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The crystal structure of 5*H*-dibenzo[*b,e*]azepine-6,11-dione, C₁₄H₉NO₂

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	29.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6181, 3727, 0.040
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3061
$N(\text{param})_{\text{refined}}$:	308
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

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Abstract

C₁₄H₉NO₂, orthorhombic, *Pca*2₁ (no. 29), $a = 27.190(4)$ Å, $b = 4.3317(6)$ Å, $c = 17.297(2)$ Å, $V = 2037.2(5)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0524$, $wR_{\text{ref}}(F^2) = 0.1122$, $T = 150.0(1)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The 5*H*-dibenzo[*b,e*]azepine-6,11-dione has been synthesized by one-pot method: the mixture of 0.1391 g lead(II)

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chloride (0.5 mmol), 0.020 g NaOH (0.5 mmol), and 0.1206 g 2'-aminobenzophenone-2-carboxylic acid (0.5 mmol) were dissolved in 15 mL (water-ethanol = 1:2) at room temperature. Then the mixture was kept at 75 °C for 4.5 h with stirring. After cooling to room temperature, the solution was moved into a small conical bottle and evaporated. The block crystals of the title compound were obtained in 5 weeks.

Experimental details

The hydrogen atoms were positioned geometrically (C-H = 0.93 Å and N-H = 0.86 Å). Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms.

Comment

Seven-membered heterocyclic compounds exhibit many potential applications in drugs and materials [5]. In a previous work, we have synthesized and structurally characterized heterocyclic compounds [6].

The compound contains two twist 5*H*-dibenzo[*b,e*]azepine-6,11-dione molecules in the asymmetric unit (see the Figure). The bond lengths of C-O are 1.223(4) Å (C14-O1), 1.235(4) Å (C7-O2), 1.249(4) Å (C24-O3) and 1.215(4) Å (C21-O4), respectively, which are obviously double bonds. Also

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.23151 (10)	1.1237 (7)	0.40372 (17)	0.0325 (7)
O2	0.38127 (10)	0.6757 (6)	0.56548 (15)	0.0246 (6)
O3	0.48003 (9)	0.8347 (6)	0.43708 (14)	0.0245 (6)
O4	0.62737 (10)	0.3006 (7)	0.60225 (16)	0.0370 (8)
N1	0.37787 (11)	1.0004 (7)	0.46560 (18)	0.0201 (7)
H1	0.406255	0.924542	0.454597	0.024*
N2	0.48208 (11)	0.5007 (7)	0.53463 (17)	0.0214 (7)
H2	0.453107	0.572572	0.543268	0.026*
C1	0.31314 (13)	1.2985 (8)	0.3969 (2)	0.0196 (8)
C2	0.30286 (14)	1.5040 (9)	0.3363 (2)	0.0241 (9)
H2A	0.270285	1.555356	0.326154	0.029*
C3	0.33918 (15)	1.6317 (9)	0.2917 (2)	0.0256 (9)
H3	0.331570	1.770661	0.252469	0.031*
C4	0.38749 (15)	1.5491 (9)	0.3063 (2)	0.0240 (9)
H4	0.412527	1.631853	0.276104	0.029*
C5	0.39913 (14)	1.3452 (9)	0.3652 (2)	0.0216 (8)
H5	0.431729	1.290081	0.373857	0.026*
C6	0.36192 (13)	1.2220 (8)	0.4117 (2)	0.0180 (8)
C7	0.35821 (13)	0.8798 (8)	0.5313 (2)	0.0188 (8)
C8	0.31187 (13)	1.0002 (8)	0.5652 (2)	0.0198 (8)
C9	0.30806 (15)	0.9743 (9)	0.6460 (2)	0.0272 (9)
H9	0.333814	0.886493	0.673718	0.033*
C10	0.26687 (15)	1.0767 (10)	0.6851 (2)	0.0337 (11)
H10	0.265285	1.063217	0.738662	0.040*
C11	0.22812 (16)	1.1991 (10)	0.6438 (2)	0.0332 (10)
H11	0.200338	1.267870	0.669994	0.040*
C12	0.22986 (14)	1.2212 (9)	0.5646 (2)	0.0264 (9)
H12	0.203017	1.299076	0.537582	0.032*
C13	0.27221 (13)	1.1262 (9)	0.5242 (2)	0.0207 (8)
C14	0.27010 (13)	1.1666 (9)	0.4385 (2)	0.0200 (8)
C15	0.55262 (13)	0.5314 (8)	0.4414 (2)	0.0205 (8)
C16	0.56090 (14)	0.6141 (9)	0.3647 (2)	0.0256 (9)
H16	0.536696	0.721675	0.337875	0.031*
C17	0.60429 (14)	0.5397 (10)	0.3275 (2)	0.0292 (10)
H17	0.609050	0.595254	0.276071	0.035*
C18	0.64080 (15)	0.3813 (10)	0.3674 (2)	0.0312 (10)
H18	0.669805	0.325489	0.342568	0.037*
C19	0.63366 (15)	0.3077 (9)	0.4439 (2)	0.0286 (10)
H19	0.658624	0.207849	0.470846	0.034*
C20	0.58978 (13)	0.3793 (8)	0.4824 (2)	0.0227 (8)
C21	0.58892 (14)	0.2939 (8)	0.5666 (2)	0.0220 (8)
C22	0.54397 (14)	0.1770 (8)	0.6046 (2)	0.0206 (8)
C23	0.49620 (14)	0.2756 (8)	0.5885 (2)	0.0197 (8)
C24	0.50384 (13)	0.6310 (9)	0.4719 (2)	0.0204 (8)
C25	0.45704 (14)	0.1648 (8)	0.6343 (2)	0.0222 (9)
H25	0.425211	0.233391	0.624704	0.027*
C26	0.46526 (15)	−0.0436 (9)	0.6931 (2)	0.0248 (9)
H26	0.439052	−0.115078	0.722685	0.030*
C27	0.51273 (16)	−0.1472 (8)	0.7084 (2)	0.0268 (9)
H27	0.518304	−0.290562	0.747461	0.032*
C28	0.55118 (16)	−0.0361 (9)	0.6654 (2)	0.0242 (9)
H28	0.582911	−0.102825	0.676544	0.029*

other bond lengths are as expected [7]. The dihedral angles of ring 1 (C1-C2-C3-C4-C5-C6) and ring 2 (C1-C6-N1-C7-C8-C13-C14), ring 2 (C1-C6-N1-C7-C8-C13-C14) and ring 3 (C8-C9-C10-C11-C12-C13), ring 1 (C1-C2-C3-C4-C5-C6) and ring 3 (C8-C9-C10-C11-C12-C13), ring 4 (C15-C16-C17-C18-C19-C20) and ring 5 (C15-C20-C21-C22-C23-N2-C24), ring 5 (C15-C20-C21-C22-C23-N2-C24) and ring 6 (C22-C23-C25-C26-C27-C28), ring 4 (C15-C16-C17-C18-C19-C20) and ring 6 (C22-C23-C25-C26-C27-C28) are 15.6°, 22.1°, 37.7°, 15.6°, 11.2°, 26.7°, respectively. The molecules are stabilized through N-H...O intermolecular hydrogen bonds, linking the molecules into dimers. In the crystal packing, the distances between two symmetry related seven-membered rings 2 (C1-C6-N1-C7-C8-C13-C14) and between two seven-membered rings 5 (C15-C20-C21-C22-C23-N2-C24) are 3.784 and 3.619 Å, respectively, showing the presence of weak π - π interactions.

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