Juan Liao, Fei Zeng and Man-Hua Ding*

The crystal structure of 1-benzyl-2-((4-(tert-butyl)phenyl)ethynyl)pyridin-1-ium bromide, $C_{24}H_{24}BrN$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.26 × 0.22 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.97 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.5°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	20663, 4052, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3368
$N(\text{param})_{\text{refined}}$:	238
Programs:	Bruker [1], SHELX [2]

<https://doi.org/10.1515/ncrs-2019-0638>

Received August 29, 2019; accepted October 16, 2019; available online October 25, 2019

Abstract

$C_{24}H_{24}BrN$, orthorhombic, $P2_1/n$ (no. 14), $a = 9.875(6)$ Å, $b = 10.415(6)$ Å, $c = 20.562(12)$ Å, $\beta = 98.130(7)^\circ$, $V = 2094(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0367$, $wR_{\text{ref}}(F^2) = 0.1044$, $T = 273(2)$ K.

CCDC no.: 1905004

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.

Source of material

2-((4-(tert-Butyl)phenyl)ethynyl)pyridine [3] (1.0 mmol) and (bromomethyl)benzene (1.2 mmol) were dissolved in 25 mL acetone. The reaction mixture was stirred at room temperature for 12 h. Upon the reaction completion (monitored by TLC), the mixture was filtered and washed with acetone to obtain the 1-benzyl-2-((4-(tert-butyl)phenyl)ethynyl)pyridin-1-

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.21114(2)	1.25943(2)	0.51416(2)	0.05462(13)
N1	0.22566(19)	0.70650(18)	0.44933(9)	0.0380(4)
C1	0.1538(2)	0.8163(2)	0.46128(13)	0.0478(6)
H1	0.062214	0.810955	0.466980	0.057*
C2	0.2180(3)	0.9313(2)	0.46458(13)	0.0526(7)
H2	0.171166	1.006162	0.472030	0.063*
C3	0.3595(3)	0.9354(2)	0.45637(13)	0.0535(7)
H3	0.404246	1.014232	0.458974	0.064*
C4	0.4335(3)	0.8237(2)	0.44442(13)	0.0501(6)
H4	0.525395	0.828388	0.439128	0.060*
C5	0.3655(2)	0.7075(2)	0.44084(11)	0.0389(5)
C6	0.4386(2)	0.5909(2)	0.42822(11)	0.0436(6)
C7	0.5118(2)	0.5004(2)	0.41617(11)	0.0439(6)
C8	0.6019(2)	0.3950(2)	0.39733(11)	0.0417(5)
C9	0.5511(3)	0.2720(2)	0.38968(14)	0.0496(6)
H5	0.464283	0.251509	0.399157	0.059*
C10	0.6398(3)	0.1742(2)	0.36588(13)	0.0514(6)
H6	0.604788	0.091329	0.361271	0.062*
C11	0.7788(2)	0.1936(2)	0.34850(11)	0.0453(6)
C12	0.8281(3)	0.3163(3)	0.35956(14)	0.0550(7)
H7	0.916300	0.336202	0.351937	0.066*
C13	0.7424(3)	0.4155(3)	0.38328(13)	0.0534(7)
H8	0.779318	0.497431	0.389973	0.064*
C14	0.8759(3)	0.0897(3)	0.31638(13)	0.0566(7)
C15	0.8036(4)	−0.0383(3)	0.3088(2)	0.0890(12)
H9	0.784466	−0.067057	0.350943	0.134*
H10	0.860995	−0.099931	0.291243	0.134*

*Corresponding author: Man-Hua Ding, Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou Hunan 425199, People's Republic of China, e-mail: 42979930@qq.com

Juan Liao and Fei Zeng: Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou Hunan 425199, People's Republic of China. <https://orcid.org/0000-0002-1555-1812> (F. Zeng)

Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
H11	0.719442	−0.029320	0.279507	0.134*
C16	0.9911(3)	0.0668(4)	0.35920(17)	0.0916(13)
H12	0.966574	0.037312	0.400200	0.137*
H13	1.043356	0.144543	0.366298	0.137*
H14	1.044894	0.002362	0.341388	0.137*
C17	0.9386(4)	0.1364(4)	0.24807(15)	0.0806(10)
H15	0.991523	0.067890	0.233124	0.121*
H16	0.995701	0.210482	0.257747	0.121*
H17	0.864293	0.157327	0.214402	0.121*
C18	0.1496(2)	0.5844(2)	0.44285(12)	0.0433(6)
H18	0.058919	0.594386	0.455147	0.052*
H19	0.198088	0.517801	0.469731	0.052*
C19	0.1424(2)	0.5543(2)	0.37125(13)	0.0470(6)
C20	0.0685(4)	0.6348(3)	0.33529(16)	0.0759(10)
H20	0.024044	0.703596	0.351891	0.091*
C21	0.0619(5)	0.6092(4)	0.27036(19)	0.1045(14)
H21	0.009321	0.664781	0.241722	0.125*
C22	0.1279(4)	0.5045(5)	0.24074(18)	0.0976(14)
H22	0.117551	0.497500	0.195217	0.117*
C23	0.1992(3)	0.4226(4)	0.2756(2)	0.0878(12)
H23	0.241931	0.353304	0.258474	0.105*
C24	0.2067(3)	0.4477(3)	0.34192(15)	0.0648(8)
H24	0.257627	0.390882	0.370513	0.078*

ium bromide as a yellow solid. Crystals of the title compound were obtained by slow evaporation of a water solution within 2 weeks.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. Some bad points were removed during the refinement.

Comment

Molecules and materials with phenylene ethynylene linkages have been widely used as (opto-)electronic materials [4], molecular wires [5–7], emissive devices and sensors [8, 9]. *N*-Heteroaromatic cations, species with quaternary pyridine-type nitrogen atoms, are of considerable interest due to their rich application potential [10–12]. Recently the synthesis of nitrogen heteroaromatic cations by [2+2+2] cycloaddition was reported. The product is comparable with the title compound [13]. Encouraged by the excellent photo-physical properties of phenylene ethynylene molecules and rich application potential of nitrogen heteroaromatic cations, we synthesized the phenylene ethynylene linked pyridinium derivatives. The title compound, built up by the C₂₄H₂₄BrN

molecules, has been synthesized. The single crystal structure verifies that all bond lengths are in normal ranges [13].

Acknowledgements: The work was supported by National Natural Science Foundation of China No. 21602055; Natural Science Foundation of Hunan Province No. 2017JJ3094 and Undergraduate Research Study and Innovative Experiment of Hunan Provincial (2016-636).

References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: SHELXT-integrated space-group and crystal-structure determination. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Ezugwu, C. I.; Mousavi, B.; Asraf, M.; Luo, Z.; Verpoort, F.: Post-synthetic modified MOF for Sonogashira cross-coupling and Knoevenagel condensation reactions. *J. Catal.* **344** (2016) 445–454.
4. Wang, C.; Dong, H.; Hu, W.; Liu, Y.; Zhu, D.: Semiconducting π -conjugated systems in field-effect transistors: a material odyssey of organic electronics. *Chem. Rev.* **112** (2012) 2208–2267.
5. Linton, K. E.; Fox, M. A.; Plisson, L.; Bryce, M. R.: Oligo (*p*-phenyleneethynylene) (OPE) molecular wires: synthesis and length dependence of photoinduced charge transfer in OPEs with triarylamine and diaryloxadiazole end groups. *Chem. Eur. J.* **21** (2015) 3997–4007.
6. Scott, A. M.; Ricks, A. B.; Colvin, M. T.; Wasielewski, M. R.: Comparing spin-selective charge transport through donor-bridge-acceptor molecules with different oligomeric aromatic bridges. *Angew. Chem. Int. Ed.* **49** (2010) 2904–2908.
7. Masai, H.; Fujihara, T.; Tsuji, Y.; Terao, J.: Programmed synthesis of molecular wires with fixed insulation and defined length based on oligo (phenylene ethynylene) and permethylated α -cyclodextrins. *Chem. Eur. J.* **23** (2017) 15073–15079.
8. McFarland, S. A.; Finney, N. S.: Fluorescent signaling based on control of excited state dynamics. biarylacetylene fluorescent chemosensors. *J. Am. Chem. Soc.* **124** (2002) 1178–1179.
9. Yang, J.-S.; Swager, T. M.: Porous shape persistent fluorescent polymer films: an approach to TNT sensory materials. *J. Am. Chem. Soc.* **120** (1998) 5321–5322.
10. Sliwa, W.: Quaternary salts of pyridines and related compounds. *Curr. Org. Chem.* **7** (2003) 995–1048.
11. Ihmels, H.; Otto, D.: Intercalation of organic dye molecules into double-stranded DNA—general principles and recent developments. *Top. Curr. Chem.* **258** (2005) 161–204.
12. Phillips, S. D.; Castle, R. N.: A review of the chemistry of the antitumor benzo[*c*]phenanthridine alkaloids nitidine and fagaronine and of the related antitumor alkaloid coralayne. *J. Heterocycl. Chem.* **18** (1984) 223–232.
13. Cizkova, M.; Kolivoska, V.; Cisarova, I.; Saman, D.; Pospisil, L.; Teply, F.: Nitrogen heteroaromatic cations by [2+2+2] cycloaddition. *Org. Biomol. Chem.* **9** (2011) 450–462.