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Ethynylenebis(2-methoxybenzene)

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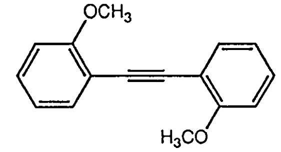
Abstract

The title compound is centrosymmetric, requiring *anti* methoxy groups about the ethynediyl link. The ethynyl bond length is 1.194 (2) Å; the ethynyl bond angle is 177.8 (1)°. The tolane ring is planar to within 0.0073 (13) Å, and the methoxy groups are almost coplanar with the ring, forming C—C—O—C torsion angles of magnitude −7.6 (2)°.

Comment

The title compound is an intermediate in the synthesis of diarylethynes that we designed to probe intramolecular recognition (Evans, Prince, Huang, Boss & Gandour, 1990). The determination of this structure contributes to our knowledge of the geometrical effects of various substituents in the *ortho* positions of diarylethynes and serves as a reference for molecular modeling studies. Our designs follow from Letsinger & Nazy (1959) who proposed that *o,o′*-disubstituted diarylethynes can be used as a framework to hold groups in ‘a prescribed geometry’.

Compound (1) was prepared by a modification of the procedure of Arcadi, Cacchi & Marinelli (1989). The reaction of 2-iodoanisole, 2-methoxyphenylethyne, tetrakis(triphenylphosphine)palladium(0), copper(I) iodide and diethylamine produced the title compound in good yield. Coleman, Holst & Maxwell (1936) first made the compound in a study of a new method for synthesizing tolanes.



(1)

A search of the January 1992 version of the Cambridge Structural Database (version 4.6; Allen, Kennard & Taylor, 1983) revealed one structure composed of a tolane backbone with oxygens in the *o,o′* positions (Evans, Horn, Fronczek & Gandour, 1990). That compound has all *ortho* positions substituted with three methoxy groups and a methoxycarbonyl. A comparison of the two molecules reveals that the three bond lengths of the ethynediyl link are identical. In the tetrasubstituted compound, the bond angles that are analogous to the C6—C7—C7′ bond angle in (1) differ – 175.0 (1) and 177.7 (1)°. The latter angle, which is on the side with two *ortho* methoxy groups, is identical to the C6—C7—C7′ bond angle in (1). The methoxy substituents display a slight deviation from planarity in relation to the phenyl ring with a C8—O1—C1—C2 torsion angle of −7.6 (2)°. The maximum deviation from planarity in the phenyl ring is 0.0073 (13) Å.

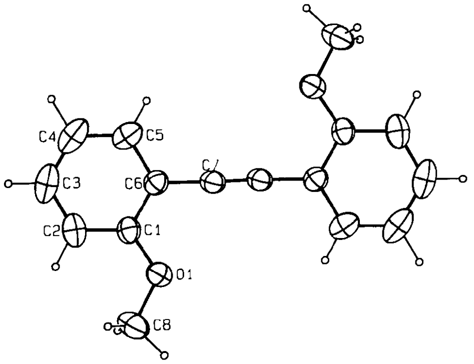


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 40% probability level.

Experimental

Crystal data

$C_{16}H_{14}O_2$
M_r = 238.3
Orthorhombic

Cell parameters from 25 reflections
θ = 25-30°

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Pbca
$a = 12.4588\ (11)$ Å
$b = 7.8993\ (5)$ Å
$c = 13.222\ (2)$ Å
$V = 1301.2\ (4)$ Å^3
$Z = 4$
$D_x = 1.216$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
$\mu = 0.60$ mm^{-1}
$T = 297$ K
Fragment
0.48 × 0.42 × 0.30 mm
Colorless
Crystal source: recrystallization from ethanol

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
$T_{\min} = 0.9294$, $T_{\max} = 0.9918$
1336 measured reflections
1336 independent reflections
1154 observed reflections $[I > 3\sigma(I)]$
$\theta_{\max} = 75°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 167 min
intensity variation: <1%

Refinement

Refinement on F
Final $R = 0.039$
$wR = 0.063$
$S = 4.054$
1154 reflections
111 parameters
All H-atom parameters refined
$w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$
$(\Delta/\sigma)_{\max} < 0.01$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.11$ e Å^{-3}
Extinction correction: $(I + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $g = 4.0\ (4) \times 10^{-6}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	$B_{\rm eq}$
O1	0.25708 (8)	0.0650 (2)	0.50833 (7)	4.74 (2)
C1	0.2980 (1)	0.1342 (2)	0.59400 (9)	3.88 (2)
C2	0.2384 (1)	0.2183 (2)	0.6668 (1)	5.28 (3)
C3	0.2885 (2)	0.2812 (2)	0.7520 (1)	6.58 (4)
C4	0.3975 (2)	0.2638 (2)	0.7652 (1)	6.64 (4)
C5	0.4572 (1)	0.1826 (2)	0.6930 (1)	5.31 (3)
C6	0.4093 (1)	0.1170 (2)	0.60610 (9)	3.82 (2)
C7	0.4722 (1)	0.0340 (2)	0.53066 (9)	3.85 (2)
C8	0.1433 (1)	0.0608 (3)	0.4975 (1)	5.96 (4)

Table 2. *Geometric parameters* (Å, °)

O1—C1	1.357 (2)	C3—C4	1.376 (3)
O1—C8	1.425 (2)	C4—C5	1.370 (2)
C1—C2	1.386 (2)	C5—C6	1.395 (2)
C1—C6	1.402 (2)	C6—C7	1.428 (2)
C2—C3	1.380 (2)	C7—C7′	1.194 (2)
C1—O1—C8	117.8 (1)	C3—C4—C5	119.6 (2)
O1—C1—C2	124.8 (1)	C4—C5—C6	121.0 (2)
O1—C1—C6	115.3 (1)	C1—C6—C5	118.8 (1)
C2—C1—C6	119.8 (1)	C1—C6—C7	120.5 (1)
C1—C2—C3	119.8 (1)	C5—C6—C7	120.7 (1)
C2—C3—C4	121.0 (2)	C6—C7—C7′	177.8 (1)

The sample was sealed in a capillary tube. Programs used were *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *MolEN* (Fair, 1990) and *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles involving H atoms, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71169 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1031]

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Structure of (−)-Muscarone Iodide

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Abstract

The crystal structure of (−)-*cis*-trimethyl{[(2*S*,5*S*)-2-methyl-3-oxotetrahydro-5-furyl]methyl}ammonium iodide (muscarone iodide) is comparable to the structure obtained for the racemic mixture [Frydenvang & Jensen (1992). *Acta Cryst.* **C48**, 469–474]. The space group is different as no mirror planes can exist for the pure enantiomer. The conformation of the muscarone ion is a distorted twist, $^1T_5-{}^1E$ [phase = 80.2 (2)°], with the puckering amplitude $\theta_m = 35.8\ (1)°$. The quaternary ammonium group adopts an *anti* orientation [O—C—C—N 161.0 (1)°]. The crystal packing is nearly identical to that of the racemic mixture. Both crystal structures are