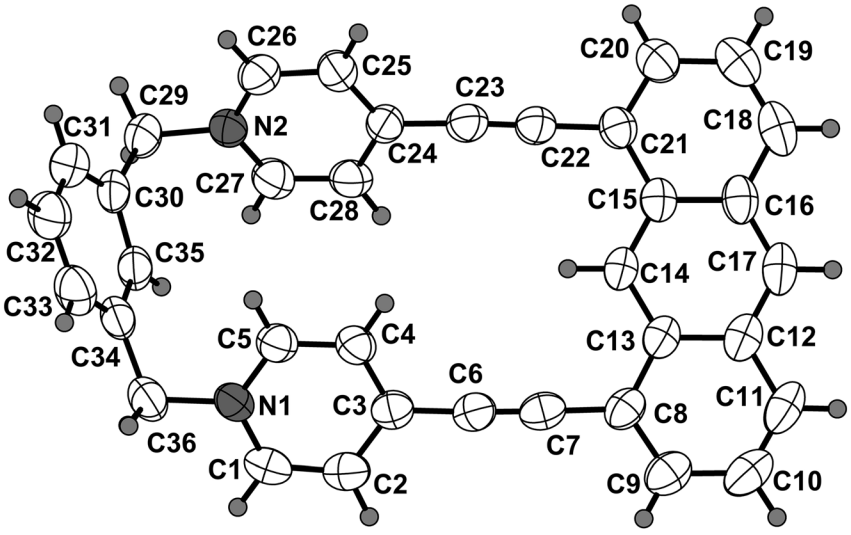
Xia Hu and Fei Zeng*

The crystal structure of 1,3(4,1)-dipyridin-1-iuma-2(1,8)-diethynylanthracena-5(1,3)-benzenacyclohexaphane-11,31-diium bis(hexafluoridophosphate), $C_{36}H_{24}F_{12}N_2P_2$



https://doi.org/10.1515/ncrs-2022-0080
Received February 18, 2022; accepted March 29, 2022; published online April 6, 2022

Abstract

$C_{36}H_{24}F_{12}N_2P_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.218(7) Å, b = 10.384(9) Å, c = 19.908(17) Å, α = 96.402(12)°, β = 91.216(12)°, γ = 101.161(12)°, V = 1655(2) Å^3, Z = 2, $R_{gt}(F)$ = 0.0685, $wR_{ref}(F^2)$ = 0.2188, T = 296(2) K.

CCDC no.: 2151727

The molecular structure is shown in the figure (The anions are omitted for clarity). 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 prism
Size:	0.45 × 0.26 × 0.21 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.23 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.5°, 97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18688, 7338, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3668
$N(param)_{refined}$:	506
Programs:	Bruker [1], SHELX [2]

Source of material

1,8-Diiodoanthracene (430 mg, 1 mmol) was placed in a 50 ml Schlenk flask along with 4-ethynylpyridine hydrochloride (278 mg, 2 mmol), $(Ph_3P)2–PdCl_2$ (10 mg), and CuI (2 mg). Diethylamine (40 ml) was distilled under nitrogen directly into the reaction flask, and the reaction was stirred in the dark at room temperature for 2 days. The solvent was then removed *in vacuo*; the residue was redissolved in dichloromethane, washed with water, and then washed with brine. The organic fraction was dried with Na_2SO_4,

***Corresponding author: Fei Zeng**, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou Hunan 425199, People's Republic of China,
E-mail: zengfei@iccas.ac.cn. https://orcid.org/0000-0002-1555-1812
Xia Hu, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou Hunan 425199, People's Republic of China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.1815 (5)	−0.0588 (4)	0.2657 (2)	0.0562 (10)
H1	−0.2025	−0.1504	0.2565	0.067*
C2	−0.1381 (5)	−0.0014 (4)	0.3302 (2)	0.0573 (10)
H2	−0.1322	−0.0543	0.3646	0.069*
C3	−0.1028 (4)	0.1348 (4)	0.34494 (19)	0.0493 (9)
C4	−0.1211 (5)	0.2088 (4)	0.29208 (19)	0.0517 (9)
H4	−0.1006	0.3006	0.3001	0.062*
C5	−0.1691 (5)	0.1472 (4)	0.22844 (19)	0.0517 (9)
H5	−0.1845	0.1973	0.1938	0.062*
C6	−0.0472 (5)	0.1979 (4)	0.4108 (2)	0.0531 (9)
C7	0.0042 (5)	0.2539 (4)	0.4648 (2)	0.0518 (9)
C8	0.0691 (5)	0.3219 (4)	0.52996 (18)	0.0500 (9)
C9	0.0111 (5)	0.2755 (4)	0.5882 (2)	0.0614 (11)
H9	−0.0717	0.1999	0.5856	0.074*
C10	0.0733 (6)	0.3387 (5)	0.6514 (2)	0.0697 (12)
H10	0.0314	0.3053	0.6904	0.084*
C11	0.1945 (6)	0.4486 (5)	0.65651 (19)	0.0655 (12)
H11	0.2375	0.4876	0.6992	0.079*
C12	0.2579 (5)	0.5063 (4)	0.59814 (18)	0.0521 (10)
C13	0.1938 (4)	0.4428 (3)	0.53255 (17)	0.0468 (9)
C14	0.2503 (4)	0.4993 (3)	0.47524 (17)	0.0461 (9)
H14	0.2105	0.4572	0.4327	0.055*
C15	0.3657 (4)	0.6182 (3)	0.48018 (17)	0.0464 (9)
C16	0.4309 (5)	0.6825 (4)	0.54476 (19)	0.0515 (9)
C17	0.3759 (5)	0.6229 (4)	0.60222 (19)	0.0583 (11)
H17	0.4200	0.6628	0.6446	0.070*
C18	0.5472 (5)	0.8051 (4)	0.5490 (2)	0.0664 (12)
H18	0.5928	0.8458	0.5911	0.080*
C19	0.5917 (5)	0.8627 (4)	0.4928 (2)	0.0715 (12)
H19	0.6658	0.9434	0.4966	0.086*
C20	0.5272 (5)	0.8017 (4)	0.4286 (2)	0.0613 (11)
H20	0.5581	0.8433	0.3905	0.074*
C21	0.4198 (4)	0.6822 (3)	0.42130 (18)	0.0481 (9)
C22	0.3653 (4)	0.6180 (4)	0.35493 (19)	0.0500 (9)
C23	0.3275 (5)	0.5637 (4)	0.2996 (2)	0.0526 (9)
C24	0.2968 (4)	0.4883 (4)	0.23359 (18)	0.0483 (9)
C25	0.2496 (5)	0.5404 (4)	0.17684 (19)	0.0547 (10)
H25	0.2342	0.6272	0.1803	0.066*
C26	0.2257 (5)	0.4632 (4)	0.11568 (19)	0.0567 (10)
H26	0.1913	0.4971	0.0777	0.068*
C27	0.2996 (5)	0.2876 (4)	0.1643 (2)	0.0576 (10)
H27	0.3190	0.2018	0.1595	0.069*
C28	0.3205 (5)	0.3588 (4)	0.2261 (2)	0.0600 (11)
H28	0.3507	0.3211	0.2635	0.072*
C29	0.2135 (5)	0.2514 (4)	0.04492 (18)	0.0617 (11)
H29A	0.2864	0.1880	0.0407	0.074*
H29B	0.2318	0.3044	0.0076	0.074*
C30	0.0332 (5)	0.1786 (4)	0.04215 (18)	0.0537 (10)
C31	−0.0839 (6)	0.2085 (4)	−0.0020 (2)	0.0690 (12)
H31	−0.0509	0.2676	−0.0332	0.083*
C32	−0.2487 (6)	0.1507 (5)	0.0007 (2)	0.0775 (14)
H32	−0.3258	0.1676	−0.0303	0.093*
C33	−0.3003 (6)	0.0682 (4)	0.0485 (2)	0.0710 (12)
H33	−0.4130	0.0350	0.0519	0.085*
C34	−0.1849 (5)	0.0339 (4)	0.09222 (19)	0.0542 (10)
C35	−0.0180 (5)	0.0880 (4)	0.08718 (18)	0.0536 (10)
H35	0.0608	0.0629	0.1145	0.064*
C36	−0.2390 (5)	−0.0545 (4)	0.1457 (2)	0.0648 (11)
H36A	−0.3584	−0.0854	0.1410	0.078*
H36B	−0.1873	−0.1311	0.1391	0.078*
F1[a]	0.2832 (10)	0.9454 (8)	0.3227 (3)	0.098 (2)
F1[b]	0.2315 (18)	0.8942 (15)	0.3036 (8)	0.098 (5)
F2[a]	0.1615 (6)	0.8405 (5)	0.2278 (5)	0.090 (2)
F2[b]	0.1910 (19)	0.8684 (15)	0.1931 (10)	0.156 (7)
F3[a]	0.3449 (12)	0.9779 (8)	0.1682 (3)	0.094 (2)
F3[b]	0.414 (2)	1.0305 (16)	0.1872 (9)	0.104 (5)
F4[a]	0.4660 (6)	1.0777 (5)	0.2647 (5)	0.089 (2)
F4[b]	0.4495 (14)	1.0532 (12)	0.2913 (9)	0.152 (7)
F5	0.1976 (3)	1.0624 (3)	0.24873 (16)	0.1047 (10)
F6	0.4341 (3)	0.8566 (3)	0.23969 (18)	0.1096 (10)
F7	0.6519 (4)	0.5371 (4)	0.18786 (14)	0.1268 (12)
F8	0.6138 (4)	0.3961 (4)	0.0933 (2)	0.1431 (14)
F9	0.8512 (4)	0.4989 (3)	0.05664 (14)	0.1058 (10)
F10	0.8908 (4)	0.6373 (3)	0.15032 (19)	0.1279 (13)
F11	0.6547 (4)	0.6122 (4)	0.08992 (16)	0.1218 (11)
F12	0.8492 (4)	0.4213 (3)	0.15544 (19)	0.1308 (13)
N1	−0.1944 (4)	0.0143 (3)	0.21560 (15)	0.0485 (7)
N2	0.2518 (4)	0.3378 (3)	0.11026 (15)	0.0493 (8)
P1	0.31585 (13)	0.95909 (10)	0.24530 (5)	0.0541 (3)
P2	0.75253 (14)	0.51553 (12)	0.12302 (6)	0.0644 (4)

[a]Occupancy: 0.641 (16), [b]Occupancy: 0.359 (16).

filtered through a silica gel plug, and then the solvent was removed on a rotary evaporator to produce 1,8-bis(4-pyridylethynyl)anthracene as a yellow solid. A mixture of 1,8-bis(4-pyridylethynyl)anthracene (380 mg, 1.0 mmol) and 1,3-bis(bromomethyl)benzene (262 mg, 1.0 mmol) in CH_3CN (300 ml) was stirred for 24 h at reflux under nitrogen atmosphere. Then, the mixture was filtrated, washed with acetone, and dried *in vacuo* and by protonation the title compound was obtained (635.1 mg, 0.82 mmol) as a yellow solid. Crystals of the title compound were obtained by slow vapor diffusion of ether to a solution of title compound in CH_3CN within 1 weeks.

Experimental details

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

Comment

Efficient synthesis of novel macrocyclic hosts with unique structures and good host–guest properties is a permanent and challenging topic in the field of supramolecular chemistry [3–5]. During the last decade, considerable effort has been devoted to the development of macrocyclic molecular and a number of new macrocyclic receptors with novel properties have been reported, such as pillar[*n*]arene [6, 7], Ex-box [8], and others [9]. These approaches often suffer from long synthetic steps and low yields, which restrict their further application in the complicated supramolecular self-assembly. Recently, we reported the high yield synthesis of a novel good water-soluble macrocycle containing two pyridinium moieties [10].

The title compound will enriched the toolbox of supramolecular chemists. The single-crystal structure verifies that all bond lengths are in normal ranges.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The work was supported by National Natural Science Foundation of China No. 21602055; Natural Science Foundation of Hunan Province No. 2017 J J3094 and Undergraduate Research Study and Innovative Experiment of Hunan Provincial (2016–636).

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

References

1. Bruker. APEX2, SAINT and SADABS; Bruker AXS Inc.: Madison, WI, USA, 2009.
2. Sheldrick G. M. SHELXTL – Integrated space-group and crystal-structure determination. *Acta Crystallogr*. 2015, *A71*, 3–8.
3. Chen C.-F. Novel triptycene-derived hosts: synthesis and their applications in supramolecular chemistry. *Chem. Commun.* 2011, *47*, 1674–1688.
4. Ogoshi T., Yamagichi T. A., Nakamoto Y. Pillar-shaped macrocyclic hosts pillar[*n*]arenes: new key players for supramolecular chemistry. *Chem. Rev.* 2016, *116*, 7937–8002.
5. Yu G., Jie K., Huang F. Supramolecular amphiphiles based on host-guest molecular recognition motifs. *Chem. Rev.* 2015, *115*, 7240–7307.
6. Ogoshi T., Kanai S., Fujinami S., Yamagishi T. A. para-Bridged symmetrical pillar[5]arenes: their Lewis acid catalyzed synthesis and host-guest property. *J. Am. Chem. Soc.* 2008, *130*, 5022–5023.
7. Xue M., Yang Y., Chi X. D., Zhang Z. B., Huang F. Pillararenes, a new class of macrocycles for supramolecular chemistry. *Acc. Chem. Res.* 2012, *45*, 1294–1308.
8. Barnes J. C., Juríček M., Strutt N. L., Frasconi M., Sampath S., Giesener M. A., McGrier P. L., Bruns C. J., Stern C. L., Sarjeant A. A., Stoddart J. F. ExBox: a polycyclic aromatic hydrocarbon scavenger. *J. Am. Chem. Soc.* 2013, *135*, 183–192.
9. Chen Y., Qian C., Zhao Q., Cheng M., Dong X., Zhao Y., Jiang J., Wang L. Adjustable chiral self-sorting and self-discriminating behaviour between diamond-like Tröger's base-linked cryptands. *Chem. Commun.* 2019, *55*, 8072–8075.
10. Ding M.-H., Liao J., Tang L.-L., Ou G.-C., Zeng F. High-yield synthesis of a novel water-soluble macrocycle for selective recognition of naphthalene. *Chin. Chem. Lett.* 2021, *32*, 1665–1668.