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Crystal structure of (*E*)-1-(4-benzyl-3,5-dioxomorpholin-2-ylidene)ethyl acetate, $C_{15}H_{15}N_1O_5$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.18 × 0.16 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.7°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4176, 2944, 0.016
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2137
$N(\text{param})_{\text{refined}}$:	192
Programs:	Bruker [1], SHELX [2, 3]

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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.

Source of material

The reaction of diglycolic acid (6.7 g, 50 mmol) and benzylamine (5.4 g, 50 mmol) yielded the product (6.1 g, 60%) as colorless crystals. A solution of potassium bis(trimethylsilyl) amide (KHMDs) (1.159 g, 5.81 mmol) in tetrahydrofuran (THF) (20 mL) was cooled to -78°C under argon. Subsequently, a solution of the colorless crystals that have been obtained (0.500 g, 2.32 mmol) [4], distilled diphenyl chlorophosphate (1.373 g, 5.11 mmol), and distilled hexamethylphosphoramide (HMPA) (1.041 g, 5.81 mmol) in THF (5 mL) was added dropwise over 5 min. After 15 min at -78°C , the reaction mixture was diluted with diethyl ether (50 mL). Water (50 mL) was then added, and the mixture was extracted with EtOAc. The organic phase was dried over anhydrous MgSO_4 and concentrated. Flash chromatography (petroleum ether/EtOAc, 7:3 + 0.1% NEt_3) was used to obtain the title compound as colorless block.

Experimental details

All hydrogen atoms were placed in the calculated positions and all the non-hydrogen atoms were refined anisotropically.

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Abstract

$C_{15}H_{15}N_1O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 5.075(4)$ Å, $b = 11.147(8)$ Å, $c = 13.319(9)$ Å, $\alpha = 106.064(10)^\circ$, $\beta = 96.091(11)^\circ$, $\gamma = 98.189(10)^\circ$, $V = 708.2(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0444$, $wR_{\text{ref}}(F^2) = 0.1581$, $T = 296$ K.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.2758 (2)	0.25763 (11)	0.77312 (9)	0.0491 (3)
O2	0.5557 (3)	0.19720 (14)	0.88394 (11)	0.0645 (4)
O3	0.7462 (3)	0.22041 (11)	0.58637 (9)	0.0540 (4)
O4	0.6944 (3)	0.45584 (11)	0.82403 (10)	0.0601 (4)
O5	1.2367 (3)	0.47210 (14)	0.57681 (12)	0.0702 (4)
N1	0.9774 (3)	0.46502 (13)	0.70509 (11)	0.0457 (4)
C1	1.1582 (4)	0.81208 (19)	0.85985 (15)	0.0645 (5)
H1	1.300609	0.810195	0.909143	0.077*
C2	1.0682 (5)	0.92528 (19)	0.86500 (18)	0.0753 (7)
H2	1.150879	0.998838	0.917953	0.090*
C3	0.8590 (5)	0.9298 (2)	0.7929 (2)	0.0744 (7)
H3	0.798946	1.005732	0.796849	0.089*
C4	0.7394 (4)	0.8211 (2)	0.7151 (2)	0.0726 (6)
H4	0.598107	0.823635	0.665680	0.087*
C5	0.8259 (4)	0.70769 (17)	0.70910 (15)	0.0527 (5)
H5	0.741517	0.634717	0.655869	0.063*
C6	1.0355 (3)	0.70137 (15)	0.78093 (12)	0.0426 (4)
C7	1.1414 (3)	0.58140 (16)	0.77989 (15)	0.0527 (5)
H7A	1.322547	0.589932	0.762390	0.063*
H7B	1.152946	0.572299	0.850493	0.063*
C8	1.0428 (4)	0.42022 (17)	0.60509 (15)	0.0517 (4)
C9	0.8524 (5)	0.30636 (19)	0.53250 (15)	0.0616 (5)
H9A	0.704602	0.334445	0.498345	0.074*
H9B	0.946114	0.262111	0.477833	0.074*
C10	0.6245 (3)	0.27695 (15)	0.67069 (12)	0.0429 (4)
C11	0.7589 (3)	0.40466 (15)	0.73986 (13)	0.0437 (4)
C12	0.4084 (3)	0.20737 (16)	0.68940 (13)	0.0445 (4)
C13	0.2845 (4)	0.07532 (18)	0.62471 (17)	0.0638 (5)
H13A	0.363514	0.053300	0.561203	0.096*
H13B	0.094045	0.070013	0.606696	0.096*
H13C	0.316132	0.017634	0.664583	0.096*
C14	0.3741 (3)	0.25106 (16)	0.87100 (13)	0.0460 (4)
C15	0.2252 (4)	0.3173 (2)	0.95281 (16)	0.0663 (6)
H15A	0.285295	0.304005	1.019070	0.099*
H15B	0.035783	0.284220	0.932244	0.099*
H15C	0.257792	0.406457	0.960155	0.099*

Comment

Heterocyclic compounds constitute one of the largest and most versatile families of organic compounds [5, 6]. There are many heterocyclic compounds that are being isolated

from natural sources and day by day the number is increasing rapidly due to their enormous utility [7]. *N*-heterocycles are important, not only because of their abundance, but above all because of their chemical, biological and technical significance [8, 9].

In the molecules forming the title crystal structure, the dihedral angle of ring formed by C8–C11 and the moiety formed by C7–C11 is 26.4°. The dihedral angle between the phenyl ring formed and the ring formed by C8–C11 is, close to a right angle. The bond lengths and angles are in the expected ranges.

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