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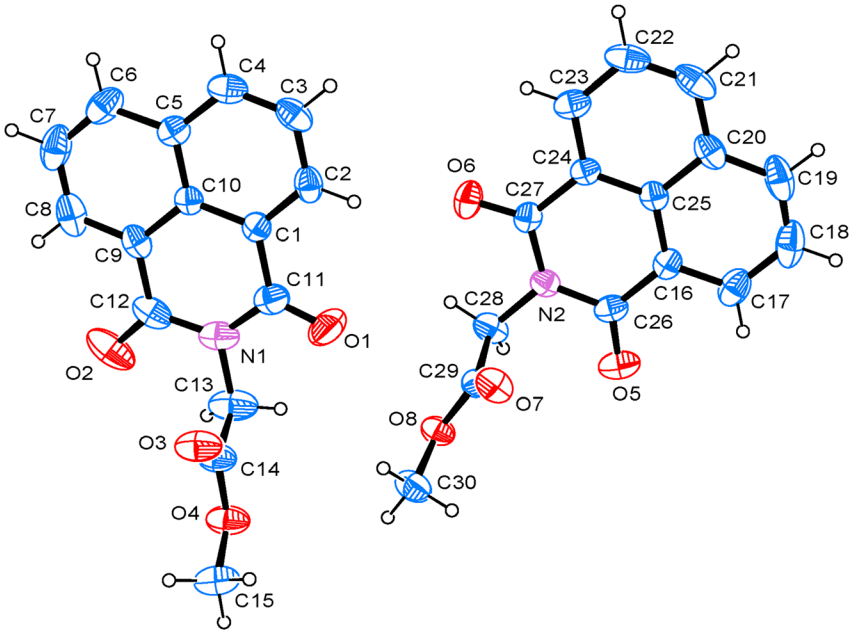
Crystal structure of methyl 2-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)acetate, $C_{15}H_{11}NO_4$



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Abstract

$C_{15}H_{11}NO_4$, monoclinic, $P2_1/n$ (no. 14), a = 8.0909(6) Å, b = 24.4463(18) Å, c = 12.6980(10) Å, β = 96.6020(10)°, V = 2494.9(3) Å^3, Z = 8, $R_{gt}(F)$ = 0.0470, $wR_{ref}(F^2)$ = 0.1273, T = 296 (2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow, block
Size:	0.30 × 0.26 × 0.22 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12813, 4421, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3376
$N(param)_{refined}$:	363
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP [4]

Source of material

All chemicals were purchased from commercial sources and used as received without further purification. The alrestatine (5 mmol, 1.276 g) was dissolved in MeOH (50 mL), then add 2 drops of 98% H_2SO_4. The mixture was refluxed for 5 h. When the reaction vessel had cooled to room temperature, the reaction mixture was filtered. The crystals of the title compound were obtained by controlled solvent evaporation.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8531 (2)	0.60315 (7)	0.61961 (13)	0.0503 (4)
C2	0.8951 (2)	0.57022 (8)	0.53833 (16)	0.0631 (5)
H2	0.873718	0.532863	0.539798	0.076*
C3	0.9690 (2)	0.59241 (10)	0.45427 (16)	0.0692 (6)
H3	0.997032	0.569746	0.400244	0.083*
C4	1.0002 (2)	0.64662 (10)	0.45061 (15)	0.0655 (5)
H4	1.051402	0.660774	0.394608	0.079*
C5	0.9563 (2)	0.68201 (8)	0.53036 (14)	0.0538 (4)
C6	0.9885 (3)	0.73896 (9)	0.5293 (2)	0.0764 (6)
H6	1.039799	0.754092	0.474209	0.092*
C7	0.9450 (3)	0.77153 (9)	0.6079 (3)	0.0902 (8)
H7	0.966144	0.808883	0.605705	0.108*
C8	0.8695 (3)	0.75024 (9)	0.6916 (2)	0.0802 (7)
H8	0.840092	0.773396	0.744510	0.096*
C9	0.8378 (2)	0.69498 (8)	0.69696 (14)	0.0565 (5)
C10	0.88116 (18)	0.66010 (7)	0.61579 (13)	0.0462 (4)
C11	0.7853 (2)	0.57892 (10)	0.71085 (16)	0.0687 (5)
C12	0.7627 (2)	0.67177 (11)	0.78701 (16)	0.0710 (5)
C13	0.6691 (3)	0.59165 (14)	0.87719 (18)	0.0962 (7)
H13A	0.608728	0.558518	0.855231	0.115*
H13B	0.590652	0.617316	0.901937	0.115*
C14	0.8011 (2)	0.57840 (9)	0.96690 (15)	0.0672 (5)
C15	0.8401 (3)	0.54357 (12)	1.14185 (18)	0.0929 (8)
H15A	0.907640	0.513425	1.123952	0.139*
H15B	0.775282	0.532681	1.197016	0.139*
H15C	0.910442	0.573746	1.166019	0.139*
C16	0.5526 (2)	0.29866 (7)	0.44875 (14)	0.0519 (4)
C17	0.5228 (3)	0.24316 (9)	0.44849 (19)	0.0745 (6)
H17	0.474549	0.227207	0.504023	0.089*
C18	0.5649 (3)	0.21084 (10)	0.3647 (3)	0.0935 (8)
H18	0.542590	0.173520	0.364574	0.112*
C19	0.6373 (3)	0.23283 (10)	0.2843 (2)	0.0870 (8)
H19	0.666207	0.210337	0.230306	0.104*
C20	0.6701 (2)	0.28953 (9)	0.28067 (15)	0.0626 (5)
C21	0.7449 (3)	0.31459 (13)	0.19863 (16)	0.0785 (7)
H21	0.774036	0.293333	0.142887	0.094*
C22	0.7757 (2)	0.36922 (12)	0.19895 (16)	0.0771 (7)
H22	0.824638	0.385004	0.143509	0.092*
C23	0.7339 (2)	0.40188 (9)	0.28288 (15)	0.0620 (5)
H23	0.755736	0.439231	0.282932	0.074*
C24	0.66094 (19)	0.37913 (7)	0.36478 (12)	0.0462 (4)
C25	0.62662 (19)	0.32267 (7)	0.36514 (13)	0.0473 (4)
C26	0.5078 (2)	0.33250 (8)	0.53666 (14)	0.0548 (4)
C27	0.6236 (2)	0.41371 (7)	0.45420 (14)	0.0497 (4)
C28	0.5091 (3)	0.42102 (9)	0.62406 (14)	0.0671 (5)
H28A	0.396816	0.412703	0.638857	0.081*
H28B	0.512754	0.459416	0.605206	0.081*
C29	0.6273 (2)	0.41103 (7)	0.72199 (14)	0.0511 (4)
C30	0.6736 (3)	0.42981 (10)	0.90465 (16)	0.0773 (6)
H30A	0.702705	0.392464	0.921173	0.116*
H30B	0.617233	0.445047	0.960372	0.116*
H30C	0.772767	0.450477	0.898003	0.116*
N1	0.73822 (19)	0.61502 (9)	0.78664 (13)	0.0721 (5)
N2	0.54838 (17)	0.38796 (6)	0.53442 (11)	0.0518 (4)
O1	0.7708 (3)	0.52976 (8)	0.72326 (15)	0.1114 (6)
O2	0.7218 (2)	0.69940 (9)	0.85941 (13)	0.1139 (7)
O3	0.94607 (17)	0.58249 (7)	0.96467 (11)	0.0827 (5)
O4	0.72982 (17)	0.56002 (7)	1.04889 (11)	0.0826 (5)
O5	0.43871 (17)	0.31496 (7)	0.60989 (11)	0.0797 (4)
O6	0.65678 (18)	0.46228 (5)	0.45978 (12)	0.0735 (4)
O7	0.75816 (16)	0.38841 (6)	0.72558 (10)	0.0698 (4)
O8	0.56556 (15)	0.43198 (6)	0.80606 (10)	0.0622 (4)

Experimental details

All H-atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq}(parent C-atom).

Comment

Alrestatin ((1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl) acetic acid) acting as aldose reductase inhibitor has been developed for the treatment of secondary complications in diabetes [5, 6], but has been withdrawn because of adverse effects [7]. Alrestatin methyl ester is an important intermediate in the derivatization of alrestatin, so the synthesis and crystal structure of it is of great significance to study the reduction of side effects.

The asymmetric part of the unit cell contains two crystallographically independent molecules, as shown in the figure. The bond lengths of C14=O3, C29=O7, C11=O1, C12=O2, C26=O5 and C27=O6 in the title molecule are 1.181(2) Å, 1.191(2) Å, 1.219(3) Å, 1.217(3) Å, 1.217(2) Å and 1.218 (2) Å respectively. They are similar to reported in the literature [8, 9]. It illustrated characteristic C=O double bonds. In addition, the bond length of N1–C11, N1–C12, N1–C13, N2–C26, N2–C27, N2–C28 in the title molecule are in the range of 1.391–1.460 Å. The bond lengths within the aromatic rings as well as C13–C14 and C28–C29 ones are close to their normal values.

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