The X-ray structure establishes the relative stereochemistry of (3) shown in Fig. 1. Since the absolute stereochemistry of the Mosher's acid was known, determination of the absolute stereochemistry of (3) and hence (1) was achieved (see scheme 1). The geometry of the tetracyclononane cage in the title structure is similar to that in two other norbornadiene adducts (De Lucchi & Valle, 1984; Caughlan, Smith, Jennings & Voecks, 1976).

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Methyl 2-[(2,6-Dimethoxyphenyl)ethynyl]benzoate

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Abstract. $C_{18}H_{16}O_4$, $M_r = 296.3$, orthorhombic, $P2_12_12_1$, $a = 7.6025\ (10)$, $b = 13.948\ (2)$, $c = 14.428\ (3)$ Å, $V = 1530.0\ (4)$ Å^3, $Z = 4$, $D_x = 1.286$ g cm^{-3} at 298 K, $\lambda(Cu\ K\alpha) = 1.54184$ Å, $\mu = 7.05$ cm^{-1}, $F(000) = 624$, 2918 unique data, final $R = 0.034$ for 2818 reflections with $I > 3.0\sigma(I)$. Maximum deviations from planarity of the two aromatic rings are 0.004 (1) Å for the ring containing the methoxyl substituents and 0.012 (1) Å for the ring containing the ester substituent. The dihedral angle formed by the two rings is 4.7 (3)°. The triple-bond distance is 1.195 (2) Å. The triple bond deviates markedly from linearity, as the bond angle about the ethynyl carbon bonded to the benzoate group is 170.5 (1)°.

Experimental. The title compound was prepared by the copper iodide and palladium-catalyzed coupling of 2-ethynyl-1,3-dimethoxybenzene with methyl 2-iodobenzoate. A colorless crystal of (1) was isolated by slow evaporation from diethyl ether. Crystal size 0.30 × 0.37 × 0.50 mm, space group from systematic

(1)
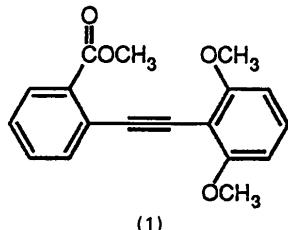
absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$. Data collection on an Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time = 90 s, scan rates varied 0.75–3.30° min^{-1}. Two octants of data having $2 < \theta < 75°$, $0 \le h \le 9$, $0 \le k \le 17$, $-18 \le l \le 18$ measured. Data corrected for background, Lorentz and polarization effects. The standard reflections 400, 020, 008 varied randomly, and no decay correction was applied. Absorption corrections were based on ψ scans, with relative transmission coefficients ranging from 0.9279 to 0.9910. 3468 total

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$_{eq}$ (Å^2)
O1	0.1781 (2)	0.49271 (7)	0.21714 (7)	5.22 (2)
O2	0.0998 (2)	0.25152 (8)	0.43712 (7)	5.68 (2)
O3	0.3408 (2)	0.07613 (8)	0.30840 (7)	7.39 (3)
O4	0.3658 (2)	−0.04984 (7)	0.21473 (7)	5.54 (2)
C1	0.1166 (2)	0.47070 (8)	0.30307 (9)	4.05 (2)
C2	0.0394 (2)	0.53626 (9)	0.3622 (1)	4.83 (3)
C3	−0.0179 (2)	0.5062 (1)	0.4472 (1)	5.34 (3)
C4	−0.0010 (2)	0.4122 (1)	0.4767 (1)	5.18 (3)
C5	0.0759 (2)	0.3463 (1)	0.41727 (9)	4.27 (2)
C6	0.1352 (2)	0.37443 (8)	0.32945 (8)	3.79 (2)
C7	0.2132 (2)	0.30668 (9)	0.26684 (9)	4.01 (2)
C8	0.2797 (2)	0.25091 (9)	0.21461 (9)	4.14 (2)
C9	0.3639 (2)	0.19731 (9)	0.14237 (9)	3.89 (2)
C10	0.3962 (2)	0.2456 (1)	0.05885 (9)	4.62 (3)
C11	0.4734 (2)	0.1999 (1)	−0.0152 (1)	5.29 (3)
C12	0.5238 (2)	0.1051 (1)	−0.0079 (1)	5.43 (3)
C13	0.4930 (2)	0.0561 (1)	0.0734 (1)	4.71 (3)
C14	0.4111 (2)	0.10033 (8)	0.14881 (9)	3.92 (2)
C15	0.3708 (2)	0.0436 (1)	0.23294 (9)	4.58 (3)
C16	0.1578 (3)	0.5887 (1)	0.1849 (1)	6.81 (4)
C17	0.0657 (3)	0.2206 (1)	0.5298 (1)	6.83 (4)
C18	0.3186 (3)	−0.1125 (1)	0.2903 (1)	7.41 (5)

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

O1—C1	1.360 (2)	C5—C6	1.401 (2)
O1—C16	1.425 (2)	C6—C7	1.435 (2)
O2—C5	1.364 (2)	C7—C8	1.195 (2)
O2—C17	1.429 (2)	C8—C9	1.434 (2)
O3—C15	1.201 (2)	C9—C10	1.403 (2)
O4—C15	1.330 (2)	C9—C14	1.402 (2)
O4—C18	1.442 (2)	C10—C11	1.376 (2)
C1—C2	1.382 (2)	C11—C12	1.382 (2)
C1—C6	1.403 (2)	C12—C13	1.377 (2)
C2—C3	1.367 (2)	C13—C14	1.397 (2)
C3—C4	1.384 (2)	C14—C15	1.481 (2)
C4—C5	1.388 (2)		

C1—O1—C16	118.2 (1)	C6—C7—C8	179.3 (1)
C5—O2—C17	117.7 (1)	C7—C8—C9	170.7 (1)
C15—O4—C18	116.8 (1)	C8—C9—C10	116.9 (1)
O1—C1—C2	124.0 (1)	C8—C9—C14	124.7 (1)
O1—C1—C6	115.4 (1)	C10—C9—C14	118.4 (1)
C2—C1—C6	120.6 (1)	C9—C10—C11	121.3 (1)
C1—C2—C3	119.1 (1)	C10—C11—C12	120.1 (1)
C2—C3—C4	122.5 (1)	C11—C12—C13	119.6 (1)
C3—C4—C5	118.5 (1)	C12—C13—C14	121.3 (1)
O2—C5—C4	124.7 (1)	C9—C14—C13	119.3 (1)
O2—C5—C6	114.8 (1)	C9—C14—C15	121.1 (1)
C4—C5—C6	120.6 (1)	C13—C14—C15	119.6 (1)
C1—C6—C5	118.8 (1)	O3—C15—O4	122.9 (1)
C1—C6—C7	120.1 (1)	O3—C15—C14	125.5 (1)
C5—C6—C7	121.2 (1)	O4—C15—C14	111.6 (1)

data were collected, and redundant data merged, R_{int} = 0.024, to yield 2918 unique data, 2818 observed with $I > 3\sigma(I)$. 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 $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the Enraf–Nonius package *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 =

0.03364 (0.035 for all data), wR = 0.04511, S = 2.819 for 264 variables. Maximum shift $< 0.07\sigma$ in the final cycle, max. residual density 0.17 e Å^{-3}, min. -0.19 e Å^{-3}, extinction coefficient (Larson, 1969) g = 1.38 (2) $\times$ 10^{-5}, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c, maximum correction 40.2% for the 202 reflection.

Refinement of the mirror-image structure under identical conditions yielded: R = 0.03383, wR = 0.04534, S = 2.833.

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

Related literature. Structure of 2-ethynyl-1,3-dimethoxybenzene: Evans, Horn, Fronczek, Gandour

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* Lists of H-atom coordinates, bond distances and angles involving H atoms, torsion angles, anisotropic thermal parameters, least-squares-planes data, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54140 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
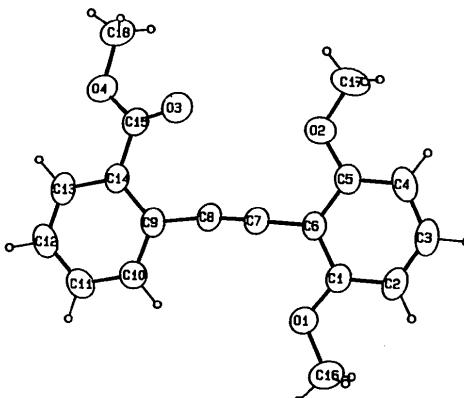

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound, with thermal ellipsoids drawn at the 40% probability level, and H atoms represented by circles of arbitary radius.
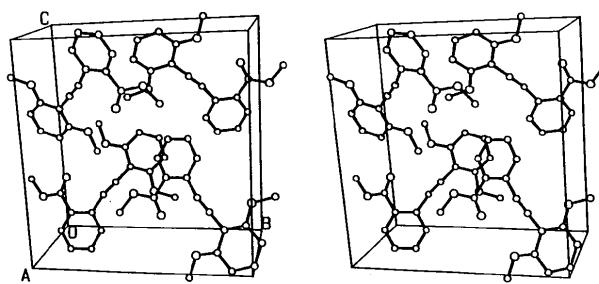

Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *a* axis, with *b* horizontal.

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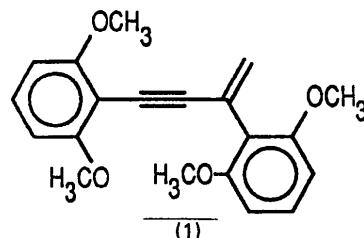
The enyne is formed by intermolecular palladium-catalyzed coupling of two molecules of 2-ethynyl-1,3-dimethoxybenzene. A colorless crystal of (1) was grown by evaporation from acetone. Crystal size

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