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The crystal structure of 1,2-bis((4-methoxyphenyl)ethynyl)benzene, C₂₄H₁₈O₂

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:	Block, colorless
Size:	0.16 × 0.10 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	21324, 6512, 0.053
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4615
$N(\text{param})_{\text{refined}}$:	473
Programs:	Bruker programs [1], SHELX [2–4], OLEX2 [5], PLATON [6]

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Abstract

C₂₄H₁₈O₂, orthorhombic, *Ccc2* (no. 37), $a = 9.7401(14)$ Å, $b = 34.509(5)$ Å, $c = 21.970(3)$ Å, $V = 7384.7(19)$ Å³, $Z = 16$, $R_{\text{gt}}(F) = 0.0738$, $wR_{\text{ref}}(F^2) = 0.1559$, $T = 298(2)$ K.

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Source of materials

In a dried Schlenk-flask filled with argon, the (Ph₃P)₂PdCl₂ (0.036 g, 0.05 mmol) and CuI (0.011 g; 0.05 mmol) were dissolved in freshly degassed NEt₃ (10 mL). Afterwards 1,2-diiodobenzene (0.330 g; 1 mmol) and 4-methoxybenzene ethyne (0.397 g; 3 mmol) were introduced successively. The resulting reaction mixture was stirred at room temperature until completion. The crude reaction mixture was diluted with dichloromethane and washed with brine. The product was purified by flash column chromatography on silica gel using petroleum ether as eluent. Crystals were grown through a slow evaporation of a dichloromethane/petroleum ether (V/V = 1:5) solution at 25 °C for one week.

Experimental details

Hydrogen atoms bonded to carbon atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with C–H = 0.93 Å (aromatic), 0.96 Å (methyl) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic})$ and $1.5U_{\text{eq}}(\text{methyl})$.

Comment

1,5-Diyne systems are gaining importance as substrates for constructing multicyclic system in organic synthesis [7]. The reactivities are always tuned by the substituents in the alkyne moieties [8]. In general, the method for synthesis of 1,5-diyne is based on the Sonogashira coupling reaction of

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.2785(6)	0.39406(16)	0.5421(2)	0.0769(17)
O2	1.0484(5)	0.64752(16)	0.1891(2)	0.0705(15)
O3	0.4707(6)	0.35389(18)	0.1958(2)	0.0859(18)
O4	0.2085(6)	0.59879(16)	0.53431(18)	0.0714(15)
C1	0.0551(8)	0.4894(3)	0.1053(3)	0.089(3)
H1	0.0942	0.4824	0.0683	0.107*
C2	0.1166(8)	0.4767(2)	0.1592(3)	0.070(2)
H2	0.1947	0.4612	0.1583	0.084*
C3	0.0570(7)	0.48795(17)	0.2155(3)	0.0526(18)
C4	0.1077(7)	0.4733(2)	0.2719(3)	0.0603(19)
C5	0.1462(7)	0.46094(19)	0.3187(3)	0.0504(18)
C6	0.1811(7)	0.44397(19)	0.3761(3)	0.0543(19)
C7	0.3064(6)	0.42507(17)	0.3834(3)	0.0567(17)
H7	0.3664	0.4237	0.3505	0.068*
C8	0.3443(6)	0.40834(18)	0.4376(3)	0.0566(16)
H8	0.4285	0.3958	0.4412	0.068*
C9	0.2561(8)	0.4103(2)	0.4868(3)	0.0566(19)
C10	0.1323(7)	0.43010(18)	0.4807(3)	0.0613(18)
H10	0.0734	0.4321	0.5139	0.074*
C11	0.0967(6)	0.44655(18)	0.4267(3)	0.0563(17)
H11	0.0138	0.4598	0.4237	0.068*
C12	0.3975(8)	0.3702(2)	0.5489(4)	0.086(3)
H12A	0.4784	0.3853	0.5410	0.129*
H12B	0.3929	0.3490	0.5206	0.129*
H12C	0.4011	0.3602	0.5897	0.129*
C13	0.7933(7)	0.7338(2)	0.6270(3)	0.080(3)
H13	0.8235	0.7231	0.6635	0.096*
C14	0.8320(8)	0.7183(2)	0.5735(3)	0.071(2)
H14	0.8872	0.6963	0.5739	0.086*
C15	0.7938(7)	0.73353(19)	0.5184(3)	0.0545(18)
C16	0.8406(8)	0.7171(2)	0.4632(4)	0.067(2)
C17	0.8746(8)	0.7032(2)	0.4149(3)	0.065(2)
C18	0.9242(7)	0.68933(19)	0.3564(3)	0.0520(17)
C19	0.9229(6)	0.71202(18)	0.3058(3)	0.0574(17)
H19	0.8938	0.7376	0.3084	0.069*
C20	0.9647(6)	0.6970(2)	0.2507(3)	0.0592(17)
H20	0.9660	0.7127	0.2163	0.071*
C21	1.0043(8)	0.6589(2)	0.2465(3)	0.0496(17)
C22	1.0018(6)	0.63560(17)	0.2963(3)	0.0537(15)
H22	1.0279	0.6097	0.2932	0.064*
C23	0.9605(6)	0.65051(18)	0.3515(3)	0.0555(16)
H23	0.9568	0.6346	0.3856	0.067*
C24	1.1059(9)	0.6099(2)	0.1845(4)	0.097(3)
H24A	1.1790	0.6073	0.2135	0.145*
H24B	1.1412	0.6061	0.1442	0.145*
H24C	1.0363	0.5909	0.1927	0.145*
C25	0.6955(9)	0.2633(2)	0.6356(3)	0.078(2)
H25	0.6584	0.2723	0.6719	0.094*
C26	0.6455(9)	0.2755(2)	0.5810(3)	0.076(2)
H26	0.5731	0.2931	0.5812	0.092*
C27	0.6949(8)	0.26362(19)	0.5266(3)	0.063(2)
C28	0.6417(7)	0.27743(19)	0.4703(4)	0.063(2)
C29	0.6109(8)	0.2903(2)	0.4213(4)	0.071(2)
C30	0.5670(7)	0.30403(17)	0.3617(3)	0.0507(16)
C31	0.4443(7)	0.32251(18)	0.3541(3)	0.0596(17)
H31	0.3836	0.3240	0.3867	0.072*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C32	0.4085(6)	0.33897(19)	0.2994(3)	0.0609(17)
H32	0.3236	0.3510	0.2950	0.073*
C33	0.4980(9)	0.3376(2)	0.2515(3)	0.059(2)
C34	0.6228(8)	0.3194(2)	0.2589(3)	0.070(2)
H34	0.6837	0.3181	0.2264	0.084*
C35	0.6582(7)	0.3031(2)	0.3133(3)	0.068(2)
H35	0.7436	0.2914	0.3179	0.082*
C36	0.3535(8)	0.3771(3)	0.1891(4)	0.104(3)
H36A	0.2731	0.3621	0.1980	0.156*
H36B	0.3486	0.3865	0.1480	0.156*
H36C	0.3587	0.3987	0.2166	0.156*
C37	0.4658(9)	0.5168(2)	0.0922(3)	0.074(3)
H37	0.4466	0.5290	0.0553	0.089*
C38	0.4230(8)	0.5339(2)	0.1462(3)	0.065(2)
H38	0.3689	0.5561	0.1456	0.078*
C39	0.4627(7)	0.51706(18)	0.2018(3)	0.0525(18)
C40	0.4200(7)	0.5342(2)	0.2594(3)	0.0595(19)
C41	0.3771(7)	0.5458(2)	0.3057(4)	0.0585(19)
C42	0.3374(6)	0.56155(18)	0.3636(3)	0.0452(15)
C43	0.3331(6)	0.53678(19)	0.4145(3)	0.0607(18)
H43	0.3579	0.5109	0.4100	0.073*
C44	0.2930(7)	0.5501(2)	0.4707(3)	0.0600(17)
H44	0.2954	0.5338	0.5043	0.072*
C45	0.2486(7)	0.5884(2)	0.4772(3)	0.0540(19)
C46	0.2526(6)	0.61283(17)	0.4276(3)	0.0544(16)
H46	0.2272	0.6387	0.4318	0.065*
C47	0.2946(6)	0.59871(16)	0.3715(3)	0.0512(15)
H47	0.2935	0.6153	0.3381	0.061*
C48	0.1466(9)	0.6359(2)	0.5417(4)	0.089(3)
H48A	0.2121	0.6557	0.5318	0.133*
H48B	0.1173	0.6390	0.5831	0.133*
H48C	0.0688	0.6380	0.5151	0.133*

1,2-dihalobenzene derivatives with terminal alkynes [9]. The structure and conformation of the 1,5-dynes are key factors for their reactivities, which could have a big influence for the activation of alkyne by different metal catalysts [10].

The title compound crystallizes in the orthorhombic *Ccc2* space group with four halves of the titled compound in the asymmetric unit. One of these four independent molecules is shown in the figure. Each complete molecule looks like a “V” shape. The bond lengths and angles within these moieties are in the expected ranges. In each independent molecule, (C₂₄H₁₈O₂), the triple bond length is in a range from 1.170(2) Å to 1.211(2) Å, and bond angles for triple bond are 174.4° and 175.0°, respectively, which exhibit a slight distortion. Although there are two similar alkyne moieties in each molecule, they are located in an unsymmetric mode in the crystal structure. The dihedral angles between these two methoxy-substituted benzene rings and parent benzene ring are 20.25° and 24.84°, respectively.

Analysis using the Platon software indicates that there are four C—H···π intermolecular interactions in the

title structure [6], C8—H8...Cg1 (3.598(6) Å, 130°), C23—H23...Cg2 (3.663(7), 137°), C32—H32...Cg3 (3.647(6), 135°), C47—H47...Cg4 (3.542(6) Å, 131°) (Cg1 Cg2 Cg3 and Cg4 are the centroids of C42—C47, C6—C11, C18—C23, C30—C35 benzene rings, respectively, all symmetry codes: 1 - x, 1 - y, z). By a combination of these interactions, the molecules are linked into a three-dimensional network.

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