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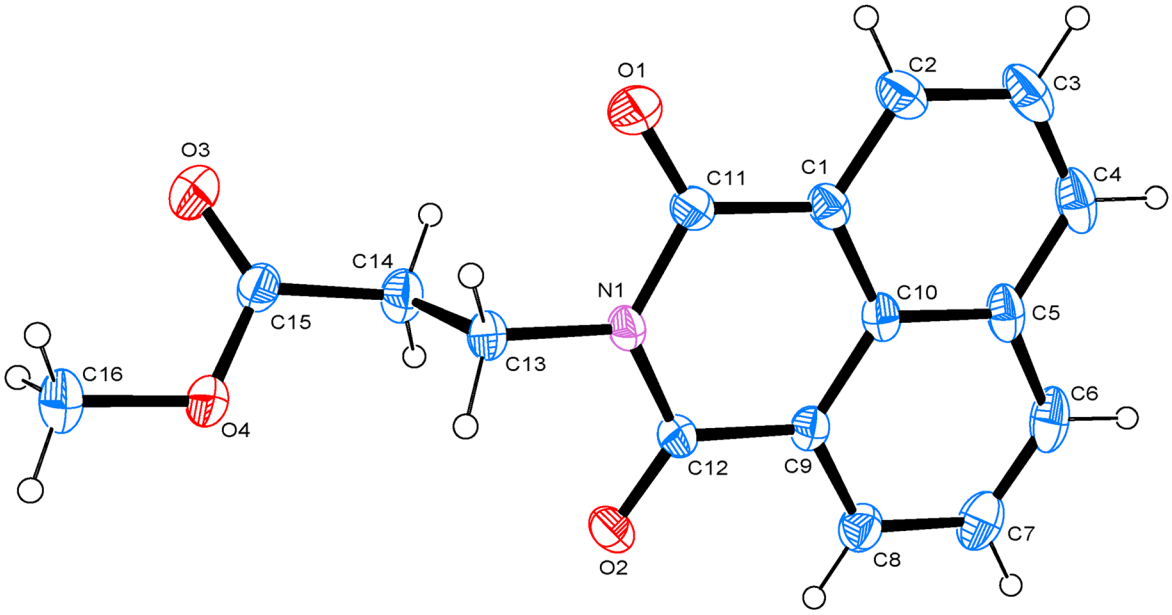
Crystal structure of methyl 3-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)propanoate, $C_{16}H_{13}NO_4$



https://doi.org/10.1515/ncrs-2022-0241
Received May 9, 2022; accepted May 30, 2022;
published online June 10, 2022

Abstract

$C_{16}H_{13}NO_4$, monoclinic, $P2_1/c$ (no. 14), a = 9.930(3) Å, b = 6.9807(18) Å, c = 18.954(5) Å, β = 93.080(4)°, V = 1311.9(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0400, $wR_{ref}(F^2)$ = 0.1142, T = 296 (2) K.

CCDC no.: 2171271

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

Table 1: Data collection and handling.

Crystal:	Yellow, block
Size:	0.24 × 0.24 × 0.22 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.10 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}:	6544, 2336, 0.020
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1813
$N(param)_{refined}$:	191
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 3-(1,3-dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl)propanoic acid (5 mmol, 1.345 g) were dissolved in MeOH (50 mL), then two drops of 98% H_2SO_4 were added. The mixture was refluxed for 5 h. When the reaction vessel had cooled to room temperature, the reaction mixture was filtered.

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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	0.43995 (15)	0.7539 (2)	0.53015 (7)	0.0351 (4)
C2	0.40334 (19)	0.7869 (2)	0.46021 (8)	0.0463 (4)
H2	0.313307	0.809033	0.446599	0.056*
C3	0.5006 (2)	0.7874 (2)	0.40924 (8)	0.0552 (5)
H3	0.474848	0.811025	0.362136	0.066*
C4	0.6326 (2)	0.7535 (2)	0.42850 (9)	0.0540 (5)
H4	0.696017	0.754312	0.394133	0.065*
C5	0.67531 (18)	0.7171 (2)	0.49974 (8)	0.0438 (4)
C6	0.81092 (18)	0.6803 (3)	0.52299 (11)	0.0571 (5)
H6	0.877159	0.679474	0.490119	0.069*
C7	0.84657 (18)	0.6462 (3)	0.59194 (11)	0.0571 (5)
H7	0.936254	0.621404	0.605556	0.068*
C8	0.74906 (15)	0.6480 (2)	0.64288 (9)	0.0443 (4)
H8	0.774277	0.624502	0.690045	0.053*
C9	0.61609 (14)	0.6846 (2)	0.62307 (7)	0.0336 (3)
	0.57628 (15)	0.71910 (19)	0.55145 (7)	0.0338 (4)
C11	0.33646 (16)	0.7571 (2)	0.58334 (8)	0.0366 (4)
C12	0.51446 (15)	0.6896 (2)	0.67723 (7)	0.0344 (3)
C13	0.28072 (15)	0.7456 (2)	0.70838 (8)	0.0378 (4)
H13A	0.210057	0.834356	0.692838	0.045*
H13B	0.323020	0.793948	0.752144	0.045*
C14	0.21953 (16)	0.5498 (2)	0.72125 (8)	0.0454 (4)
H14A	0.186431	0.496725	0.676312	0.055*
H14B	0.289671	0.465384	0.740686	0.055*
C15	0.10621 (15)	0.5536 (2)	0.77041 (8)	0.0412 (4)
C16	0.03774 (18)	0.6404 (3)	0.88320 (10)	0.0614 (5)
H16A	−0.033515	0.719961	0.863399	0.092*
H16B	0.073734	0.695963	0.926543	0.092*
H16C	0.002903	0.515098	0.892445	0.092*
N1	0.38194 (12)	0.73347 (16)	0.65429 (6)	0.0336 (3)
O1	0.21654 (11)	0.77945 (19)	0.56834 (6)	0.0572 (4)
O2	0.54267 (11)	0.65881 (18)	0.73959 (5)	0.0506 (3)
O3	−0.00549 (12)	0.4978 (2)	0.75541 (6)	0.0703 (4)
O4	0.14359 (10)	0.62542 (18)	0.83370 (6)	0.0504 (3)

The crystals of the title compound were obtained by controlled solvent evaporation.

Experimental details

All H-atoms bonded to C 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 acting as aldose reductase inhibitors has been developed for the treatment of secondary complications in diabetes [5, 6], but has been withdrawn because of adverse effects [7]. The title compound 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.

Single-crystal structure analysis revealed that the title compound crystallized in the monoclinic space group $P2_1/c$. The molecular title structure is presented in the Figure. The bond lengths of C15=O3, C11=O1, and C12=O2 in the title molecule are 1.195(2) Å, 1.219(2) Å, and 1.219(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 in the title molecule are in the range of 1.398–1.476 Å. The bond lengths within the aromatic ring as well as C13–C14 and C14–C15 ones are close to their normal values.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
Research funding: This work was supported by the 22nd College Students Extracurricular Academic Science and Technology Works Competition Project of Hengyang Normal University (13) and the College students Innovation and Entrepreneurship Training Program (cxcy2022038).
Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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