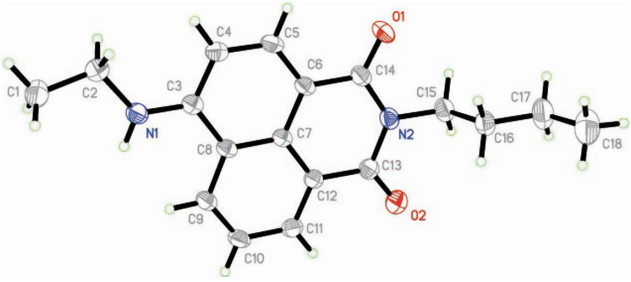
Peilian Liu, Fangru Jiang, Guiyuan Huang, Ying Sun and Liping Huang*

Crystal structure of 2-butyl-6-(ethylamino)-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione, $C_{18}H_{20}N_2O_2$



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Abstract

$C_{18}H_{20}N_2O_2$, monoclinic, $C2/c$ (no. 15), $a = 27.5693(13)$ Å, $b = 8.3005(4)$ Å, $c = 16.8797(8)$ Å, $\beta = 127.0225(12)°$, $V = 3084.0(3)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0642$, $wR_{ref}(F^2) = 0.2248$, $T = 293(2)$ K.

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The molecular 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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.24 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	31095, 3550, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2671
$N(param)_{refined}$:	201
Programs:	SHELX [1], Bruker programs [2]

Source of material

Synthesis of the title compound: 6-bromo-2-butyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione (3.32 g, 10 mmol), ethylamin (0.9 g, 20 mmol), 2-methoxyethan-1-ol (50 mL) was added to a 100 mL steel vessel. Then the mixture was reacted at 130 °C for 12 h. After cooled down to room temperature, the mixture was poured into water and then extracted with ethyl acetate three times. The combined organic phase was washed by brine three times. After concentrated under vacuum, the crude product was recrystallized in ethanol to afford the product as yellowish solid. Crystals of the title compound were grown from ethanol at room temperature.

Experimental details

The hydrogen atoms were placed in calculated positions and refined using a riding on attached atoms with isotropic thermal parameters 1.2 times those of their carrier atoms. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq}$(C). A minor disorder issue occurs at C18, which was not included in the refinement. This minor issue produces a difference electron density peak of 0.61 eÅ^{-1} near the C18 position and non-optimal R-factors.

Comment

Recently, organic conjugated molecular materials have become a research hotspot in the field of photoelectric molecular materials because of their wide application in photoluminescence [3–5], photoelectric devices [6, 7], field effect transistors [8, 9] and functional complexes [10]. 1,8-Naphthalimide derivatives are important organic conjugated molecules which can be used as fluorophores in the design of fluorescent probe [11] and prodrug [12]. The photophysical properties of 1,8-naphthalimide can be easily adjusted by varying the electron-donating/electron-withdrawing capability of the substituent at the 4th position of the naphthalic ring [13]. That is to say, the adsorption and emission of

***Corresponding author: Liping Huang,** School of Chemistry and Chemical Engineering, Key Laboratory of Clean Energy Materials Chemistry of Guangdong Higher Education Institutes, Lingnan Normal University, Zhanjiang, Guangdong 524048, P.R. China, e-mail: chemhxxxx@163.com. https://orcid.org/0000-0001-6708-688X
Peilian Liu, Fangru Jiang and Guiyuan Huang: School of Chemistry and Chemical Engineering, Key Laboratory of Clean Energy Materials Chemistry of Guangdong Higher Education Institutes, Lingnan Normal University, Zhanjiang, Guangdong 524048, P.R. China
Ying Sun: Guangdong Preschool Normal College in Maoming, Maoming, Guangdong 525200, P.R. China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.30031(7)	0.6297(2)	0.30484(11)	0.0686(5)
O2	0.45605(7)	0.8653(2)	0.32676(11)	0.0647(5)
N1	0.22534(7)	0.4389(2)	−0.12224(12)	0.0474(4)
H1	0.243974	0.462069	−0.147217	0.057*
N2	0.37735(7)	0.7497(2)	0.31439(11)	0.0479(4)
C8	0.30429(8)	0.5873(2)	0.02425(13)	0.0380(4)
C7	0.32833(7)	0.6424(2)	0.12118(12)	0.0365(4)
C14	0.32363(9)	0.6604(3)	0.26366(14)	0.0477(5)
C3	0.24894(8)	0.4942(2)	−0.03004(13)	0.0402(4)
C6	0.29836(8)	0.6073(2)	0.16359(13)	0.0408(4)
C12	0.38248(8)	0.7334(2)	0.17485(13)	0.0409(4)
C5	0.24489(9)	0.5204(2)	0.10866(15)	0.0468(5)
H5	0.224728	0.498626	0.136053	0.056*
C9	0.33562(9)	0.6288(2)	−0.01509(14)	0.0459(5)
H9	0.320540	0.594598	−0.078555	0.055*
C11	0.41153(9)	0.7712(3)	0.13367(15)	0.0484(5)
H11	0.447037	0.831904	0.169437	0.058*
C2	0.17041(9)	0.3427(2)	−0.18122(15)	0.0497(5)
H2A	0.136607	0.402436	−0.192208	0.060*
H2B	0.175468	0.244614	−0.145707	0.060*
C13	0.40872(9)	0.7890(2)	0.27621(14)	0.0466(5)
C4	0.22038(8)	0.4649(2)	0.01444(15)	0.0460(5)
H4	0.184224	0.406976	−0.020090	0.055*
C16	0.44929(10)	0.6748(3)	0.49171(15)	0.0569(5)
H16A	0.428294	0.572829	0.477405	0.068*
H16B	0.481230	0.659335	0.484264	0.068*
C10	0.38772(9)	0.7185(3)	0.03847(15)	0.0518(5)
H10	0.407466	0.744505	0.010895	0.062*
C1	0.15702(11)	0.3012(3)	−0.27853(17)	0.0651(6)
H1A	0.120117	0.239840	−0.317425	0.098*
H1B	0.189871	0.238747	−0.267571	0.098*
H1C	0.152461	0.398437	−0.313261	0.098*
C15	0.40485(11)	0.7990(3)	0.41703(15)	0.0572(5)
H15A	0.372998	0.815460	0.424585	0.069*
H15B	0.425833	0.900747	0.430410	0.069*
C17	0.47777(14)	0.7215(4)	0.59809(18)	0.0834(8)
H17A	0.445951	0.738580	0.605652	0.100*
H17B	0.499623	0.822184	0.613198	0.100*
C18	0.52110(18)	0.5936(5)	0.6709(2)	0.1059(11)
H18A	0.498885	0.496722	0.660393	0.159*
H18B	0.540558	0.631153	0.737466	0.159*
H18C	0.551286	0.571726	0.660978	0.159*

this fluorophore can be modulated readily. Therefore, 1,8-naphthalimide, as a single fluorophore, can furnish ratiometric fluorescence change conveniently. In our group continuing efforts to study the 1,8-naphthalimide derivatives go on. In this context, we report the crystal structure of the title compound.

In the molecule, O1—C14, O2—C13, N1—C3, N1—C2, N2—C14, N2—C13, N2—C15, C8—C7, C8—C7, C8—C3, C8—C9, C7—C6, C7—C12, C14—C6, C3—C4, C6—C5, C12—C11, C12—C13, C5—C4, C9—C10, C11—C10, C2—C1, C16—C15, C16—C17, and C17—C18 bond lengths are found to be 1.225(2) Å, 1.355(2) Å, 1.451(3) Å, 1.396(3) Å, 1.393(3) Å, 1.470(2) Å, 1.421(2) Å, 1.442(3) Å, 1.413(2) Å, 1.411(2) Å, 1.411(3) Å, 1.455(3) Å, 1.398(3) Å, 1.381(3) Å, 1.376(3) Å, 1.474(3) Å, 1.380(3) Å, 1.368(3) Å, 1.391(3) Å, 1.491(3) Å, 1.515(3) Å, 1.514(3) Å, and 1.516(4) Å, respectively, which are in the expected ranges [14]. The bond angles C3—N1—C2, C14—N2—C15, C13—N2—C14, C13—N2—C15, C7—C8—C3, C9—C8—C7, C9—C8—C3, C6—C7—C8, C12—C7—C8, C12—C7—C6, O1—C14—N2, O1—C14—C6, N2—C14—C6, N1—C3—C8, N1—C3—C4, C4—C3—C8, C7—C6—C14, C5—C6—C7, C5—C6—C14, C7—C12—C13, C11—C12—C7, C11—C12—C13, C4—C5—C6, C10—C9—C8, C12—C11—C10, N1—C2—C1, O2—C13—N2, O2—C13—C12, N2—C13—C12, C5—C4—C3, C17—C16—C15, C9—C10—C11, N2—C15—C16, and C16—C17—C18 are 123.74(16)°, 118.32(17)°, 124.42(16)°, 117.18(18)°, 119.04(15)°, 117.97(16)°, 122.98(16)°, 120.52(16)°, 119.41(15)°, 120.06(16)°, 119.22(18)°, 123.2(2)°, 117.56(16)°, 120.25(15)°, 121.42(18)°, 118.32(17)°, 120.60(18)°, 118.78(17)°, 120.62(16)°, 120.19(16)°, 120.69(17)°, 119.13(18)°, 122.15(16)°, 121.20(17)°, 119.90(18)°, 110.28(18)°, 119.85(18)°, 123.00(18)°, 117.14(17)°, 121.17(18)°, 113.22(19)°, 120.81(17)°, 112.07(17)°, 112.0(2)°, respectively. One dimensional chains are formed by intermolecular N1—H1···O1 hydrogen bonds. The chains extend through C9—H9···O1 contacts to form a two dimensional supramolecular layer.

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