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1,3-Bis(2,6-dimethoxyphenyl)butenyne

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Abstract. $C_{20}H_{20}O_4$, $M_r = 324.4$, monoclinic, $P2_1/n$, $a = 7.8870\,(7)$, $b = 7.8150\,(9)$, $c = 27.478\,(4)$ Å, $\beta = 91.229\,(10)°$, $V = 1693.3\,(6)$ Å^3, $Z = 4$, $D_x = 1.272$ g cm^{-3} at 297 K, $\lambda(\text{Cu }K\alpha) = 1.54184$ Å, $\mu = 6.78$ cm^{-1}, $F(000) = 688$, 3468 unique data measured, final $R = 0.039$ for 3042 reflections with $I > 3.0\sigma(I)$. Maximum deviations from planarity of the two aromatic rings are 0.0064 (13) Å for the ring containing the ethynyl substituent and 0.0159 (12) Å for the ring containing the ethenyl substituent. The dihedral angle formed by the two rings is 79.76 (5)°. The ethenyl group is roughly orthogonal to the ring to which it is attached, as the dihedral angle between the plane encompassing the ethenyl substituent and the attached ring is 78.92 (6)°. All the methoxyl substituents are nearly coplanar with the rings to which they are attached with torsional deviations ranging from 2.5 (2) to 9.6 (2)°. The double-bond distance is 1.332 (2) Å; the triple-bond distance is 1.195 (2) Å; and the bond between the two has a length of 1.433 (2) Å.

Experimental. The enyne, (1), was isolated as a side product in the palladium-catalyzed coupling of 2-ethynyl-1,3-dimethoxybenzene with 2-methoxy-6-methoxycarbonylphenyl trifluoromethanesulfonate.

(1)
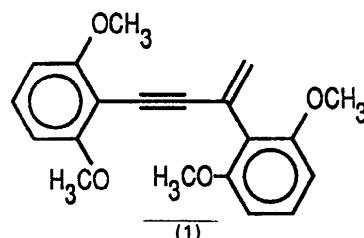
The enyne is formed by intermolecular palladium-catalyzed coupling of two molecules of 2-ethynyl-1,3-dimethoxybenzene. A colorless crystal of (1) was grown by evaporation from acetone. Crystal size

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$0.12 \times 0.35 \times 0.38$ mm, space group from systematic absences $h0l$ with $h + l$ odd and $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$. Data collection on an Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 90s, scan rates varied 0.61–$3.30°$ min^{-1}. One quadrant of data having $2 < \theta < 75°$, $0 \le h \le 9$, $0 \le k \le 9$, $-34 \le l \le 34$ measured. Data corrected for background, Lorentz and polarization effects. The standard reflections 400, 040, 014 varied randomly, and no decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0.9108 to 0.9973. 4088 total data were collected, and redundant $0kl$ and $0k\bar{l}$ data merged, $R_{int} = 0.010$, to yield 3468 unique data, 3042 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using MULTAN82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using Enraf–Nonius SDP/VAX (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located from difference maps and refined isotropically. Final $R = 0.039$ (0.046 for all data), $wR = 0.062$, $S = 3.457$ for 298 variables. Maximum shift 0.01σ in the final cycle, max. residual density 0.15 (3) e Å^{-3}, min. -0.14 (3) e Å^{-3}, extinction coefficient (Larson, 1969) $g = 2.6$ (2) $\times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Maximum correction 20.3% for the 006 reflection.

Coordinates* and equivalent isotropic thermal parameters are given in Table 1; bond distances, angles and selected torsion angles are given in Table 2. The molecule is illustrated in Fig. 1, and the unit cell is shown in Fig. 2.

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Table 1. *Coordinates and isotropic thermal parameters*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O1	0.2703 (1)	0.2442 (2)	0.25405 (4)	4.70 (2)
O2	0.7881 (1)	0.1340 (1)	0.33326 (4)	4.09 (2)
O3	0.0269 (1)	0.3479 (2)	0.43566 (4)	4.66 (2)
O4	0.5051 (1)	0.6824 (1)	0.40814 (4)	4.26 (2)
C1	0.4290 (2)	0.1797 (2)	0.24966 (5)	3.61 (3)
C2	0.4928 (2)	0.1128 (2)	0.20669 (5)	4.38 (3)
C3	0.6560 (2)	0.0517 (2)	0.20675 (5)	4.75 (3)
C4	0.7589 (2)	0.0540 (2)	0.24792 (6)	4.33 (3)
C5	0.6964 (2)	0.1217 (2)	0.29083 (5)	3.49 (2)
C6	0.5298 (2)	0.1839 (2)	0.29237 (4)	3.22 (2)
C7	0.4664 (2)	0.2508 (2)	0.33690 (5)	3.22 (2)
C8	0.4217 (2)	0.3027 (2)	0.37545 (5)	3.22 (2)
C9	0.3718 (2)	0.3651 (2)	0.42205 (4)	2.97 (2)
C10	0.4239 (2)	0.2895 (2)	0.46314 (5)	3.93 (3)
C11	0.2635 (2)	0.5219 (2)	0.42369 (4)	3.13 (2)
C12	0.0896 (2)	0.5096 (2)	0.43240 (5)	3.73 (3)
C13	−0.0074 (2)	0.6564 (2)	0.43744 (6)	4.97 (3)
C14	0.0690 (2)	0.8135 (2)	0.43356 (7)	5.67 (4)
C15	0.2396 (2)	0.8311 (2)	0.42372 (6)	4.95 (4)
C16	0.3361 (2)	0.6832 (2)	0.41834 (5)	3.63 (3)
C17	0.1645 (2)	0.2499 (3)	0.21155 (6)	5.23 (4)
C18	0.9652 (2)	0.0989 (3)	0.33200 (7)	5.32 (4)
C19	−0.1523 (2)	0.3284 (3)	0.44082 (6)	5.42 (4)
C20	0.5835 (2)	0.8414 (2)	0.39697 (7)	5.40 (4)

Table 2. *Bond distances (Å), angles (°), and selected torsion angles (°)*

O1—C1	1.357 (2)	C5—C6	1.403 (2)
O1—C17	1.421 (2)	C6—C7	1.430 (2)
O2—C5	1.362 (2)	C7—C8	1.195 (2)
O2—C18	1.424 (2)	C8—C9	1.433 (2)
O3—C12	1.361 (2)	C9—C10	1.332 (2)
O3—C19	1.432 (2)	C9—C11	1.495 (2)
O4—C16	1.367 (2)	C11—C12	1.401 (2)
O4—C20	1.424 (2)	C11—C16	1.394 (2)
C1—C2	1.395 (2)	C12—C13	1.388 (2)
C1—C6	1.404 (2)	C13—C14	1.373 (3)
C2—C3	1.372 (2)	C14—C15	1.385 (3)
C3—C4	1.378 (2)	C15—C16	1.393 (2)
C4—C5	1.392 (2)		

C1—O1—C17	117.7 (1)	C7—C8—C9	178.8 (1)
C5—O2—C18	117.9 (1)	C8—C9—C10	121.4 (1)
C12—O3—C19	117.8 (1)	C8—C9—C11	118.3 (1)
C16—O4—C20	118.0 (1)	C10—C9—C11	120.3 (1)
O1—C1—C2	124.4 (1)	C9—C11—C12	120.7 (1)
O1—C1—C6	115.1 (1)	C9—C11—C16	120.1 (1)
C2—C1—C6	120.5 (1)	C12—C11—C16	119.1 (1)
C1—C2—C3	119.0 (1)	O3—C12—C11	115.7 (1)
C2—C3—C4	122.2 (1)	O3—C12—C13	124.0 (1)
C3—C4—C5	119.2 (1)	C11—C12—C13	120.3 (1)
O2—C5—C4	124.1 (1)	C12—C13—C14	119.2 (1)
O2—C5—C6	115.5 (1)	C13—C14—C15	122.3 (2)
C4—C5—C6	120.4 (1)	C14—C15—C16	118.3 (1)
C1—C6—C5	118.8 (1)	O4—C16—C11	115.0 (1)
C1—C6—C7	121.4 (1)	O4—C16—C15	124.2 (1)
C5—C6—C7	119.8 (1)	C11—C16—C15	120.8 (1)
C6—C7—C8	176.2 (1)		

C6—C1—O1—C17	177.5 (1)
C6—C5—O2—C18	−170.1 (1)
C10—C9—C11—C12	−78.6 (2)
C11—C12—O3—C19	−175.3 (1)
C11—C16—O4—C20	173.3 (1)

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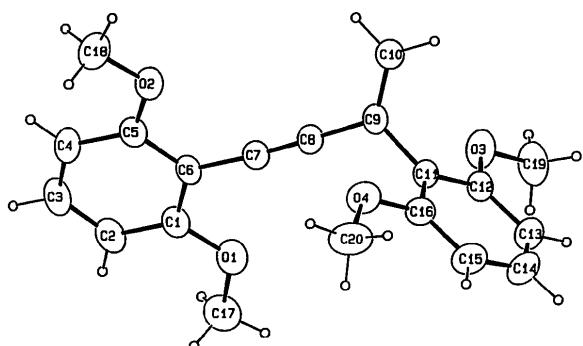
Fig. 1. ORTEP (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 40% probability level.
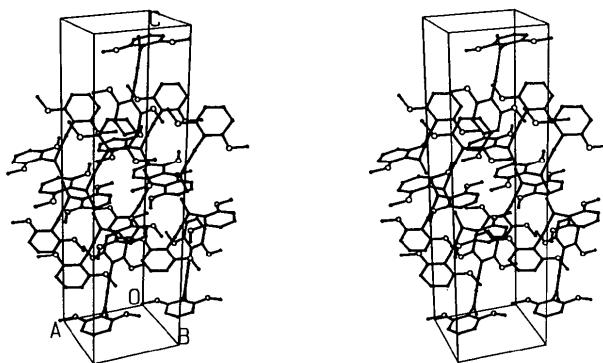

Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the b axis, with a horizontal.

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Methyl 2-(2,6-Dimethoxyphenyl)benzofuran-4-carboxylate

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Abstract. $C_{18}H_{16}O_5$, $M_r = 312.3$, orthorhombic, *Pbca*, $a = 11.4064(7)$, $b = 13.098(2)$, $c = 20.848(2)$ Å, $V = 3114.8(6)$ Å^3, $Z = 8$, $D_x = 1.332$ g cm^{-3} at 293 K, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 7.68$ cm^{-1}, $F(000) = 1312$, 3203 unique data measured, final $R = 0.040$ for 2738 reflections with $I > 3.0\sigma(I)$. Maximum deviation from planarity of the ring containing the two methoxyl substituents is 0.004(2) Å, and the maximum deviation from planarity of the benzofuran is 0.0259(12) Å. The dihedral angle formed between the benzofuran and the ring bearing the two methoxyl groups is 62.39(3)°. Both of the methoxyl substituents are nearly coplanar with the ring, and *anti* to the benzofuran. The torsional deviations are 2.3(2)° for the methoxyl group nearest the benzofuran oxygen and 5.8(2)° for the other. The ester substituent is nearly coplanar with the benzofuran ring system with a

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