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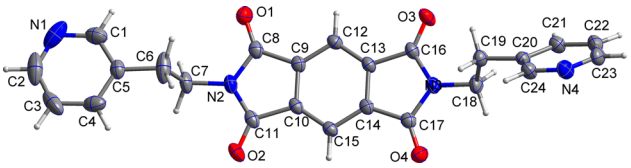
Crystal structure of 2,6-bis(2-(pyridin-3-yl)ethyl) pyrrolo[3,4-*f*]isoindole-1,3,5,7(2*H*,6*H*)-tetraone, $C_{24}H_{18}N_4O_4$



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Abstract

$C_{24}H_{18}N_4O_4$, monoclinic, $P2_1/c$ (no. 14), a = 16.5930(5) Å, b = 5.7298(2) Å, c = 20.8714(6) Å, β = 99.8620(10)°, V = 1955.02(11) Å^3, Z = 4, $R_{gt}(F)$ = 0.0415, $wR_{ref}(F^2)$ = 0.1115, T = 193 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.

Table 1: Data collection and handling.

Crystal:	Yellow plate
Size:	0.79 × 0.20 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	33246, 4504, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3667
$N(param)_{refined}$:	289
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

A mixture of pyromellitic dianhydride (0.218 g, 1 mmol) and 3-(2-aminoethyl)pyridine (0.234 mL, 2 mmol) in toluene (1 mL) and quinoline (3 mL) was heated at 473 K with stirring for 1 h. Upon cooling to room temperature, brown solid was precipitated and dark liquid was filtered off. The solid was successively washed with water, methanol, and diethyl ether and crude product was obtained. After dissolving in dichloromethane, the product was purified by column chromatography over silica gel eluting with a gradient of ethyl acetate 100% to give the light brown solid. Yield: 0.239 g (56%). A small amount of the title compound was dissolved in acetone and slow evaporation yielded yellow crystals. NMR data have been measured with a Bruker DRX300 spectrometer. IR measurements have been made with a Thermo Scientific Nicolet iS10 IR spectrometer at the Core–Facility Center for Photochemistry & Nanomaterials.

^{1}H NMR (300 MHz, ppm, DMSO-d$_6$, r.t.): 8.41 (d, 2H, H2, H23); 8.39 (d, 2H, H1, H24); 8.16 (s, 2H, H12, H15); 7.69–7.65 (m, 2H, H4, H21); 7.30–7.26 (d, 2H, H3, H22); 3.91–3.87 (t, 2H, H7, H18); 2.99–2.95 (t, 2H, H6, H19).

^{13}C NMR (75.4 MHz, ppm, DMSO-d$_6$, r.t.): 165.99, 149.78, 147.83, 136.82, 136.33, 123.49, 117.32, 44.59 and 30.73.

IR (n, cm^{-1}): 3455(m); 3095 (m); 3031 (m); 2965 (s); 2941 (s); 2847(s); 1761(s); 1700(s); 1576 (s).

Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–0.99 Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Comment

Pyromellitic diimide derivatives would be important compounds in terms of their optoelectronic and electrochemical properties

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.88225 (7)	0.1089 (2)	0.00346 (5)	0.0389 (3)
O2	0.80184 (7)	0.7719 (2)	−0.11129 (5)	0.0368 (3)
O3	0.68164 (7)	0.38724 (19)	0.20650 (5)	0.0372 (3)
O4	0.59838 (6)	1.04529 (19)	0.09029 (5)	0.0361 (3)
N1	1.13414 (10)	0.1282 (3)	−0.18454 (11)	0.0643 (5)
N2	0.85154 (7)	0.4217 (2)	−0.06601 (5)	0.0307 (3)
N3	0.63232 (7)	0.7386 (2)	0.16195 (5)	0.0280 (3)
N4	0.62521 (9)	1.3046 (2)	0.40612 (6)	0.0365 (3)
C1	1.08607 (11)	0.1844 (3)	−0.14137 (9)	0.0446 (4)
H1	1.0808	0.0744	−0.1083	0.054*
C2	1.14040 (12)	0.2881 (5)	−0.22962 (10)	0.0648 (7)
H2	1.1736	0.2532	−0.2612	0.078*
C3	1.10243 (12)	0.4969 (5)	−0.23317 (8)	0.0584 (6)
H3	1.1099	0.6060	−0.2659	0.070*
C4	1.05337 (10)	0.5494 (3)	−0.18930 (9)	0.0438 (4)
H4	1.0258	0.6952	−0.1915	0.053*
C5	1.04381 (8)	0.3914 (3)	−0.14193 (7)	0.0314 (3)
C6	0.99111 (11)	0.4391 (4)	−0.09156 (9)	0.0591 (6)
H6A	0.9912	0.6088	−0.0826	0.071*
H6B	1.0147	0.3588	−0.0506	0.071*
C7	0.90325 (9)	0.3574 (3)	−0.11305 (7)	0.0342 (3)
H7A	0.8810	0.4280	−0.1557	0.041*
H7B	0.9026	0.1857	−0.1184	0.041*
C8	0.84580 (8)	0.2889 (3)	−0.01128 (6)	0.0289 (3)
C9	0.78840 (8)	0.4176 (2)	0.02392 (6)	0.0253 (3)
C10	0.76340 (8)	0.6194 (2)	−0.01122 (6)	0.0253 (3)
C11	0.80505 (9)	0.6254 (3)	−0.06927 (6)	0.0285 (3)
C12	0.76313 (8)	0.3623 (2)	0.08202 (6)	0.0259 (3)
H12	0.7801	0.2244	0.1059	0.031*
C13	0.71116 (8)	0.5246 (2)	0.10233 (6)	0.0243 (3)
C14	0.68582 (8)	0.7257 (2)	0.06718 (6)	0.0247 (3)
C15	0.71022 (8)	0.7801 (3)	0.00862 (6)	0.0263 (3)
H15	0.6920	0.9158	−0.0158	0.032*
C16	0.67554 (8)	0.5295 (3)	0.16336 (6)	0.0268 (3)
C17	0.63349 (8)	0.8636 (3)	0.10483 (6)	0.0267 (3)
C18	0.59323 (9)	0.8217 (3)	0.21534 (7)	0.0335 (3)
H18A	0.5563	0.6994	0.2272	0.040*
H18B	0.5599	0.9617	0.2011	0.040*
C19	0.65698 (9)	0.8821 (3)	0.27433 (6)	0.0287 (3)
H19A	0.6873	0.7385	0.2899	0.034*
H19B	0.6966	0.9934	0.2609	0.034*
C20	0.62214 (8)	0.9872 (2)	0.32979 (6)	0.0250 (3)
C21	0.56593 (9)	0.8722 (3)	0.36054 (7)	0.0311 (3)
H21	0.5459	0.7229	0.3457	0.037*
C22	0.53917 (9)	0.9757 (3)	0.41287 (7)	0.0345 (3)
H22	0.4997	0.9005	0.4338	0.041*
C23	0.57066 (9)	1.1898 (3)	0.43421 (7)	0.0349 (3)
H23	0.5527	1.2589	0.4707	0.042*
C24	0.64907 (9)	1.2038 (3)	0.35471 (7)	0.0309 (3)
H24	0.6870	1.2859	0.3338	0.037*

[5]. In the extension of the study of pyromellitic diimide derivatives [6], the title compound was synthesized by the similar method for synthesizing *N*, *N*′-bis[3-(methylsulfanyl) propyl]-1,8:4,5-naphthalenetetracarboxylic diimide [7]. Similar to the previously reported compounds of *N*, *N*′-bis (2-pyridylethyl)pyromellitic diimide [8], this title compound also has heterocyclic groups separated by ethylene moieties. This ethylene connection allows free rotation of the terminal rings. All bond lengths and bond angles are normal and similar to those observed for similar crystal structures [8, 9].

In the crystal structure, the *m*-pyridyl groups on both sides of the central pyromellitic diimide plane are of a *syn* form. The dihedral angles between the mean planes of the central pyromellitic diimide ring [r.m.s. deviation = 0.0432 Å] and the terminal *m*-pyridyl groups are 7.84(8) and 56.66(4)°. Four C–H···N, C–H···O hydrogen bonds and four weak intermolecular $\pi\cdots\pi$ interactions further stabilize the crystal structure. The distances of each hydrogen bond are [C1–H1···O1^{i} = 2.41 Å, C15–H15···N4^{ii} = 2.42 Å, C19–H19···O2)^{iii} = 2.55 Å, C19–H19B···O3^{iv} = 2.52 Å for symmetry operations (i): 2 − *x*, −*y*, −*z*; (ii): *x*, 5/2 − *y*, −1/2 + *z* (iii): *x*, 3/2 − *y*, 1/2 + *z* and (iv): *x*, 1 + *y*, *z*] and the distances of each $\pi\cdots\pi$ interaction are [Cg(N2–C11)···Cg(N4–C24)^{v} = 3.7622 (9) Å, Cg(N3–C17)···Cg(N2–C2)^{vi} = 3.9723 (10) Å, Cg(N2–C2)···Cg(C9–C12)^{vii} = 3.7444(9) Å, Cg(N4–C24)···Cg(C9–C12)^{iii} = 3.8904(8) Å for symmetry operations (v): *x*, 3/2 − *y*, −1/2 + *z*; (vi): 2 − *x*, 1 − *y*, −*z*; and (vii): 2 − *x*, 1 − *y*, −*z*.].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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