Crystal structure of 3,4-dihydro-3-oxo-6-ethoxycarbonyl-2*H*-1,4-benzoxazine-2-acetate, $C_{15}H_{17}NO_6$

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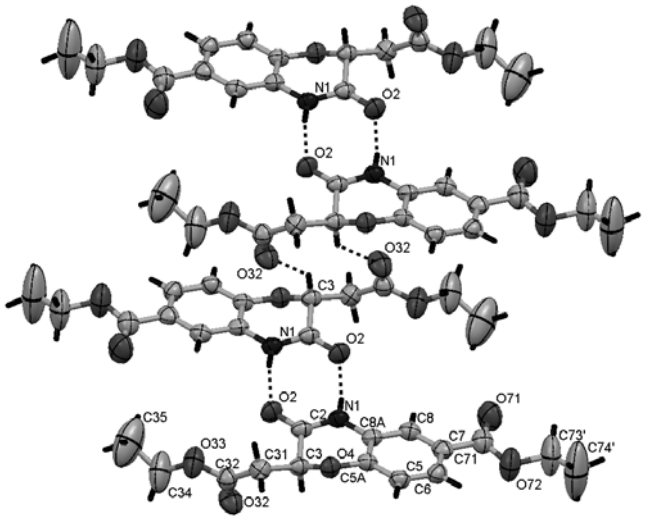

Abstract

$C_{15}H_{17}NO_6$, triclinic, $P\bar{1}$ (no. 2), a = 7.6096(3) Å, b = 8.3617(5) Å, c = 12.3169(5) Å, α = 80.681(4)°, β = 84.982(3)°, γ = 84.376(4)°, V = 767.6 Å^3, Z = 2, $R_{gt}(F)$ = 0.049, $wR_{ref}(F^2)$ = 0.169, T = 290 K.

Source of material

The title compound synthesis was described and published previously [1]. The crystals for X-ray studies were obtained from ethanol solution by slow solvent evaporation.

Experimental details

The end-standing methyl (C35) and ethyl (C73, C74) groups are disordered. The occupancy factors for disordered atoms were refined as 0.56(1) for C35 and 0.59(1) for C73' and C74' atoms. At the final stage they were fixed as 0.60 and 0.40 for respective pair of atoms in both disordered fragments.

Discussion

Benzo[b][1,4]oxazin-3(4*H*)-ones are an important class of heteroaromatic ring systems that have shown a wide range of biological activities [2]. As part of our investigations of the relationship between structure and bioactivity [1], we have synthesized a series of 3,4-dihydro-2*H*-benzo[*b*][1,4]oxazine-3(4*H*)-one derivatives.

In the studied molecule, the six-membered heterocyclic ring adopts envelope conformation with C2 and C3 atoms in flap position. The deviations of those atoms from the plane, passing through aromatic ring and both heteroatoms, are 0.255 Å and 0.730 Å, respectively. Among 25 hits found in CSD [3] for related compounds, heterocyclic ring exhibits also a screw-boat conformation. There are two aliphatic substituents in the studied structure. First, ethyl carboxylate - located at aromatic ring - lies almost in the plane of main molecule moiety with maximal out-of-plane deviation of 0.527 Å for C74. On the other hand, 2-ethoxy-2-oxoethyl, the second equatorial substituent in the heteroring, is located under the main plane (minimal deviation for C31 atom equals 0.395 Å). Moreover, terminal atoms in both substituents (C73, C74 and C35) are disordered in two sites each with s.o.f. = 0.60 and 0.40. The crystal packing is governed by strong hydrogen bond N1–H1···O2 = 2.886(2) Å joining molecules into centrosymmetric dimers. The crystal structure architecture is supplemented by two C–H···O32 weak hydrogen bonds. The strong one C3–H3···O32 = 3.386 Å arranged the dimers into columns (with interlayer distance of 4.952 Å). Then, that adjacent columns are joined by interaction of C5–H5···O32 = 3.550 Å. Both weak intermolecular interactions are based on the oxygen from 2-ethoxy-2-oxoethyl substituents.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.07 × 0.22 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.04 cm^{-1}
Diffractometer, scan mode:	Kuma KM-4 CCD, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17686, 2677
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2152
$N(param)_{refined}$:	233
Programs:	SHELXTL [4], Mercury [5], PLATON [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	2*i*		0.6632	0.5484	0.9487	0.059
H(3)	2*i*		0.6560	0.9087	1.0576	0.054
H(5)	2*i*		1.1779	0.8570	1.0288	0.061
H(6)	2*i*		1.3245	0.8006	0.8652	0.064
H(8)	2*i*		0.9067	0.5801	0.8015	0.060
H(31A)	2*i*		0.6164	0.7238	1.2719	0.062
H(31B)	2*i*		0.6783	0.9005	1.2553	0.062
H(35A)	2*i*	0.60	0.1059	0.7334	1.4474	0.223
H(35B)	2*i*	0.60	0.0013	0.7071	1.3484	0.223
H(35C)	2*i*	0.60	–0.0697	0.8415	1.4203	0.223
H(35D)	2*i*	0.40	–0.0654	0.8548	1.4278	0.246
H(35E)	2*i*	0.40	0.0965	0.9113	1.4777	0.246
H(35F)	2*i*	0.40	0.1042	0.7349	1.4487	0.246
H(73A)	2*i*	0.40	1.4987	0.8402	0.5486	0.107
H(73B)	2*i*	0.40	1.4192	0.6835	0.5229	0.107

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(74A)	2*i*	0.40	1.7057	0.6862	0.6525	0.144
H(74B)	2*i*	0.40	1.7128	0.6061	0.5454	0.144
H(74C)	2*i*	0.40	1.6111	0.5274	0.6541	0.144
H(74D)	2*i*	0.60	1.5961	0.8549	0.5552	0.203
H(74E)	2*i*	0.60	1.7151	0.7051	0.5202	0.203
H(74F)	2*i*	0.60	1.6926	0.7299	0.6443	0.203
H(73C)	2*i*	0.60	1.4144	0.6571	0.5285	0.100
H(73D)	2*i*	0.60	1.5124	0.5301	0.6177	0.100
H(34A)	2*i*		0.1238	1.0085	1.3422	0.151
H(34B)	2*i*		0.0944	0.9215	1.2418	0.151

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*		0.7049(2)	0.6182(2)	0.9815(1)	0.0491(9)	0.0430(8)	0.0584(9)	–0.0123(6)	–0.0070(7)	–0.0139(6)
C(2)	2*i*		0.6082(2)	0.6716(2)	1.0670(1)	0.044(1)	0.0427(9)	0.056(1)	–0.0062(7)	–0.0092(8)	–0.0059(7)
O(2)	2*i*		0.4685(2)	0.6191(2)	1.1070(1)	0.0500(8)	0.0644(8)	0.0765(9)	–0.0207(6)	0.0022(6)	–0.0208(7)
C(3)	2*i*		0.6832(2)	0.8106(2)	1.1094(1)	0.044(1)	0.0439(9)	0.0501(9)	–0.0081(7)	–0.0061(7)	–0.0070(7)
O(4)	2*i*		0.8718(2)	0.7854(2)	1.11016(9)	0.0434(7)	0.0595(8)	0.0534(7)	–0.0104(5)	–0.0053(5)	–0.0160(6)
C(5)	2*i*		1.1233(2)	0.8028(2)	0.9825(2)	0.048(1)	0.046(1)	0.062(1)	–0.0092(7)	–0.0061(8)	–0.0126(8)
C(5A)	2*i*		0.9546(2)	0.7563(2)	1.0111(1)	0.046(1)	0.0387(8)	0.0502(9)	–0.0032(7)	–0.0075(7)	–0.0065(7)
C(6)	2*i*		1.2109(2)	0.7688(2)	0.8851(2)	0.046(1)	0.047(1)	0.066(1)	–0.0049(7)	–0.0018(8)	–0.0045(8)
C(7)	2*i*		1.1287(2)	0.6863(2)	0.8160(2)	0.056(1)	0.0437(9)	0.053(1)	0.0010(8)	–0.0050(8)	–0.0032(8)
C(8)	2*i*		0.9602(2)	0.6370(2)	0.8466(1)	0.057(1)	0.0430(9)	0.052(1)	–0.0050(7)	–0.0097(8)	–0.0085(7)
C(8A)	2*i*		0.8715(2)	0.6719(2)	0.9437(1)	0.047(1)	0.0352(8)	0.053(1)	–0.0046(7)	–0.0092(7)	–0.0033(7)
C(31)	2*i*		0.6089(2)	0.8291(2)	1.2252(1)	0.051(1)	0.055(1)	0.051(1)	–0.0077(8)	–0.0079(8)	–0.0081(8)
C(32)	2*i*		0.4200(3)	0.8981(2)	1.2243(2)	0.057(1)	0.064(1)	0.052(1)	–0.0055(9)	–0.0016(8)	–0.0166(9)
O(32)	2*i*		0.3628(2)	0.9979(2)	1.1523(1)	0.075(1)	0.082(1)	0.0675(9)	0.0172(8)	–0.0117(7)	–0.0054(8)
O(33)	2*i*		0.3232(2)	0.8388(2)	1.3124(1)	0.061(1)	0.126(2)	0.067(1)	–0.0016(9)	0.0122(7)	0.0001(9)
C(35)	2*i*	0.60	0.035(1)	0.787(1)	1.3892(6)	0.092(4)	0.23(1)	0.121(5)	–0.060(5)	–0.035(4)	0.023(6)
C(35')	2*i*	0.40	0.061(1)	0.846(2)	1.4270(6)	0.047(5)	0.31(2)	0.141(8)	–0.022(7)	0.036(6)	–0.087(9)
C(71)	2*i*		1.2199(3)	0.6438(3)	0.7123(2)	0.067(1)	0.071(1)	0.057(1)	–0.002(1)	–0.0019(9)	–0.009(1)
O(72)	2*i*		1.3678(2)	0.7163(3)	0.6851(1)	0.072(1)	0.152(2)	0.077(1)	–0.026(1)	0.0197(8)	–0.039(1)
C(73)	2*i*	0.40	1.476(1)	0.729(1)	0.5773(9)	0.089(6)	0.111(8)	0.064(5)	–0.011(6)	0.008(3)	–0.006(6)
C(74)	2*i*	0.40	1.641(1)	0.628(1)	0.6103(8)	0.073(6)	0.111(6)	0.099(6)	–0.013(4)	0.009(4)	–0.009(5)
C(74')	2*i*	0.60	1.634(1)	0.742(1)	0.5770(6)	0.093(5)	0.190(8)	0.118(5)	–0.037(5)	0.037(4)	–0.016(5)
C(73')	2*i*	0.60	1.4777(9)	0.6446(8)	0.5948(6)	0.078(4)	0.098(4)	0.069(3)	–0.010(3)	0.027(2)	–0.013(4)
O(71)	2*i*		1.1703(3)	0.5520(2)	0.6587(1)	0.116(2)	0.121(2)	0.074(1)	–0.027(1)	0.0150(9)	–0.043(1)
C(34)	2*i*		0.1365(4)	0.9049(5)	1.3152(3)	0.066(2)	0.200(4)	0.104(2)	0.006(2)	0.012(2)	–0.018(2)

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