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The crystal structure of 1,5-di(naphthalen-2-yl)-3-(pyridin-2-yl)pentane-1,5-dione, $C_{30}H_{23}NO_2$

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Abstract

$C_{30}H_{23}NO_2$, orthorhombic, *Pbca* (no. 61), $a = 10.7674(16)$ Å, $b = 19.542(3)$ Å, $c = 20.970(3)$ Å, $V = 4412.5(11)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0350$, $wR_{\text{ref}}(F^2) = 0.0963$, $T = 153$ K.

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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:	Clear light colourless block
Size:	$0.50 \times 0.40 \times 0.30$ mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ :	0.63 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max} , completeness:	68.2° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	74683, 4022, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3738
$N(\text{param})_{\text{refined}}$:	298
Programs:	Olex2 [1], Bruker [2], SHELX [3]

Source of material

To the stirred mixture of 1-(naphthalen-2-yl)ethan-1-one (1.70 g, 0.01 mol), sodium hydroxide (0.60 g, 0.015 mol), ethanol (50 mL) and water (20 mL) was added a solution of picolinaldehyde (0.54 g, 5.0 mmol) in ethanol (10 mL) drop wisely. After 3 h, the solvent was evaporated under reduced pressure to get a yellow residue which was dissolved in the mixture of ethyl acetate (60 mL) and water (50 mL). The organic layer was separated, washed with water twice, dried by anhydrous sodium sulfate and then filtered. The solvent (ethylacetate) was evaporated to get a yellow solid which was purified by flash chromatography on silica gel (PE:EA = 30:1) to afford the title compound (a white solid, 0.92 g, yield 43%). Crystals were obtained by recrystallization with petroleum ether at room temperature.

Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2 [2]. The hydrogen atoms were placed at calculated positions as riding atoms.

Comment

With the advantages of unique pharmacological activity [4, 5], high catalytic activity [6], photoelectric properties [7], biological activity [8], simple operation and low cost, pyridine derivatives were widely used in the areas of pharmacy and chemical industry [9, 10]. Obviously, the synthesis of new pyridine derivatives have drawn attentions in recent decades. In this paper, a pyridine derivative containing two naphthalene units is reported, which was synthesized using picolinaldehyde by condensation reaction.

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	0.55551(9)	0.69014(5)	0.74005(4)	0.0444(2)
O2	0.55434(8)	0.56334(5)	0.91555(4)	0.0395(2)
N1	0.84556(11)	0.50708(5)	0.82866(5)	0.0397(3)
C1	0.69427(10)	0.59706(6)	0.80890(5)	0.0269(2)
H1	0.6627	0.6385	0.8317	0.032 [*]
C2	0.73538(10)	0.61899(6)	0.74177(5)	0.0288(2)
H2A	0.8161	0.6430	0.7448	0.035 [*]
H2B	0.7476	0.5777	0.7152	0.035 [*]
C3	0.64149(10)	0.66567(6)	0.70959(5)	0.0299(3)
C4	0.65393(10)	0.68280(6)	0.64039(5)	0.0286(2)
C5	0.57472(11)	0.73497(6)	0.61613(6)	0.0326(3)
H5	0.5196	0.7580	0.6442	0.039 [*]
C6	0.57668(11)	0.75230(6)	0.55330(6)	0.0335(3)
H6	0.5240	0.7877	0.5382	0.040 [*]
C7	0.65631(10)	0.71811(6)	0.51046(6)	0.0298(2)
C8	0.65880(11)	0.73441(6)	0.44434(6)	0.0352(3)
H8	0.6088	0.7707	0.4286	0.042 [*]
C9	0.73210(12)	0.69854(7)	0.40333(6)	0.0389(3)
H9	0.7321	0.7097	0.3592	0.047 [*]
C10	0.80813(12)	0.64494(7)	0.42588(6)	0.0387(3)
H10	0.8578	0.6197	0.3967	0.046 [*]
C11	0.81080(11)	0.62897(6)	0.48955(6)	0.0351(3)
H11	0.8636	0.5934	0.5044	0.042 [*]
C12	0.73518(10)	0.66523(6)	0.53332(5)	0.0294(2)
C13	0.73273(10)	0.64928(6)	0.59948(5)	0.0297(2)
H13	0.7865	0.6149	0.6156	0.036 [*]
C14	0.58785(10)	0.54514(6)	0.80445(5)	0.0285(2)
H14A	0.5244	0.5628	0.7745	0.034 [*]
H14B	0.6205	0.5018	0.7868	0.034 [*]
C15	0.52657(10)	0.53059(6)	0.86801(5)	0.0278(2)
C16	0.42883(10)	0.47645(6)	0.87155(5)	0.0263(2)
C17	0.37896(11)	0.44688(6)	0.81783(5)	0.0289(2)
H17	0.4099	0.4597	0.7771	0.035 [*]
C18	0.28251(11)	0.39779(6)	0.82188(5)	0.0292(2)
C19	0.22948(13)	0.36785(7)	0.76672(6)	0.0389(3)
H19	0.2579	0.3813	0.7257	0.047 [*]
C20	0.13778(13)	0.31976(7)	0.77196(6)	0.0419(3)
H20	0.1032	0.2999	0.7346	0.050 [*]
C21	0.09420(12)	0.29944(6)	0.83238(6)	0.0377(3)
H21	0.0308	0.2658	0.8355	0.045 [*]
C22	0.14256(11)	0.32788(6)	0.88649(6)	0.0337(3)
H22	0.1120	0.3141	0.9270	0.040 [*]
C23	0.23790(10)	0.37780(6)	0.88283(5)	0.0281(2)
C24	0.29232(11)	0.40802(6)	0.93761(5)	0.0303(3)
H24	0.2645	0.3946	0.9787	0.036 [*]
C25	0.38369(10)	0.45588(6)	0.93245(5)	0.0286(2)
H25	0.4177	0.4758	0.9699	0.034 [*]
C26	0.80269(10)	0.56841(6)	0.84664(5)	0.0261(2)
C27	0.85456(10)	0.60413(6)	0.89716(6)	0.0308(3)
H27	0.8228	0.6477	0.9088	0.037 [*]
C28	0.95278(11)	0.57594(7)	0.93037(6)	0.0345(3)
H28	0.9888	0.5997	0.9653	0.041 [*]
C29	0.99782(12)	0.51296(7)	0.91219(6)	0.0375(3)
H29	1.0654	0.4923	0.9340	0.045 [*]
C30	0.94162(14)	0.48089(7)	0.86126(6)	0.0454(3)
H30	0.9729	0.4375	0.8484	0.055 [*]

There is one crystallographically independent molecule (*cf.* the figure) in the asymmetric unit, in which all bond lengths are in normal ranges. The dihedral angle between two naphthalene rings is 89.63°. In molecular packing, nonclassical hydrogen bonds were observed as following: C14—H14B···N1 hydrogen bond (*d*(H14B···N1) = 2.58 Å, C17—H17···N1 hydrogen bond (*d*(H17···N1) = 2.50 Å, C25—H25···O2 hydrogen bond (*d*(H25···O2) = 2.54 Å).

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References

- Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Cryst.* **42** (2009) 339–341.
- Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
- Abdelaziz, M. E.; El-Miligy, M. M. M.; Fahmy, S. M.; Mahran, M. A.; Hazzaa, A. A.: Design, synthesis and docking study of pyridine and thieno[2,3-*b*] pyridine derivatives as anticancer PIM-1 kinase inhibitors. *Bioorg. Chem.* **80** (2018) 674–692.
- Shrestha, A.; Park, S.; Shin, S.; Kadayat, T. M.; Bist, G.; Katila, P.; Kwon, Y.; Lee, E. S.: Design, synthesis, biological evaluation, structure-activity relationship study, and mode of action of 2-phenol-4,6-dichlorophenyl-pyridines. *Bioorg. Chem.* **79** (2018) 1–18.
- Chen, J.-J.; Gan, Z.-L.; Huang, Q.; Yi, X.-Y.: Well-defined dinuclear silver phosphine complexes based on nitrogen donor ligand and their high efficient catalysis for A3-coupling reaction. *Inorg. Chim. Acta* **466** (2017) 93–99.
- Li, H.; Pang, M.; Wu, B.; Meng, J.: Synthesis, crystal structure and photochromism of a novel spiro[indoline–naphthalene] oxazine derivative. *J. Mol. Struct.* **1087** (2015) 73–79.
- Khojasteh, V.; Kakanejadifard, A.; Zabardasti, A.; Azarbani, F.: Spectral, structural, solvatochromism, biological and computational investigation of some new azoazomethines containing N-alkylpyridinium salts. *J. Mol. Struct.* **1175** (2019) 261–268.
- Xia, C.; Wang, X.; Lin, J.; Jiang, W.; Ni, Y.; Huang, W.: Organic light-emitting devices (OLED) based on new triphenylamine derivatives. *Synth. Met.* **159** (2009) 194–200.
- Natelson, D.; Yu, L. H.; Cizek, J. W.; Keane, Z. K.; Tour, J. M.: Single-molecule transistors: Electron transfer in the solid state. *Chem. Phys.* **324** (2006) 267–275.