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Crystal structure of ethyl (*E*)-1-(3-ethoxy-3-oxoprop-1-en-1-yl)-2-(4-nitrophenyl)-2,5-dihydro-1*H*-benzo[*b*][1,4]diazepine-3-carboxylate, C₂₃H₂₃N₃O₆

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Abstract

C₂₃H₂₃N₃O₆, monoclinic, *P*₂₁/*c* (no. 14), *a* = 11.235(6) Å, *b* = 11.251(7) Å, *c* = 17.814(10) Å, β = 98.872(12)°, *V* = 2225(2) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0581, *wR*_{ref}(*F*²) = 0.1590, *T* = 296(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.32 × 0.28 × 0.25 mm
Wavelength:	Mo <i>K</i> α radiation (0.71073 Å)
μ:	0.10 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	10842, 3899, 0.049
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2268
<i>N</i> (<i>param</i>) _{refined} :	289
Programs:	Olex2 [1], SHELX [2], CrysAlis ^{PRO} [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N1	0.4313(2)	0.90636(19)	0.23680(12)	0.0516(6)
N2	0.4406(2)	0.7899(2)	0.09301(13)	0.0680(7)
H2B	0.4493	0.7288	0.0653	0.082*
N3	0.0891(3)	1.3336(4)	0.1265(3)	0.1056(11)
O1	0.6725(2)	0.9493(2)	0.47618(11)	0.0750(6)
O2	0.5099(2)	0.8317(2)	0.46818(11)	0.0791(7)
O3	0.6767(2)	1.1342(2)	0.16645(15)	0.0863(7)
O4	0.7177(2)	1.0092(2)	0.07702(13)	0.0891(8)
O5	0.0360(3)	1.3347(4)	0.0618(2)	0.1530(15)
O6	0.0637(3)	1.3964(4)	0.1776(2)	0.1564(15)
C1	0.2235(3)	0.8383(3)	0.22637(18)	0.0699(9)
H1A	0.2160	0.8798	0.2705	0.084*
C2	0.1289(3)	0.7715(4)	0.1917(2)	0.0894(11)
H2A	0.0581	0.7672	0.2127	0.107*
C3	0.1385(4)	0.7110(4)	0.1261(2)	0.0917(12)
H3A	0.0750	0.6642	0.1030	0.110*
C4	0.2416(4)	0.7197(3)	0.09493(19)	0.0788(10)
H4A	0.2470	0.6795	0.0500	0.095*
C5	0.3386(3)	0.7871(3)	0.12872(16)	0.0586(8)
C6	0.3305(3)	0.8450(2)	0.19665(16)	0.0541(7)
C7	0.4651(3)	0.8854(2)	0.31229(15)	0.0530(7)
H7A	0.4223	0.8264	0.3331	0.064*
C8	0.5517(3)	0.9384(3)	0.36043(15)	0.0555(7)
H8A	0.5978	0.9989	0.3440	0.067*
C9	0.5722(3)	0.8996(3)	0.43854(16)	0.0593(8)
C10	0.7016(3)	0.9160(4)	0.55509(18)	0.0949(12)
H10A	0.6370	0.9398	0.5823	0.114*
H10B	0.7109	0.8305	0.5594	0.114*
C11	0.8069(4)	0.9711(6)	0.5860(2)	0.145(2)
H11A	0.8273	0.9487	0.6385	0.217*
H11B	0.7967	1.0557	0.5824	0.217*
H11C	0.8704	0.9473	0.5589	0.217*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C12	0.5277(3)	0.8726(3)	0.09486(15)	0.0599(8)
H12A	0.5808	0.8605	0.0604	0.072*
C13	0.5500(3)	0.9696(2)	0.13843(15)	0.0524(7)
C14	0.4807(3)	1.0089(2)	0.19971(14)	0.0509(7)
H14A	0.5388	1.0477	0.2388	0.061*
C15	0.6515(3)	1.0461(3)	0.12985(17)	0.0621(8)
C16	0.8269(4)	1.0748(4)	0.0714(3)	0.1030(13)
H16A	0.8414	1.0753	0.0191	0.124*
H16B	0.8183	1.1564	0.0873	0.124*
C17	0.9244(5)	1.0207(7)	0.1181(4)	0.185(3)
H17A	0.9969	1.0641	0.1147	0.278*
H17B	0.9331	0.9403	0.1017	0.278*
H17C	0.9098	1.0207	0.1698	0.278*
C18	0.3799(3)	1.0967(2)	0.17752(15)	0.0516(7)
C19	0.3199(3)	1.1063(3)	0.10509(16)	0.0617(8)
H19A	0.3438	1.0601	0.0667	0.074*
C20	0.2240(3)	1.1839(3)	0.08806(19)	0.0717(9)
H20A	0.1826	1.1898	0.0388	0.086*
C21	0.1917(3)	1.2515(3)	0.1452(2)	0.0696(9)
C22	0.2491(3)	1.2450(3)	0.2175(2)	0.0756(10)
H22A	0.2251	1.2923	0.2553	0.091*
C23	0.3435(3)	1.1674(3)	0.23402(17)	0.0644(8)
H23A	0.3838	1.1619	0.2836	0.077*

Source of material

The solution of 4-nitrobenzaldehyde (151 mg, 1 mmol), o-phenylenediamine (108 mg, 1 mmol), ethyl propiolate (245 mg, 2.5 mmol) and 0.1 mL acetic acid in ethanol (20 mL) was heated in a steam bath for 2 h. The reaction mixture was evaporated to dryness in vacuo. The residue was purified by flash column chromatography (silica gel) using EA/hexane (10–15%) to afford a yellow solid. Yield, 354 mg, 81%. The title compound (87 mg, 0.2 mmol) was dissolved in 30% EA/Hex, and slowly evaporated at room temperature. After six days, yellow crystals were obtained.

Experimental details

Using Olex2 [1] the structure was solved with the SHELXS refinement package [2, 3]. All of the hydrogen atoms were placed in the calculated positions.

Comment

Benzo[b][1,4]diazepines are important nitrogen-containing heterocyclic compounds, which have extensive applications in synthetic chemistry [4–6] and medicinal chemistry [7–10].

The crystal structure of title compound is shown in the figure. The NMR results are consistent with the proposed structure. The bond lengths and angles in the title molecule are in the normal ranges. In conclusion, an efficient and

novel one-pot multi-component approach to the synthesis of benzo[b][1,4]diazepines from simple and readily available starting materials under refluxing conditions without modifications has been developed. We hope that this approach may be of value to others seeking novel synthetic fragments for medicinal chemistry.

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