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Crystal structure of 1-(5-(anthracen-9-yl)-3-(4-hydroxyphenyl)-4,5-dihydro-1H-pyrazol-1-yl)propan-1-one, C₂₆H₂₂N₂O₂

Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	0.30 × 0.20 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	36437, 9316, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5793
$N(\text{param})_{\text{refined}}$:	542
Programs:	Bruker programs [1], SHELX [2]

Source of materials

The compound were synthesized by using previously reported synthetic protocol [3, 4]. A mixture of 4-hydroxyacetophenone (1.24 g), 9-anthraldehyde (2.07 g), and 3 M of aqueous sodium hydroxide (6 mL) in ethanol (25 mL) was stirred at room temperature for 16 h. The resulting solid was filtered to afford 3-(anthracen-9-yl)-1-(4-hydroxyphenyl)prop-2-en-1-one (chalcone), which was used directly without further purification. Then chalcone (1.3 g) and hydrazine hydrate aqueous solution (3.6 g, 80%) were dissolved in 20 mL of propionic acid. The mixture was then stirred at 120 °C for 5 h, and the resulting solution was cooled to room temperature and slowly poured into a beaker containing ice water. The crude product was collected by filtration and recrystallized from ethyl acetate to give pure product as a yellow prism: 0.72 g, yield: 47.4%. Crystals were obtained by slow evaporation from methanol or ethanol at room temperature.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Anthracene derivatives represent a class of compounds which are interesting not only from chemical, but also because they are the subject of fluorescence materials research endeavors [5–7].

The title compound forms crystals, which crystallizes in the monoclinic and centrosymmetric space group $P2_1/c$

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Abstract

C₂₆H₂₂N₂O₂, monoclinic, $P2_1/c$ (no. 14), $a = 16.4367(8)$ Å, $b = 12.7804(6)$ Å, $c = 19.5474(8)$ Å, $\beta = 96.1700(14)^\circ$, $V = 4082.5(3)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0516$, $wR_{\text{ref}}(F^2) = 0.1195$, $T = 273(2)$ K.

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One of two crystallographically independent molecules of the title 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 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
N1	0.83917(8)	0.13858(11)	0.59979(7)	0.0465(4)
N2	0.77933(8)	0.12951(11)	0.64470(7)	0.0459(3)
N3	0.65103(8)	0.73234(11)	0.14754(7)	0.0467(4)
N4	0.72722(8)	0.71665(11)	0.12410(7)	0.0447(3)
O1	0.87738(8)	0.12885(11)	0.49473(7)	0.0671(4)
O2	0.62886(8)	0.15937(11)	0.93087(6)	0.0689(4)
H2B	0.5837	0.1839	0.9181	0.103*
O3	0.51753(8)	0.76191(13)	0.12497(7)	0.0811(5)
O4	1.10596(7)	0.61039(11)	0.13947(6)	0.0571(3)
H4B	1.1144	0.6174	0.0991	0.086*
C1	1.09639(11)	0.14725(14)	0.61092(9)	0.0527(5)
H1	1.0694	0.0866	0.6220	0.063*
C2	1.17656(12)	0.14154(16)	0.60175(10)	0.0582(5)
H2A	1.2034	0.0774	0.6064	0.070*
C3	1.21999(12)	0.23102(18)	0.58529(9)	0.0594(5)
H3	1.2751	0.2260	0.5789	0.071*
C4	1.18186(11)	0.32350(17)	0.57881(9)	0.0562(5)
H4A	1.2111	0.3824	0.5679	0.067*
C5	1.05867(12)	0.43008(14)	0.58304(9)	0.0526(5)
H5	1.0887	0.4889	0.5735	0.063*
C6	0.93818(15)	0.54208(15)	0.58610(10)	0.0654(6)
H6	0.9692	0.6007	0.5779	0.078*
C7	0.85827(15)	0.55339(16)	0.59260(11)	0.0713(6)
H7	0.8342	0.6192	0.5883	0.086*
C8	0.81124(13)	0.46612(15)	0.60586(10)	0.0650(5)
H8	0.7557	0.4746	0.6095	0.078*
C9	0.84482(12)	0.36954(14)	0.61349(9)	0.0549(5)
H9	0.8120	0.3136	0.6237	0.066*
C10	0.96757(10)	0.25233(12)	0.61305(8)	0.0411(4)
C11	1.05177(10)	0.24289(13)	0.60419(8)	0.0421(4)
C12	1.09730(11)	0.33374(14)	0.58824(8)	0.0457(4)
C13	0.97672(12)	0.44148(13)	0.59159(8)	0.0495(4)
C14	0.92918(11)	0.35113(13)	0.60625(8)	0.0435(4)
C15	0.81425(10)	0.13887(12)	0.70651(8)	0.0417(4)
C16	0.90488(10)	0.15718(13)	0.71098(9)	0.0464(4)
H16A	0.9347	0.1022	0.7370	0.056*
H16B	0.9195	0.2241	0.7322	0.056*
C17	0.92241(10)	0.15570(12)	0.63509(8)	0.0434(4)
H17	0.9557	0.0939	0.6278	0.052*
C18	0.82105(11)	0.12635(14)	0.53129(9)	0.0496(4)
C19	0.73302(11)	0.11357(16)	0.50366(9)	0.0570(5)
H19A	0.7282	0.1227	0.4541	0.068*
H19B	0.7015	0.1687	0.5225	0.068*
C20	0.69563(13)	0.00934(17)	0.51936(11)	0.0735(6)
H20A	0.6395	0.0077	0.4999	0.110*
H20B	0.7252	−0.0460	0.4998	0.110*
H20C	0.6985	0.0001	0.5683	0.110*
C21	0.76621(10)	0.13768(12)	0.76531(8)	0.0400(4)
C22	0.80286(11)	0.14361(13)	0.83266(9)	0.0491(4)
H22	0.8596	0.1447	0.8410	0.059*
C23	0.75635(11)	0.14798(14)	0.88733(9)	0.0534(5)
H23	0.7820	0.1502	0.9321	0.064*
C24	0.67232(11)	0.14906(13)	0.87607(9)	0.0486(4)
C25	0.63451(11)	0.14064(14)	0.80928(9)	0.0513(4)
H25	0.5777	0.1388	0.8012	0.062*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C26	0.68087(11)	0.13501(13)	0.75534(9)	0.0488(4)
H26	0.6548	0.1292	0.7108	0.059*
C27	0.70435(11)	0.91169(14)	0.24438(8)	0.0474(4)
H27	0.7303	0.8706	0.2140	0.057*
C28	0.73126(12)	1.01042(15)	0.25783(9)	0.0577(5)
H28	0.7768	1.0345	0.2381	0.069*
C29	0.69198(14)	1.07719(16)	0.30084(10)	0.0649(5)
H29	0.7110	1.1451	0.3090	0.078*
C30	0.62719(13)	1.04302(15)	0.33010(9)	0.0592(5)
H30	0.6003	1.0884	0.3573	0.071*
C31	0.53482(11)	0.90006(16)	0.35456(9)	0.0536(5)
H31	0.5083	0.9457	0.3818	0.064*
C32	0.44651(12)	0.75814(19)	0.38745(10)	0.0643(6)
H32	0.4206	0.8041	0.4149	0.077*
C33	0.42373(13)	0.6574(2)	0.38446(11)	0.0743(7)
H33	0.3826	0.6339	0.4098	0.089*
C34	0.46226(13)	0.5880(2)	0.34290(10)	0.0738(6)
H34	0.4468	0.5180	0.3414	0.089*
C35	0.52129(12)	0.62031(16)	0.30494(10)	0.0605(5)
H35	0.5453	0.5721	0.2776	0.073*
C36	0.60951(10)	0.76522(13)	0.26694(8)	0.0400(4)
C37	0.63706(10)	0.86916(13)	0.27561(8)	0.0409(4)
C38	0.59832(11)	0.93758(13)	0.32031(8)	0.0467(4)
C39	0.50957(10)	0.79642(16)	0.34945(8)	0.0483(4)
C40	0.54791(10)	0.72664(14)	0.30558(8)	0.0438(4)
C41	0.77571(10)	0.67656(12)	0.17310(8)	0.0399(4)
C42	0.73705(11)	0.66022(14)	0.23869(9)	0.0488(4)
H42A	0.7621	0.7048	0.2752	0.059*
H42B	0.7416	0.5878	0.2535	0.059*
C43	0.64712(10)	0.69097(14)	0.21834(8)	0.0451(4)
H43	0.6143	0.6268	0.2148	0.054*
C44	0.58568(12)	0.75854(16)	0.10392(9)	0.0573(5)
C45	0.59718(13)	0.7817(2)	0.03051(10)	0.0754(7)
H45A	0.6455	0.7452	0.0187	0.090*
H45B	0.5505	0.7548	0.0012	0.090*
C46	0.6061(2)	0.8932(3)	0.01638(15)	0.1258(11)
H46A	0.6132	0.9028	−0.0313	0.189*
H46B	0.5579	0.9298	0.0268	0.189*
H46C	0.6530	0.9202	0.0444	0.189*
C47	0.86062(10)	0.65380(12)	0.16339(8)	0.0388(4)
C48	0.88825(11)	0.66307(13)	0.09855(8)	0.0468(4)
H48	0.8511	0.6797	0.0608	0.056*
C49	0.96889(11)	0.64813(13)	0.08933(8)	0.0479(4)
H49	0.9860	0.6550	0.0457	0.057*
C50	1.02519(10)	0.62275(12)	0.14495(8)	0.0412(4)
C51	0.99824(10)	0.60886(13)	0.20897(8)	0.0453(4)
H51	1.0350	0.5886	0.2461	0.054*
C52	0.91710(10)	0.62489(12)	0.21798(8)	0.0435(4)
H52	0.8999	0.6162	0.2614	0.052*

with two independent molecules in the asymmetric unit. The molecules show twisted structures, in which the dihedral angles between the mean plane of the anthracene ring and the plane of the pyrazoline ring are 84.74° and 81.96°, respectively. Bond lengths are all in the expected ranges and in excellent accord with parameters derived from related

structure [8]. The two neighboring molecules with opposite chirality are connected with each other by $O-H \cdots O$ hydrogen bonds to form the 1D chain motifs. These achiral chains associate through $C-H \cdots \pi$ interactions between anthracene rings to construct the overall racemic 3D structure.

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