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1-Chloro-3-ethynyl-2,4-dimethoxybenzene

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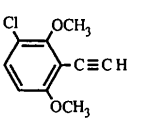
Abstract. $C_{10}H_9ClO_2$, $M_r = 196{\cdot}6$, monoclinic, $P2_1/c$, $a = 10{\cdot}8146\ (6)$, $b = 8{\cdot}8883\ (8)$, $c = 10{\cdot}4110\ (9)$ Å, $\beta = 103{\cdot}101\ (6)°$, $V = 974{\cdot}7\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}340$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 32{\cdot}27$ cm^{-1}, $F(000) = 408$, $T = 296$ K, $R = 0{\cdot}061$ for 1405 observations (of 2004 unique data). The molecule contains two methoxy groups; one is nearly coplanar with the benzenoid ring, with C—C—O—C torsion angle $-5{\cdot}2\ (5)°$, and the other, which resides between the chloro and the ethynyl groups, is nearly orthogonal, with the corresponding torsion angle 86·5 (4)°. The coplanar methoxy has an angle about O of 118·3 (2)° and the orthogonal, 114·7 (1)°. The O—CH_3 distance in the coplanar methoxy is 1·426 (2) Å compared to 1·439 (2) Å in the orthogonal. The six-membered ring is planar, with maximum deviation 0·008 (3) Å. The C—Cl distance is 1·734 (2) Å, and the triple-bond distance is 1·179 (3) Å. The ethynyl group forms a nearly linear C—H···O contact with the O atom of the orthogonal methoxy on a glide-related molecule, having C···O distance 3·293 (3) Å and angle at H of 167 (3)°.

Experimental. Colorless plates of (1), m.p. 365·5–365·9 K, were isolated by recrystallization in hexane from the crude reaction product of 2,6-dimethoxyacetophenone and phosphorus pentachloride in benzene at room temperature. Crystal size 0·08 × 0·30 × 0·38 mm, space group from systematic absences $h0l$ with l odd and $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$.



(1)

Data collection on 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 1·10–3·30° min^{-1}. One quadrant of data having $2 < \theta < 75°$, $0 \le h \le 13$, $0 \le k \le 11$, $-13 \le l \le 13$ measured. Data corrected for background, Lorentz and polarization effects. Since the crystal sublimed during data collection, the standard reflections 300, 060, 002 decreased by 16·74%, and linear decay correction was applied. Absorption corrections were based on ψ scans, with min. relative transmission coefficient 62·95%. 2486 total data were collected, and redundant data merged, $R_{int} = 0{\cdot}016$, to yield 2004 unique data, 1405 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN* (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). Non-H atoms refined anisotropically; H atoms located by ΔF map. Methyl H atoms fixed with C—H 0·95 Å and $B_{iso} = 1{\cdot}3B_{eq}$ for the methyl C atom. Other H atoms were refined isotropically. Final $R = 0{\cdot}061$ (0·090 for all data), $wR = 0{\cdot}072$, $S = 3{\cdot}007$ for 131 variables. Max. shift $< 0{\cdot}01\sigma$ in the final cycle, max. residual density 0·42, min. −0·31 e Å^{-3}, extinction coefficient $g = 3{\cdot}6\ (5) \times 10^{-6}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Coordinates† are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1.

† Tables of H-atom coordinates, least-squares planes, torsion angles, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52060 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac\beta_{13}\cos\beta]$.

	x	y	z	B_{eq} (Å^2)
Cl	0·1522 (1)	0·3331 (1)	0·03160 (9)	7·50 (3)
O1	0·0889 (2)	0·5477 (3)	−0·1901 (2)	4·78 (5)
O2	0·4703 (2)	0·4920 (3)	−0·3351 (2)	4·91 (5)
C1	0·2484 (3)	0·3797 (4)	−0·0749 (3)	4·82 (7)
C2	0·2048 (3)	0·4804 (4)	−0·1773 (3)	4·02 (6)
C3	0·2812 (3)	0·5166 (3)	−0·2629 (3)	3·72 (6)
C4	0·4030 (3)	0·4523 (4)	−0·2448 (3)	4·04 (7)
C5	0·4456 (3)	0·3535 (4)	−0·1404 (3)	5·16 (8)
C6	0·3682 (4)	0·3193 (4)	−0·0576 (3)	5·71 (9)
C7	0·2361 (3)	0·6212 (4)	−0·3684 (3)	3·92 (6)
C8	0·1955 (3)	0·7091 (4)	−0·4522 (4)	5·28 (8)
C9	−0·0134 (3)	0·4745 (5)	−0·2812 (4)	6·2 (1)
C10	0·5976 (3)	0·4389 (5)	−0·3168 (4)	6·09 (9)

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

Cl1	C1	1·734 (2)	C3	C4	1·408 (2)	
O1	C2	1·368 (2)	C3	C7	1·437 (3)	
O1	C9	1·439 (2)	C4	C5	1·392 (3)	
O2	C4	1·360 (2)	C5	C6	1·364 (3)	
O2	C10	1·426 (2)	C5	H5	0·95 (2)	
C1	C2	1·390 (3)	C6	H6	0·90 (2)	
C1	C6	1·377 (3)	C7	C8	1·179 (3)	
C2	C3	1·383 (2)	C8	H8	1·02 (3)	
C2	O1	C9	114·7 (1)	O2	C4	C3	115·7 (2)
C4	O2	C10	118·3 (2)	O2	C4	C5	124·7 (2)
Cl1	C1	C2	119·8 (2)	C3	C4	C5	119·6 (2)
Cl1	C1	C6	120·4 (2)	C4	C5	C6	119·5 (2)
C2	C1	C6	119·8 (2)	C4	C5	H5	121 (1)
O1	C2	C1	119·9 (2)	C6	C5	H5	119 (1)
O1	C2	C3	120·3 (2)	C1	C6	C5	121·6 (2)
C1	C2	C3	119·7 (2)	C1	C6	H6	116 (1)
C2	C3	C4	119·7 (2)	C5	C6	H6	123 (1)
C2	C3	C7	119·6 (2)	C3	C7	C8	177·3 (2)
C4	C3	C7	120·7 (2)	C7	C8	H8	172 (1)

Related literature. Structure of 2,6-dimethoxybenzoic acid: Swaminathan, Vimala & Lotter (1976), also Bryan & White (1982); structure of 2,7-dimethoxynaphthalene: Prince, Fronczek & Gandour (1989*a*); structure of 1-acetyl-2,7-dimethoxynaphthalene: Prince, Fronczek & Gandour (1989*b*); rotational barriers in dimethoxybenzenes: Anderson, Kollman, Domelsmith & Houk (1979); structures of crowded methoxybenzenes: Schuster, Parvez & Freyer (1988).

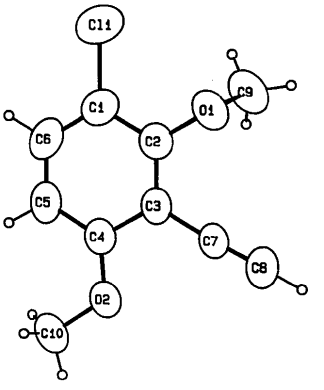

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound.

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