scheme to obtain flat dependence of Δ^2F vs F_o and $\sin\theta/\lambda$ was used (Martínez-Ripoll & Cano, 1975); overdetermination ratio was 8.4; maximum shift/ e.s.d. = 0.37; minimum and maximum heights in final ΔF map were -0.14 and $0.32\,e\,\text{Å}^{-3}$, respectively. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Geometrical calculations were made with *PARST* (Nardelli, 1983).

The molecule and numbering scheme are shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1,* and interatomic distances and bond angles in Table 2.

Related literature. Spectral studies were described by Rivera, Astudillo & Cataldo (1990). For related

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54699 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0492]

compounds see Rivera, Norte, Cataldo, Podestá & González (1990), Faulkner (1987) and Carrik & Paisley (1974).

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Methyl 8-[(2,6-Dimethoxyphenyl)ethynyl]-7-methoxy-2-naphthoate

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Abstract. $C_{23}H_{20}O_5$, $M_r = 376.4$, orthorhombic, $P2_12_12_1$, $a = 7.0784\,(8)$, $b = 12.1106\,(8)$, $c = 22.721\,(2)\,\text{Å}$, $V = 1947.7\,(5)\,\text{Å}^3$, $Z = 4$, $D_x = 1.284\,\text{g cm}^{-3}$, $\lambda(\text{Cu }K\alpha) = 1.54184\,\text{Å}$, $\mu = 7.0\,\text{cm}^{-1}$, $F(000) = 792$, $T = 296\,\text{K}$, $R = 0.036$ for 3727 observations (of 3902 unique data). The average deviations from planarity are 0.003 (2) Å with a maximum of 0.006 (2) Å for the phenyl ring, and 0.005 (2) Å with a maximum of 0.012 (2) Å for the naphthyl ring. The dihedral angle between the two rings is 14.7 (1)°. The two methoxy groups on the phenyl ring are nearly coplanar with the ring, with C—C—O—C torsion angles of 9.2 (2)° for the methoxy group opposite the ester, and -2.7 (3)° for the other one. The third methoxy group is nearly coplanar with the naphthyl ring, with a C—C—O—C torsion angle of -8.0 (2)°. The triple-bond distance is 1.184 (2) Å, and bond angles at the two ethynylic C atoms are 175·9 (1) and 176·9 (1)°.

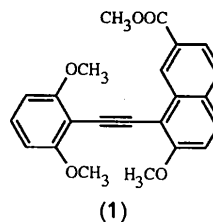* To whom correspondence should be addressed.

Experimental. The title compound (1), was prepared by the palladium-catalyzed coupling of 1-ethynyl-2,6-dimethoxybenzene and methyl 8-iodo-7-

(1)

methoxy-2-naphthoate in diethylamine at room temperature (Sonogashira, Tohda & Hagihara, 1975). Pale yellow plates of (1) were isolated by slow evaporation of dichloromethane. Crystal size 0.28 × 0.40 × 0.45 mm, mounted on a glass fiber in random orientation on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator, $\lambda(\text{Cu }K\alpha) = 1.54184\,\text{Å}$. Cell dimensions from setting angles of 25 reflections having $25 < \theta < 29°$. Space

Table 1. *Coordinates and equivalent isotropic thermal parameters* ($\mathring{A}^2$)

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

	x	y	z	B_{eq}
O1	0.5428 (2)	0.6571 (1)	0.55794 (5)	5.80 (3)
O2	1.0584 (1)	0.5838 (1)	0.67857 (5)	4.83 (2)
O3	0.3270 (2)	0.8288 (1)	0.69620 (5)	5.54 (2)
O4	1.1419 (2)	0.6153 (2)	0.93953 (5)	7.47 (4)
O5	1.1459 (2)	0.5731 (1)	0.84435 (5)	5.22 (2)
C1	0.7131 (2)	0.6065 (1)	0.56188 (6)	4.31 (3)
C2	0.7969 (3)	0.5457 (2)	0.51703 (6)	5.50 (4)
C3	0.9718 (3)	0.4984 (2)	0.52680 (7)	5.95 (4)
C4	1.0656 (2)	0.5092 (2)	0.57902 (8)	5.29 (3)
C5	0.9822 (2)	0.5692 (1)	0.62434 (6)	4.03 (3)
C6	0.8041 (2)	0.6180 (1)	0.61616 (5)	3.66 (2)
C7	0.7180 (2)	0.6746 (1)	0.66471 (5)	3.54 (2)
C8	0.6499 (2)	0.7158 (1)	0.70694 (5)	3.56 (2)
C9	0.5614 (2)	0.7604 (1)	0.75789 (5)	3.53 (2)
C10	0.3907 (2)	0.8171 (1)	0.75205 (6)	4.19 (3)
C11	0.2956 (2)	0.8576 (1)	0.80202 (8)	5.04 (3)
C12	0.3697 (2)	0.8423 (2)	0.85671 (7)	5.09 (3)
C13	0.5425 (2)	0.7869 (1)	0.86500 (6)	4.24 (3)
C14	0.6222 (3)	0.7717 (2)	0.92149 (6)	5.26 (3)
C15	0.7881 (3)	0.7192 (2)	0.92882 (6)	5.28 (4)
C16	0.8888 (2)	0.6774 (1)	0.87949 (6)	4.32 (3)
C17	0.8159 (2)	0.6906 (1)	0.82375 (5)	3.64 (2)
C18	0.6422 (2)	0.7455 (1)	0.81502 (5)	3.56 (2)
C19	1.0701 (2)	0.6200 (1)	0.89170 (6)	4.65 (3)
C20	0.4430 (3)	0.6527 (2)	0.50352 (8)	6.90 (5)
C21	1.2475 (2)	0.5493 (2)	0.68799 (9)	5.89 (4)
C22	0.1435 (3)	0.8723 (2)	0.6875 (1)	6.87 (5)
C23	1.3178 (3)	0.5103 (2)	0.8538 (1)	6.18 (4)

Table 2. *Bond distances* ($\mathring{A}$) *and angles* (°)

O1—C1	1.356 (2)	C6—C7	1.434 (2)
O1—C20	1.425 (2)	C7—C8	1.184 (2)
O2—C5	1.357 (2)	C8—C9	1.423 (2)
O2—C21	1.419 (2)	C9—C10	1.396 (2)
O3—C10	1.354 (2)	C9—C18	1.430 (2)
O3—C22	1.415 (2)	C10—C11	1.408 (2)
O4—C19	1.201 (2)	C11—C12	1.362 (2)
O5—C19	1.330 (2)	C12—C13	1.407 (2)
O5—C23	1.451 (2)	C13—C14	1.414 (2)
C1—C2	1.390 (2)	C13—C18	1.428 (2)
C1—C6	1.398 (2)	C14—C15	1.346 (3)
C2—C3	1.382 (3)	C15—C16	1.421 (2)
C3—C4	1.366 (2)	C16—C17	1.377 (2)
C4—C5	1.392 (2)	C16—C19	1.486 (2)
C5—C6	1.405 (2)	C17—C18	1.411 (2)

C1—O1—C20	118.8 (1)	O3—C10—C9	115.4 (1)
C5—O2—C21	118.3 (1)	O3—C10—C11	124.0 (1)
C10—O3—C22	118.4 (1)	C9—C10—C11	120.6 (1)
C19—O5—C23	116.2 (1)	C10—C11—C12	120.3 (1)
O1—C1—C2	124.8 (1)	C11—C12—C13	121.5 (1)
O1—C1—C6	115.0 (1)	C12—C13—C14	122.0 (1)
C2—C1—C6	120.2 (1)	C12—C13—C18	119.4 (1)
C1—C2—C3	118.9 (1)	C14—C13—C18	118.6 (1)
C2—C3—C4	122.4 (2)	C13—C14—C15	121.5 (1)
C3—C4—C5	119.1 (2)	C14—C15—C16	120.5 (1)
O2—C5—C4	124.8 (1)	C15—C16—C17	119.8 (1)
O2—C5—C6	115.0 (1)	C15—C16—C19	116.9 (1)
C4—C5—C6	120.2 (1)	C17—C16—C19	123.3 (1)
C1—C6—C5	119.2 (1)	C16—C17—C18	120.7 (1)
C1—C6—C7	122.0 (1)	C9—C18—C13	118.7 (1)
C5—C6—C7	118.7 (1)	C9—C18—C17	122.4 (1)
C6—C7—C8	175.9 (1)	C13—C18—C17	119.0 (1)
C7—C8—C9	176.9 (1)	O4—C19—O5	122.8 (2)
C8—C9—C10	119.4 (1)	O4—C19—C16	123.8 (2)
C8—C9—C18	121.0 (1)	O5—C19—C16	113.4 (1)
C10—C9—C18	119.6 (1)		

collection. Lorentz and polarization corrections were group determined to be $P2_12_12_1$ from systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd. Two octants of data having $2 < 2\theta < 150°$, $0 \le h \le 8$, $0 \le k \le 15$, $-28 \le l \le 28$ were collected using ω–2θ scans designed for $I = 25\sigma(I)$, subject to maximum scan time of 90 s, scan rates varied 0.61–3.30° min^{-1}. Three reflections (400, 040, 016) were measured every 166 min, and their intensities exhibited only random fluctuations during data applied. An empirical absorption correction based on a series of ψ scans was applied to the data. Relative transmission coefficients ranged from 0.9253 to 0.9986 with an average value of 0.9696. The extinction coefficient was refined in the least squares to $g = 4.0$ (2) × 10^{-6}, where the correction factor (1 + gI_c)$^{-1}$ was applied to F_c. $R_{int} = 0.019$ for averaging redundant zonal data. Structure solved by direct methods, using *RANTAN* (Yao, 1981), and successive difference Fourier syntheses. The structure was refined by weighted full-matrix least squares; non-H atoms refined anisotropically; H atoms refined isotropically except for the methyl H atoms, which were allowed to ride on the C atoms with C—H distance 0.95 Å. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$ and weights were assigned as $w = 4F_o^2 \mathrm{Lp}[S^2(C + R^2B) + (0.02F_o^2)^2]^{-1}$, where S = scan rate, C = total integrated peak count, R = scan time/ background counting time, B = total background count, and Lp = Lorentz–polarization factor, using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scat-

tering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Of 3902 unique data, 3727 reflections having $I > 3\sigma(I)$ were used in the refinement. The final cycle included 286 variables and converged (largest $\Delta/\sigma = 0.02$) with $R = 0.03616$, $wR = 0.05073$, $R(\text{all}) = 0.038$, and $S = 2.982$. The maximum and minimum residual densities were 0.22 and -0.18 e Å^{-3}, respectively. Refinement of the inversion related structure under identical circumstances yielded $R = 0.03623$, $wR = 0.05093$, $S = 2.994$. Table 1 presents the final atomic coordinates* and equivalent isotropic thermal parameters for the former refinement, and Table 2 presents bond distances and angles. Fig. 1 illustrates the molecule and the numbering scheme, and Fig. 2 shows the unit cell.

Related literature. Crystal structures of methyl 2-[(2,6-dimethoxyphenyl)ethynyl]-3-methoxybenzoate: Evans, Horn, Fronczek & Gandour (1990),
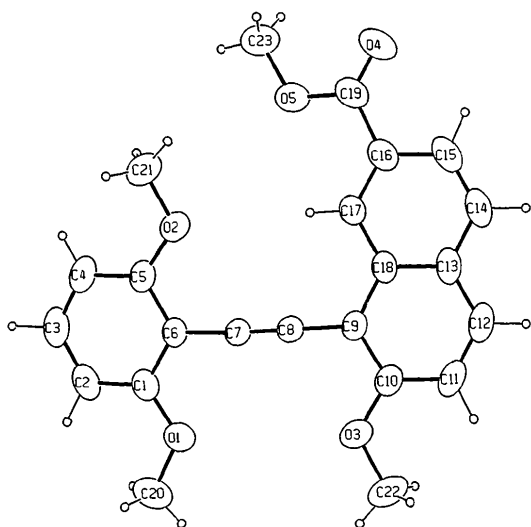
Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with arbitrary radius.

methyl 2-[(2,6-dimethoxyphenyl)ethynyl]benzoate: Huang, Evans, Fronczek & Gandour (1991) and 1-ethynyl-2,7-dimethoxynaphthalene: Prince, Fronczek & Gandour (1990).

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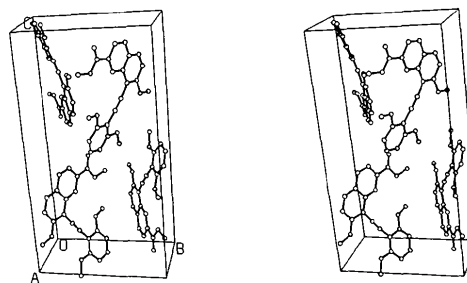

Fig. 2. Stereoview of the unit cell.

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Structure of 2-Cyanobenzophenone

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Abstract. 2-Benzoylbenzonitrile, $C_{14}H_9NO$, $M_r = 207.23$, monoclinic, $P2_1/c$, $a = 12.771$ (7), $b = 7.873$ (5), $c = 11.571$ (4) Å, $\beta = 112.47$ (3)°, $V = 1075$ (1) Å^3, $Z = 4$, $D_x = 1.280$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0.71073$ Å, $\mu = 0.08$ mm^{-1}, $F(000) = 432$, $T = 291$ (1) K, final $R = 0.048$ for 981 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The structure consists of discrete molecular units. All bond lengths, bond angles and dihedral angles are normal. The torsion angles O(1)—C(1)—C(8)—C(13) -42.6 (4) and C(1)—C(8)—C(13)—C(14) -2.8 (5)° indicate relatively strong repulsion between the C=O and C≡N groups. The planes of the two rings form a dihedral angle of 66.2 (1)°.