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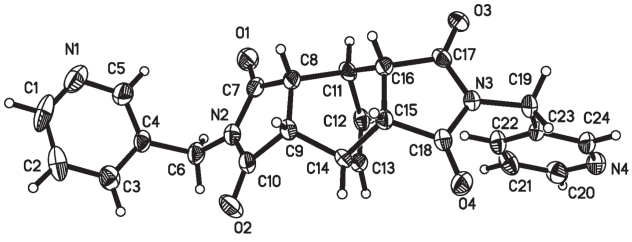
Crystal structure of 2,6-bis(3-methylpyridinyl) hexahydro-4,8-ethenopyrrolo-[3,4-*f*]isoindole-1,3,5,7(2*H*,6*H*)-tetrone, $C_{24}H_{20}N_4O_4$



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Abstract

$C_{24}H_{20}N_4O_4$, orthorhombic, $Pna2_1$ (no. 33), $a = 23.942(3)$ Å, $b = 12.366(3)$ Å, $c = 6.823(4)$ Å, $V = 2020.1(9)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0457$, $wR_{ref}(F^2) = 0.0963$, $T = 153$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A solution of 3-(aminomethyl)-pyridine and bicyclo[2.2.2]oct-7-ene-2,3,5,6-tetracarboxylic acid dianhydride in *N*,*N*-dimethylformamide was refluxed for 3 h, and then the crude product was isolated by filtration and recrystallized from methanol to yield the colourless title compound. Finally, the title compound was dissolved in a small amount of $CHCl_3$ at ambient temperature for a month to yield a colourless crystalline product.

Experimental details

All hydrogen atoms were placed geometrically on calculated positions using a riding model with the help of the SHELXL program (AFIX 43 option for aromatic H atoms, AFIX 13 for tertiary and AFIX 23 for secondary H atoms).

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.10×0.10×0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.98 cm^{-1}
Diffractometer, scan mode:	multiwire proportional, φ and ω scans
$2\theta_{max}$:	55.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10942, 4287
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2395
$N(param)_{refined}$:	289
Programs:	BRUKER SHELXTL [6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(16A)	4*a*	0.5565	0.5863	−0.0785	0.044
H(11A)	4*a*	0.5418	0.6680	−0.3978	0.051
H(15A)	4*a*	0.5462	0.6862	0.1883	0.046
H(14A)	4*a*	0.5311	0.8789	0.1443	0.049
H(8A)	4*a*	0.6264	0.7030	−0.2137	0.046
H(22A)	4*a*	0.3879	0.7416	−0.2220	0.061
H(13A)	4*a*	0.4744	0.9252	−0.1257	0.050
H(12)	4*a*	0.4765	0.8136	−0.4006	0.052
H(21A)	4*a*	0.3200	0.8353	−0.3915	0.04(1)
H(5A)	4*a*	0.7230	0.9351	−0.6219	0.077
H(6A)	4*a*	0.6264	1.0980	−0.3717	0.066
H(6B)	4*a*	0.6319	1.0254	−0.5592	0.066
H(19A)	4*a*	0.3670	0.5882	0.1898	0.064
H(19B)	4*a*	0.3750	0.5085	0.0136	0.064
H(24A)	4*a*	0.2676	0.5768	0.0445	0.064
H(3A)	4*a*	0.7101	1.1661	−0.2218	0.071
H(9A)	4*a*	0.6207	0.8013	0.0508	0.046
H(20A)	4*a*	0.2275	0.7945	−0.3390	0.071
H(1A)	4*a*	0.8580	1.0595	−0.4074	0.099
H(2A)	4*a*	0.8082	1.1739	−0.2118	0.100

Discussion

In recent years, bipyridyl derivatives were used as bridging ligands in various coordination polymers (CPs) which aroused considerable interests not only owing to their intriguing structures but also for their potential applications such as

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(3)	4*a*	0.4353(1)	0.6206(3)	0.0302(6)	0.032(2)	0.043(2)	0.047(2)	−0.008(2)	0.002(2)	−0.003(2)
O(3)	4*a*	0.4572(1)	0.5160(2)	−0.2394(6)	0.060(2)	0.054(2)	0.070(3)	−0.018(2)	0.006(2)	−0.024(2)
O(4)	4*a*	0.4366(1)	0.7428(2)	0.2854(5)	0.060(2)	0.073(2)	0.051(2)	−0.012(2)	0.018(2)	−0.015(2)
O(1)	4*a*	0.6239(2)	0.8156(3)	−0.5653(5)	0.074(2)	0.075(2)	0.041(2)	−0.013(2)	0.012(2)	−0.002(2)
C(16)	4*a*	0.5268(2)	0.6397(3)	−0.0989(6)	0.036(2)	0.036(2)	0.038(3)	0.002(2)	−0.004(2)	0.000(2)
C(7)	4*a*	0.6170(2)	0.8367(4)	−0.3938(8)	0.036(2)	0.054(3)	0.045(3)	−0.001(2)	0.006(3)	0.002(3)
N(2)	4*a*	0.6244(2)	0.9386(3)	−0.3105(6)	0.040(2)	0.042(2)	0.043(3)	−0.004(2)	0.004(2)	0.006(2)
C(4)	4*a*	0.7062(2)	1.0456(4)	−0.4175(8)	0.042(2)	0.046(3)	0.053(3)	−0.003(2)	0.006(3)	0.015(3)
C(10)	4*a*	0.6143(2)	0.9408(4)	−0.1121(8)	0.041(3)	0.051(3)	0.049(4)	−0.008(2)	0.007(3)	0.001(3)
O(2)	4*a*	0.6187(2)	1.0210(3)	−0.0124(6)	0.101(3)	0.053(2)	0.064(3)	−0.030(2)	0.012(2)	−0.014(2)
C(11)	4*a*	0.5408(2)	0.7107(3)	−0.2768(7)	0.042(2)	0.040(2)	0.045(3)	−0.007(2)	−0.005(2)	−0.006(2)
C(17)	4*a*	0.4705(2)	0.5826(3)	−0.1123(8)	0.044(3)	0.033(2)	0.056(3)	−0.003(2)	−0.005(3)	0.000(3)
C(18)	4*a*	0.4603(2)	0.6968(3)	0.1487(7)	0.047(3)	0.041(2)	0.047(3)	−0.007(2)	0.003(3)	0.003(2)
N(4)	4*a*	0.2382(2)	0.6820(3)	−0.1418(7)	0.043(2)	0.066(3)	0.075(3)	−0.006(2)	0.000(3)	−0.015(3)
C(23)	4*a*	0.3355(2)	0.6491(3)	−0.0677(7)	0.034(2)	0.043(2)	0.051(3)	−0.006(2)	0.002(2)	−0.005(2)
C(15)	4*a*	0.5209(2)	0.7116(3)	0.0851(7)	0.035(2)	0.041(2)	0.039(3)	−0.004(2)	−0.004(2)	0.003(2)
C(14)	4*a*	0.5348(2)	0.8308(3)	0.0309(7)	0.044(2)	0.034(2)	0.044(3)	−0.007(2)	0.002(2)	−0.006(2)
C(8)	4*a*	0.5987(2)	0.7604(3)	−0.2315(7)	0.033(2)	0.039(2)	0.043(3)	0.003(2)	−0.004(3)	0.003(2)
C(22)	4*a*	0.3503(2)	0.7264(4)	−0.2004(8)	0.037(2)	0.061(3)	0.054(3)	−0.014(2)	−0.007(3)	0.001(3)
C(13)	4*a*	0.4973(2)	0.8646(3)	−0.1337(7)	0.033(2)	0.037(2)	0.054(3)	−0.001(2)	0.004(2)	0.006(3)
C(12)	4*a*	0.4990(2)	0.8023(3)	−0.2913(8)	0.032(2)	0.052(3)	0.046(3)	−0.009(2)	−0.009(2)	0.011(3)
C(21)	4*a*	0.3101(2)	0.7819(4)	−0.3020(9)	0.055(3)	0.056(3)	0.071(4)	−0.014(2)	−0.010(3)	0.006(3)
C(5)	4*a*	0.7402(2)	0.9830(4)	−0.5359(9)	0.055(3)	0.064(3)	0.074(4)	−0.002(3)	0.020(3)	0.006(3)
C(6)	4*a*	0.6437(2)	1.0332(3)	−0.4240(8)	0.045(3)	0.048(3)	0.070(4)	−0.002(2)	0.008(3)	0.017(3)
C(19)	4*a*	0.3773(2)	0.5838(3)	0.0526(8)	0.043(2)	0.051(3)	0.068(4)	−0.014(2)	0.008(3)	0.000(3)
C(24)	4*a*	0.2784(2)	0.6297(4)	−0.0446(8)	0.044(3)	0.056(3)	0.060(4)	−0.008(2)	0.000(3)	−0.004(3)
C(3)	4*a*	0.7315(2)	1.1197(4)	−0.2986(8)	0.068(3)	0.063(3)	0.046(3)	−0.016(3)	0.004(3)	0.011(3)
C(9)	4*a*	0.5953(2)	0.8297(3)	−0.0493(7)	0.034(2)	0.041(2)	0.040(3)	−0.007(2)	−0.007(2)	0.004(2)
C(20)	4*a*	0.2546(2)	0.7570(4)	−0.2690(9)	0.048(3)	0.063(3)	0.066(4)	−0.003(2)	−0.006(3)	−0.004(3)
C(1)	4*a*	0.8192(2)	1.0568(6)	−0.413(1)	0.037(3)	0.126(6)	0.083(5)	−0.006(4)	0.002(4)	0.053(5)
N(1)	4*a*	0.7960(2)	0.9865(4)	−0.5354(9)	0.060(3)	0.096(4)	0.083(4)	0.013(3)	0.025(3)	0.024(3)
C(2)	4*a*	0.7900(3)	1.1252(5)	−0.294(1)	0.068(4)	0.101(5)	0.082(5)	−0.042(4)	−0.007(4)	0.032(4)

porosity [1], catalysts [2], nonlinear optics [3]. The title compound have been used for the synthesis of some metal complexes [4, 5]. In the title crystal structure one molecule is present in the asymmetric unit. The backbone of the structure is essentially in a conformation. The bond angles of C4–C6–N2 and C23–C19–N3 are 112.1(4)° and 113.6(4)° respectively, which is in accord to similar metal compounds.

References

1. Jiang, J. J.; Liu, Y. R.; Yang, R.; Pan, M.; Cao, R.; Su, C. Y.: The interplay of coordinative and hydrogen-bonding in directing the [M(4,4′-bpy)$_2$(H$_2$O)$_2$] square-grid networks: formation of 3D porous framework [Cd(4,4′-bpy)$_2$(H$_2$O)$_2$](ClO$_4$)$_2$(4,4′-bpy)(CH$_3$OH)$_2$. CrystEngComm **10** (2008) 1147–1153.
2. Li, W.; Xie, J. H.; Yuan, M. L.; Zhou, Q. L.: Ruthenium complexes of tetradentate bipyridine ligands: highly efficient catalysts for the hydrogenation of carboxylic esters and lactones. Green Chem. **16** (2014) 4081–4085.
3. Kuppler, R. J.; Timmons, D. J.; Fang, Q.-R.; Li, J.-R.; Makala, T. A.; Young, M. D.; Yuan, D.; Zhao, D.; Zhuang, W.; Zhou, H.-C.: Potential applications of metal-organic frameworks. Coord. Chem. Rev. **253** (2009) 3042–3066.
4. Liu, Z. M.; Liu,Y.; Zheng, S. R.; Yu, Z. Q.; Pan, M.; Su, C. Y.: Assembly of trigonal and tetragonal prismatic cages from octahedral metal ions and a flexible molecular clip. Inorg. Chem. **46** (2007) 5814–5816.
5. Yu, Z. Q.; Pan, M.; Jiang, J. J.; Liu, Z. M.; Su, C. Y.: Anion modulated structural diversification in the assembly of Cd(II) complexes based on a balance-like dipodal ligand. Cryst. Growth Des. **12** (2012) 2389–2396.
6. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.