Lingfan Zhang and Man-Hua Ding*

The crystal structure of 1,8-bis(pyridin-4-ylethynyl)anthracene-1,2,4,5-tetrafluoro-3,6-diiodobenzene (2/1), $C_{62}H_{32}F_4I_2N_4$

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Abstract

$C_{62}H_{32}F_4I_2N_4$, triclinic, $P\bar{1}$ (no. 2), $a = 10.201(9)$ Å, $b = 10.897(9)$ Å, $c = 13.499(12)$ Å, $\alpha = 106.740(9)^\circ$, $\beta = 98.981(9)^\circ$, $\gamma = 115.198(9)^\circ$, $V = 1231.1(18)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0675$, $wR_{ref}(F^2) = 0.0645$, $T = 296(2)$ K.

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The molecular structure is shown in the figure (The asymmetric unit is labelled). 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:	$0.42 \times 0.36 \times 0.23$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.34 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11786, 4317, 0.036
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3920
$N(\text{param})_{\text{refined}}$:	325
Programs:	Bruker [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.16536(2)	0.83169(2)	0.77971(2)	0.06046(8)
F1	0.36054(18)	1.15645(16)	0.97117(13)	0.0677(4)
F2	0.38611(18)	0.71614(15)	0.86054(12)	0.0659(4)
N1	0.8650(3)	0.6543(3)	0.6110(2)	0.0788(7)
N2	1.0337(4)	0.3256(4)	0.7559(3)	0.1050(11)
C1	0.7440(4)	0.5694(3)	0.6318(2)	0.0750(8)
H1	0.7423	0.5987	0.7032	0.090*
C2	0.6205(3)	0.4406(3)	0.5545(2)	0.0696(7)
H2	0.5379	0.3854	0.5736	0.084*
C3	0.6209(3)	0.3942(3)	0.4478(2)	0.0536(6)
C4	0.7459(3)	0.4819(3)	0.4249(2)	0.0749(8)
H4	0.7511	0.4556	0.3543	0.090*

*Corresponding author: Man-Hua Ding, Department of Biology and Chemistry, Hunan University of Science and Engineering, Key Laboratory Comprehensive Utilization of Dominant Plants Resources in South Hunan, Yongzhou Hunan 425199, P.R. China, e-mail: 42979930@qq.com

Lingfan Zhang: Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou Hunan 425199, P.R. China

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C5	0.8639(4)	0.6101(4)	0.5089(3)	0.0914(11)
H5	0.9476	0.6687	0.4924	0.110*
C6	0.4948(3)	0.2604(3)	0.3640(2)	0.0598(6)
C7	0.3914(3)	0.1505(3)	0.2926(2)	0.0554(6)
C8	0.2738(3)	0.0166(2)	0.2027(2)	0.0518(5)
C9	0.1474(3)	0.0114(3)	0.1459(2)	0.0653(7)
H9	0.1341	0.0932	0.1677	0.078*
C10	0.0364(3)	−0.1176(3)	0.0540(2)	0.0701(8)
H10	−0.0513	−0.1212	0.0176	0.084*
C11	0.0561(3)	−0.2359(3)	0.0184(2)	0.0632(7)
H11	−0.0159	−0.3175	−0.0445	0.076*
C12	0.1846(3)	−0.2384(2)	0.07492(19)	0.0507(5)
C13	0.2057(3)	−0.3595(3)	0.0415(2)	0.0571(6)
H13	0.1379	−0.4396	−0.0234	0.069*
C14	0.3251(3)	−0.3648(3)	0.1021(2)	0.0555(6)
C15	0.4293(3)	−0.2408(2)	0.20265(19)	0.0484(5)
C16	0.4128(3)	−0.1160(2)	0.23321(18)	0.0468(5)
H16	0.4833	−0.0340	0.2964	0.056*
C17	0.2941(3)	−0.1105(2)	0.17208(18)	0.0464(5)
C18	0.3466(4)	−0.4897(3)	0.0697(3)	0.0739(8)
H18	0.2817	−0.5692	0.0037	0.089*
C19	0.4581(4)	−0.4950(3)	0.1322(3)	0.0844(9)
H19	0.4698	−0.5777	0.1090	0.101*
C20	0.5579(4)	−0.3766(3)	0.2327(3)	0.0758(8)
H20	0.6332	−0.3833	0.2759	0.091*
C21	0.5459(3)	−0.2515(3)	0.2681(2)	0.0576(6)
C22	0.6467(3)	−0.1326(3)	0.3715(2)	0.0593(6)
C23	0.7273(3)	−0.0343(3)	0.4583(2)	0.0637(7)
C24	0.8289(3)	0.0867(3)	0.5603(2)	0.0640(7)
C25	0.8165(4)	0.2129(4)	0.5962(3)	0.0823(9)
H25	0.7398	0.2205	0.5557	0.099*
C26	0.9222(6)	0.3279(4)	0.6946(3)	0.1035(13)
H26	0.9131	0.4120	0.7184	0.124*
C27	1.0416(4)	0.2026(5)	0.7217(3)	0.0967(12)
H27	1.1178	0.1976	0.7651	0.116*
C28	0.9439(3)	0.0821(4)	0.6259(3)	0.0774(8)
H28	0.9551	−0.0011	0.6056	0.093*
C29	0.3666(3)	0.9329(2)	0.91049(18)	0.0473(5)
C30	0.4283(3)	1.0770(2)	0.9843(2)	0.0489(5)
C31	0.4411(3)	0.8580(2)	0.92884(19)	0.0479(5)

Source of material

1,8-Diiodoanthracene (430 mg, 1 mmol) was placed in a 50 mL flask along with 4-ethynylpyridine hydrochloride (278 mg, 2 mmol), (Ph₃P)₂-PdCl₂ (10 mg), and CuI (2 mg). Diethylamine (40 mL) was distilled under nitrogen directly into the reaction, and the mixture was stirred in the dark at room temperature for 2 days. The solvent was removed *in vacuo*; the residue was dissolved in dichloromethane, washed with water, and then washed with brine. The organic fraction was dried with Na₂SO₄, filtered through a silica gel plug, and the solvent was removed on a rotary evaporator to produce 1,8-bis(4-pyridylethynyl)anthracene as a yellow solid. 1,4-Diiodotetrafluorobenzene (0.1 mmol) was added to the 1,8-diiodoanthracene (0.1 mmol) in chloroform giving a yellow solution. Crystals of the title compound were obtained by slow evaporation within 1 week.

Experimental details

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

Discussion

Comment

The halogen bond is an attractive non-covalent interaction between a polarized halogen atom (donor) and a Lewis base (acceptor) [3]. Halogen bonding was successfully used to construct three-dimensional supramolecular networks [4, 5] and halogen-bonded liquid crystals [6] that show enhanced stability and enable control over mesophase formation by varying the degree of fluorination accompanied by a change of the halogen bond strength. Clarke et al. [7] describe the cocrystallisation of the halogen bond donor 1,4-diiodotetrafluorobenzene (tfib) and the halogen bond acceptor 4,4'-bipyridyl (bipy) on a graphite surface. Resnati et al. [8] reported the terpyridine self-assembling with tfib. Tiekink et al. [9] describe the formation of 1:1 complex between tfib and 4-(pyridin-4-ylsulfanyl)pyridine *via* halogen bond interactions. 1,8-bis(4-pyridylethynyl)anthracene, which looks like a molecule clip, has been used as donor building block to prepare supramolecular boxes using the rigid u-shaped anthracene and anthracene-9,10-dione building blocks bearing two 3,5-lutidine acceptors in 1 and 8 position [10, 11].

Herein, we reported the synthesis of 2:1 supramolecule complexes 1,4-diiodotetrafluorobenzene and 1,8-bis(4-pyridylethynyl)anthracene through halogen bond interactions. The single crystal structure verifies that all bond lengths are in normal ranges.

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