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Crystal structure of 9-(3-phenoxyphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione, $C_{25}H_{23}NO_3$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.26 × 0.22 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11714, 4510, 0.101
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1572
$N(\text{param})_{\text{refined}}$:	273
Programs:	CrysAlis ^{PRO} [1], SHELX [2], OLEX2 [3]

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Abstract

$C_{25}H_{23}NO_3$, monoclinic, $P2_1/c$ (no. 14), $a = 10.972(6)$ Å, $b = 7.392(4)$ Å, $c = 25.804(10)$ Å, $\beta = 109.349(18)^\circ$, $V = 1974.6(17)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0640$, $wR_{\text{ref}}(F^2) = 0.2381$, $T = 293(2)$ K.

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The crystal 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

A mixture of 1,3-cyclohexanedione (20 mmol), 3-phenoxybenzaldehyde (10 mmol), and ammonium acetate (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6422(3)	0.3106(4)	0.12157(12)	0.0522(8)
H1	0.672443	0.203602	0.130801	0.063*
O1	0.6962(2)	0.9363(3)	0.15260(11)	0.0670(8)
O2	0.2914(3)	0.6657(4)	0.01915(11)	0.0808(9)
O3	0.1647(3)	0.8810(4)	0.17725(13)	0.0875(10)
C1	0.7413(3)	0.7811(5)	0.16030(14)	0.0500(9)
C2	0.8822(3)	0.7517(6)	0.19168(19)	0.0752(12)
H2AA ^a	0.931811	0.782932	0.168058	0.090*
H2AB ^a	0.907693	0.834081	0.222702	0.090*
H2BC ^b	0.930286	0.853530	0.184641	0.090*
H2BD ^b	0.893524	0.752487	0.230594	0.090*
C3A ^a	0.9153(14)	0.5727(15)	0.2115(7)	0.062(5)
H3AA ^a	0.894273	0.559467	0.244984	0.074*
H3AB ^a	1.008225	0.559894	0.221415	0.074*
C3B ^b	0.9399(5)	0.5837(8)	0.1792(4)	0.061(2)
H3BA ^b	1.022002	0.562611	0.207854	0.073*
H3BB ^b	0.956558	0.598842	0.144844	0.073*
C4	0.8550(3)	0.4220(5)	0.17495(16)	0.0577(10)
H4AA ^a	0.864355	0.311731	0.196306	0.069*
H4AB ^a	0.898731	0.404795	0.148242	0.069*
H4BC ^b	0.883454	0.326512	0.155845	0.069*
H4BD ^b	0.865505	0.378795	0.211680	0.069*
C5	0.7159(3)	0.4571(5)	0.14590(13)	0.0446(8)
C6	0.6612(3)	0.6229(5)	0.14003(12)	0.0422(8)
C7	0.5163(3)	0.6476(4)	0.11372(13)	0.0428(8)
H7	0.501075	0.759079	0.091948	0.051*
C8	0.4611(3)	0.4917(5)	0.07538(12)	0.0432(8)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C9	0.5216(3)	0.3294(5)	0.08290(13)	0.0472(9)
C10	0.4691(3)	0.1651(5)	0.04887(16)	0.0645(11)
H10A	0.510917	0.151813	0.021327	0.077*
H10B	0.489201	0.058807	0.072205	0.077*
C11	0.3267(4)	0.1763(6)	0.02115(18)	0.0812(14)
H11A	0.283912	0.154673	0.047989	0.097*
H11B	0.299950	0.082038	−0.006473	0.097*
C12	0.2842(4)	0.3582(6)	−0.00589(16)	0.0692(12)
H12A	0.307442	0.365075	−0.038982	0.083*
H12B	0.190860	0.366196	−0.016747	0.083*
C13	0.3420(3)	0.5171(6)	0.02991(14)	0.0538(10)
C14	0.4475(3)	0.6645(4)	0.15619(13)	0.0422(8)
C15	0.3418(3)	0.7771(5)	0.14717(13)	0.0467(9)
H15	0.315458	0.850789	0.116221	0.056*
C16	0.2757(3)	0.7797(5)	0.18423(15)	0.0525(9)
C17	0.3136(3)	0.6719(5)	0.23059(14)	0.0541(10)
H17	0.268191	0.674479	0.255276	0.065*
C18	0.4186(3)	0.5615(5)	0.23979(14)	0.0573(10)
H18	0.444755	0.488426	0.270871	0.069*
C19	0.4859(3)	0.5581(5)	0.20320(14)	0.0532(10)
H19	0.557794	0.483553	0.210056	0.064*
C20	0.1346(4)	1.0253(6)	0.14092(16)	0.0567(10)
C21	0.2017(4)	1.1814(7)	0.15464(18)	0.0730(12)
H21	0.273634	1.187749	0.186275	0.088*
C22	0.1629(5)	1.3304(6)	0.1216(2)	0.0871(15)
H22	0.208975	1.438092	0.130608	0.105*
C23	0.0565(6)	1.3201(8)	0.0755(2)	0.0927(17)
H23	0.029199	1.421525	0.053388	0.111*
C24	−0.0101(5)	1.1609(10)	0.0618(2)	0.1072(19)
H24	−0.082028	1.152795	0.030191	0.129*
C25	0.0309(4)	1.0135(7)	0.0953(2)	0.0876(14)
H25	−0.013517	0.904561	0.086317	0.105*

Occupancies: ^a = 0.313(15), ^b = 0.687(15).

Experimental details

H atoms bonded to C atoms from organic ligands were positioned geometrically and refined using a riding model, with C—H = 0.93 Å, with *U*_{iso}(H) = 1.2 times *U*_{eq}(C).

Discussion

Natural products have impact on chemical biology and drug discovery. Hexahydropyridine derivatives cover a wide range of biological properties, including anti-oxidant,

anti-inflammatory and anti-microbial as well as anticancer activities [4, 5]. In this paper, we describe a new layered compound by means of the interaction of hydrogen bonds.

The compound displays a two-dimensional layered structure through two kinds of hydrogen bonds including N—H⋯O and C—H⋯O type connections. The molecular structural unit is provided in Figure. In this structure, the dihedral angle between two adjacent six-membered rings [six-membered ring: N(1), C(5)—C(9) and C(14)—C(19)] is 86.89°. The bond lengths of C—C, C—N and C—O are in the range of 1.351–1.524 Å, 1.371–1.376 Å and 1.223–1.394 Å, respectively. These bond distances presented in this compound fall in their normal scopes [6]. In addition, the two types of hydrogen bonds (N—H⋯O and C—H⋯O) could be found in its packing structure. By means of these interactions of hydrogen bonds, a two-dimensional layered structure is generated. The features of the structure are similar with those in compounds 3,4,6,7,9,10-hexahydro-9-(4-methoxyphenyl)-1,8(2*H*,5*H*)-acridinedione [6].

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