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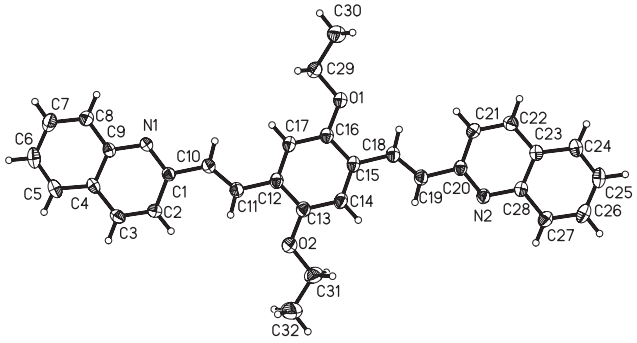
Crystal structure of 2,5-diethoxy-1,4-bis[2-(quinoline)ethenyl]benzene, $C_{32}H_{28}N_2O_2$



DOI 10.1515/ncrs-2014-9118
Received September 29, 2015; accepted January 12, 2016; available online February 3, 2016

Abstract

$C_{32}H_{28}N_2O_2$, monoclinic, $P2_1/n$ (no. 14), $a = 10.1291(10)$ Å, $b = 25.7313(3)$ Å, $c = 10.2395(10)$ Å, $\beta = 106.521(1)°$, $V = 2558.6(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0402$, $wR_{ref}(F^2) = 0.1045$, $T = 296(2)$ K.

CCDC no.: 1427061

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

The title compound was synthesized by solid-phase Wittig reaction. 2,5-Diethoxy-1,4-bis(triphenylphosphonium)benzene dichloride (1.18 g, 1.5 mmol) and quinoline-2-carbaldehyde (0.63 g, 4 mmol), fresh t-BuOK (1.12 g, 10 mmol) were crashed together with a pestle and mortar at room temperature for 0.5 h. Then, the product was purified silicagel column chromatography (8:1 v/v petroleum/ethyl acetate) in 52% yield as yellow solid. Single crystals of the title compound suitable for X-ray analysis were obtained from CH_3CN solvent.

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Table 1: Data collection and handling.

Crystal:	Yellow, block, size 0.10×0.10×0.10 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.77 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18052, 4498
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3008
$N(param)_{refined}$:	327
Programs:	SADABS [9], Bruker data collection and reduction program [10], SHELX [11]

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

Atom	Site	x	y	z	U_{iso}
H(10)	4e	1.0811	0.3108	0.0391	0.055
H(2)	4e	1.3936	0.3243	0.3136	0.055
H(22)	4e	0.2665	0.6065	−0.2321	0.058
H(14)	4e	0.9197	0.5148	0.1957	0.060
H(3)	4e	1.5719	0.2662	0.3640	0.058
H(11)	4e	1.2210	0.3788	0.2331	0.058
H(27)	4e	0.5551	0.7255	0.1643	0.064
H(19)	4e	0.7621	0.5718	0.0963	0.061
H(21)	4e	0.4517	0.5520	−0.1771	0.058
H(17)	4e	0.9328	0.3686	−0.0609	0.055
H(24)	4e	0.1565	0.6904	−0.1817	0.065
H(8)	4e	1.2868	0.1638	−0.0680	0.065
H(18)	4e	0.6273	0.5019	−0.0945	0.059
H(5)	4e	1.6748	0.1825	0.3013	0.070
H(25)	4e	0.1727	0.7637	−0.0524	0.073
H(29A)	4e	0.7158	0.3482	−0.1837	0.065
H(29B)	4e	0.7849	0.3776	−0.2822	0.065
H(7)	4e	1.4656	0.1060	−0.0337	0.078
H(26)	4e	0.3717	0.7814	0.1195	0.072
H(6)	4e	1.6585	0.1152	0.1526	0.081
H(31A)	4e	1.0611	0.5065	0.4124	0.086
H(31B)	4e	1.1530	0.5303	0.3267	0.086
H(30A)	4e	0.5001	0.3791	−0.3117	0.118
H(30B)	4e	0.5679	0.3460	−0.4038	0.118
H(30C)	4e	0.5700	0.4068	−0.4111	0.118
H(32A)	4e	1.2566	0.4683	0.5597	0.152
H(32B)	4e	1.2753	0.5288	0.5573	0.152
H(32C)	4e	1.3478	0.4928	0.4750	0.152

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(1)	4*e*	1.2645(1)	0.24469(5)	0.0678(1)	0.0393(8)	0.0424(7)	0.0433(8)	0.0059(6)	0.0025(6)	0.0005(6)
N(2)	4*e*	0.5782(1)	0.63842(5)	0.0525(1)	0.0438(8)	0.0394(7)	0.0583(9)	0.0014(6)	0.0142(7)	−0.0027(6)
O(1)	4*e*	0.7132(1)	0.42498(4)	−0.1656(1)	0.0565(8)	0.0486(7)	0.0581(8)	0.0125(5)	0.0071(6)	−0.0047(6)
C(1)	4*e*	1.2738(2)	0.28603(5)	0.1470(2)	0.0392(9)	0.0385(8)	0.0406(9)	0.0015(7)	0.0081(8)	0.0044(7)
C(28)	4*e*	0.4679(2)	0.67175(6)	0.0182(2)	0.0441(9)	0.0380(8)	0.050(1)	0.0018(7)	0.0199(8)	0.0030(7)
C(23)	4*e*	0.3471(2)	0.66147(6)	−0.0877(2)	0.0431(9)	0.0389(8)	0.049(1)	0.0020(7)	0.0184(8)	0.0074(7)
C(4)	4*e*	1.4905(2)	0.21669(6)	0.2061(2)	0.0366(9)	0.0471(9)	0.044(1)	0.0049(7)	0.0075(8)	0.0125(7)
C(15)	4*e*	0.8163(2)	0.47726(6)	0.0242(2)	0.044(1)	0.0371(8)	0.057(1)	0.0035(7)	0.0204(9)	0.0045(7)
C(9)	4*e*	1.3723(2)	0.21058(5)	0.0945(2)	0.0426(9)	0.0406(8)	0.0401(9)	0.0048(7)	0.0091(8)	0.0068(7)
C(10)	4*e*	1.1572(2)	0.32192(6)	0.1080(2)	0.0394(9)	0.0447(9)	0.049(1)	0.0046(7)	0.0062(8)	0.0012(7)
C(2)	4*e*	1.3899(2)	0.29495(6)	0.2597(2)	0.045(1)	0.0453(9)	0.043(1)	−0.0007(7)	0.0063(8)	−0.0023(7)
C(20)	4*e*	0.5700(2)	0.59479(6)	−0.0185(2)	0.0428(9)	0.0368(8)	0.053(1)	0.0011(7)	0.0164(8)	0.0022(7)
C(22)	4*e*	0.3438(2)	0.61488(6)	−0.1610(2)	0.049(1)	0.0442(9)	0.049(1)	−0.0020(7)	0.0093(8)	0.0037(7)
C(14)	4*e*	0.9214(2)	0.48509(6)	0.1444(2)	0.052(1)	0.0355(8)	0.065(1)	0.0025(7)	0.019(1)	−0.0035(8)
C(12)	4*e*	1.0336(2)	0.40512(6)	0.1132(2)	0.043(1)	0.0371(8)	0.057(1)	0.0014(7)	0.0165(9)	0.0045(7)
C(16)	4*e*	0.8210(2)	0.43196(6)	−0.0508(2)	0.044(1)	0.0399(8)	0.051(1)	0.0020(7)	0.0163(9)	0.0034(7)
C(3)	4*e*	1.4959(2)	0.26069(6)	0.2892(2)	0.041(1)	0.054(1)	0.044(1)	−0.0035(8)	0.0010(8)	0.0044(8)
C(11)	4*e*	1.1480(2)	0.36840(6)	0.1599(2)	0.044(1)	0.0431(9)	0.055(1)	0.0022(7)	0.0101(8)	0.0027(8)
C(27)	4*e*	0.4755(2)	0.71787(6)	0.0952(2)	0.059(1)	0.0447(9)	0.060(1)	−0.0017(8)	0.0221(9)	−0.0066(8)
C(19)	4*e*	0.6880(2)	0.55988(6)	0.0262(2)	0.047(1)	0.0410(9)	0.062(1)	0.0033(7)	0.0118(9)	−0.0007(8)
C(21)	4*e*	0.4532(2)	0.58244(6)	−0.1279(2)	0.054(1)	0.0381(8)	0.053(1)	0.0013(8)	0.0152(9)	−0.0034(7)
O(2)	4*e*	1.1313(1)	0.45513(4)	0.3094(1)	0.0688(9)	0.0522(7)	0.0789(9)	0.0102(6)	−0.0071(8)	−0.0143(7)
C(17)	4*e*	0.9293(2)	0.39756(6)	−0.0077(2)	0.047(1)	0.0372(8)	0.056(1)	0.0048(7)	0.0189(9)	0.0015(7)
C(13)	4*e*	1.0280(2)	0.44995(6)	0.1895(2)	0.048(1)	0.0409(9)	0.059(1)	0.0001(7)	0.0122(9)	0.0009(8)
C(24)	4*e*	0.2368(2)	0.69695(6)	−0.1123(2)	0.049(1)	0.052(1)	0.062(1)	0.0098(8)	0.0184(9)	0.0116(9)
C(8)	4*e*	1.3647(2)	0.16822(6)	0.0056(2)	0.061(1)	0.0463(9)	0.051(1)	0.0053(8)	0.0112(9)	−0.0016(8)
C(18)	4*e*	0.7004(2)	0.51320(6)	−0.0225(2)	0.045(1)	0.0420(9)	0.060(1)	0.0030(7)	0.0171(9)	0.0028(8)
C(5)	4*e*	1.5972(2)	0.17946(7)	0.2268(2)	0.047(1)	0.063(1)	0.062(1)	0.0138(9)	0.0118(9)	0.0171(9)
C(25)	4*e*	0.2465(2)	0.74068(7)	−0.0356(2)	0.063(1)	0.051(1)	0.075(1)	0.0192(9)	0.032(1)	0.0119(9)
C(29)	4*e*	0.7088(2)	0.37834(6)	−0.2424(2)	0.062(1)	0.048(1)	0.055(1)	0.0034(8)	0.018(1)	−0.0021(8)
C(7)	4*e*	1.4708(2)	0.13347(7)	0.0266(2)	0.083(2)	0.047(1)	0.069(1)	0.014(1)	0.028(1)	0.0011(9)
C(26)	4*e*	0.3661(2)	0.75122(6)	0.0681(2)	0.078(1)	0.0410(9)	0.074(1)	0.0076(9)	0.040(1)	−0.0009(9)
C(6)	4*e*	1.5871(2)	0.13923(7)	0.1384(2)	0.066(1)	0.060(1)	0.081(2)	0.027(1)	0.027(1)	0.018(1)
C(31)	4*e*	1.1431(2)	0.50111(7)	0.3832(2)	0.086(2)	0.056(1)	0.069(1)	−0.009(1)	0.014(1)	−0.010(1)
C(30)	4*e*	0.5747(2)	0.37750(8)	−0.3521(2)	0.083(2)	0.075(1)	0.068(1)	0.003(1)	0.005(1)	−0.005(1)
C(32)	4*e*	1.2668(3)	0.49741(9)	0.5047(2)	0.106(2)	0.086(2)	0.090(2)	−0.008(1)	−0.006(2)	−0.017(1)

Experimental details

All H-atoms were placed in calculated positions and treated as riding: C—H = 0.93–0.97 Å, with $U_{iso}(H) = 1.2$ or 1.5 U_{eq}(parent C-atom).

Discussion

The oligo(phenylene vinylene) and its derivatives (OPVs) are one kind of highly conjugated linear molecules, attracting increasing interest in the fields of nonliear optic [1], solid state opto-electronics [2] and molecular devices [3]. A large number of OPVs with large two-photon absorption sections (nonliear optical properties) were synthesized and investigated by changing the donor/acceptor(D/A) strength, conjugation length [4], for their potential applications in two-photon fluorescence imaging, optical power limiting, two-photon up-conversion lasing, three-dimensional optical data storage, 3D microfabrication, and photodynamic therapy [5]. Some of OPVs's single crystal structures are reported to get an insight into packing of the molecules and to explain the mechanisms of luminescence phenomena [6–8]. Here, a quinoline-terminated OPV has been synthesized and its crystal structure is reported. The crystal structure reveals that the C = C double bonds adopt an all *trans*-conformation with the bond length 1.320(2), 1.323(2) Å. The dihedral angles between terminated quinoline rings and benzene ring are 6.3 and 9.9°, respectivelly. So the backbone is nearly coplanar. In the crystal structure intermolecular π-π interactions play a major role in the molecular packing. With a contact distance of 3.32 Å they connect molecules forming a ladder-like structure, which are further stacked into a 3D structure

based on weak C—H· · · π interactions and C—H· · · N hydrogen bonds.

Acknowledgements: This project is supported by Educational Commission of Anhui Province of China (2014KJ016), Natural Science Foundation of Fuyang Normal College (2015FSKJ02ZD, 2014FSKJ06, FS201402001B) and the Innovation Training Program for the College students (201510371034, 201510371041).

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