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2-Ethynyl-1,3-dimethoxybenzene

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Abstract. $C_{10}H_{10}O_2$, $M_r = 162{\cdot}2$, orthorhombic, $Cmc2_1$, $a = 11{\cdot}587\ (2)$, $b = 9{\cdot}262\ (2)$, $c = 8{\cdot}603\ (3)$ Å, $V = 923{\cdot}3\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}17$ g cm^{-3}, λ(Cu $K\alpha$) $= 1{\cdot}54184$ Å, $\mu = 6{\cdot}2$ cm^{-1}, $F(000) = 344$, $T = 295\ (1)$ K, 1000 unique data measured, final $R = 0{\cdot}034$ for 929 reflections with $I > 3{\cdot}0\sigma(I)$. The molecule lies across a crystallographic mirror. The six-membered ring is nearly planar, with maximum deviation 0·002 (3) Å. The two methoxy substituents are nearly coplanar with the benzenoid ring with C—C—O—C torsion angles ± 1·4 (2)°, and are oriented *anti* to the ethynyl substituent. The triple-bond distance is 1·163 (2) Å.

Experimental. A white crystal of (1) was isolated by slow sublimation under reduced pressure and elevated temperature from the crude reaction product of 2-(1-chlorovinyl)-1,3-dimethoxybenzene and lithium diisopropylamide in tetrahydrofuran at room temperature. Crystal size 0·55 × 0·50 × 0·28 mm, cell dimensions from setting angles of 25 reflections having $20 < \theta < 30°$. 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 = 120 s, scan rates varied 0·33–3·59° min^{-1}. Two octants of data having $4 < 2\theta < 150°$, $0 \le h \le 14$, $0 \le k \le 11$, $-10 \le l \le 10$ measured. Data corrected for background, Lorentz and polarization effects. The standard reflections 200, 040, 004 decreased by 5·4%; thus, a linear decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0·948 to 0·999. 1059 total data were collected, of which 1000 were unique in point group $mm2$ and not systematically absent; 929 observed with $I > 3\sigma(I)$. When hkl and $hk\bar{l}$ were averaged R_{int} was 0·03. Systematic absences hkl with $h + k$ odd and $h0l$ with l odd indicated space groups $Cmcm$ or $Cmc2_1$, or with re-indexing $Ama2$. $Cmc2_1$ was shown to be correct by successful refinement, while no successful model was found in the other two. Structure solved by direct methods, using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Two weak reflections which had F_{obs} much greater than F_{calc} and F_{obs} much greater than F_{obs} for the Friedel-related reflection were given zero weight in the refinement. Non-H atoms refined

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

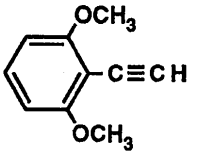
$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
O1	0·20095 (8)	0·2320 (1)	0	7·71 (2)
C1	0·1043 (1)	0·2942 (1)	−0·0591 (2)	6·31 (3)
C2	0	0·2382 (1)	−0·0048 (2)	5·20 (3)
C5	0	0·4589 (3)	−0·2181 (3)	11·16 (8)
C6	0·1042 (2)	0·4065 (2)	−0·1671 (2)	9·33 (5)
C7	0	0·1224 (2)	0·1043 (2)	5·33 (3)
C8	0	0·0268 (2)	0·1920 (3)	7·51 (4)
C9	0·3105 (2)	0·2876 (2)	−0·0494 (3)	11·03 (6)

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

O1—C1	1·358 (1)	C2—C7	1·425 (1)
O1—C9	1·434 (1)	C6—C5	1·373 (2)
C1—C2	1·396 (1)	C7—C8	1·163 (2)
C1—C6	1·395 (1)		
C1—O1—C9	117·80 (9)	C1—C2—C7	120·02 (5)
O1—C1—C2	115·52 (7)	C1—C6—C5	118·5 (1)
O1—C1—C6	124·5 (1)	C2—C7—C8	179·2 (1)
C2—C1—C6	120·0 (1)	C1—C2—C1	120·0 (1)
C6—C5—C6	123·1 (1)		
C9—O1—C1—C2	−178·7 (2)	C6—C1—C2—C7	179·34 (14)
C9—O1—C1—C6	1·4 (2)	O1—C1—C6—C5	179·6 (2)
O1—C1—C2—C7	−0·5 (2)	C2—C1—C6—C5	−0·3 (3)

anisotropically. All H atoms were fixed in calculated positions with C—H 0·95 Å and $B_{iso} = 1{\cdot}3B_{eq}$ for the C atoms to which they are bonded. Final $R = 0{\cdot}034$ (0·037 for all data), $wR = 0{\cdot}049$, $S = 2{\cdot}78$ for 61 variables. Maximum shift $< 0{\cdot}01\sigma$ in the final cycle, max. residual density 0·13 (3), min. −0·14 (3) e Å^{-3}, extinction coefficient $g = 3{\cdot}7\,(2) \times 10^{-5}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Refinement of the structure with $-x$, $-y$, $-z$ was not conclusive in determining the direction of the polar axis.



(1)

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

* Lists of hydrogen positional parameters, least-squares planes, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52212 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

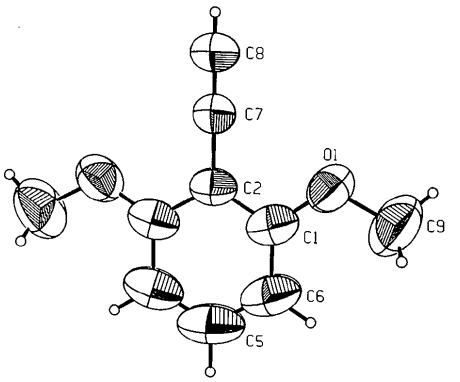


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 50% probability level.

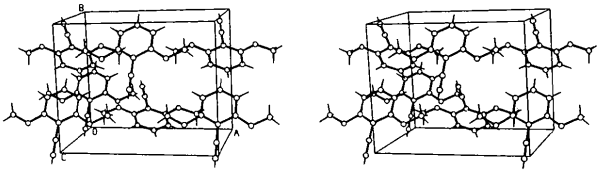


Fig. 2. Stereoview of the unit cell of the title compound.

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