between O21 and N4. Atom N4^{ii} is only 2.772 (4) Å from O22 but an intramolecular hydrogen bond does not seem possible, the N···H22—O22 angle being 102° and the N4···H22 distance of 2.51 Å being too large [symmetry code: (ii) $2-x, \frac{1}{2}+y, \frac{1}{2}-z$].



(IV)

Experimental

To a solution (0.003 mol) of sodium nitrite in dimethylformamide (15 ml) and water (15 ml) at 273 K, a solution (0.003 mol) of (II) in dimethylformamide (15 ml) was added dropwise under vigorous stirring. The mixture was stirred for 12 h, then the solvent was removed under vacuum and the resulting material was chromatographed on a silica-gel column using a 10:90 mixture of ethyl acetate and hexane, to give compounds (III) and (IV).

Crystal data

$C_{12}H_{12}N_2O_5$	D_x = 1.44 Mg m^{-3}
M_r = 264.24	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2522 reflections
a = 7.6475 (4) Å	θ = 1.0–25.6°
b = 9.2255 (3) Å	μ = 0.114 mm^{-1}
c = 17.521 (5) Å	T = 298 K
β = 99.122 (2)°	Prism, yellow–red
V = 1220.5 (4) Å^3	0.25 × 0.20 × 0.15 mm
Z = 4	

Data collection

KappaCCD diffractometer	R_{int} = 0.035
φ scans	θ_{max} = 25.6°
2363 measured reflections	$h = 0 \rightarrow 9$
2363 independent reflections	$k = 0 \rightarrow 10$
1919 reflections with $I > 3\sigma(I)$	$l = -21 \rightarrow 21$

Refinement

R = 0.049	H-atom parameters not refined
wR = 0.054	$w = 1/[\sigma^2(F_o^2) + 0.03F_o^2]$
S = 1.472	$(\Delta/\sigma)_{max}$ = 0.048
1919 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
175 parameters	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O5^{i}	0.90	1.88	2.7741 (5)	171
O21—H21···N4^{ii}	0.96	1.99	2.8762 (5)	152

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

Data collection: *KappaCCD Software* (Nonius, 1998); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *maXus* (Mackay *et al.*, 1999); software used to prepare material for publication: *maXus*.

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