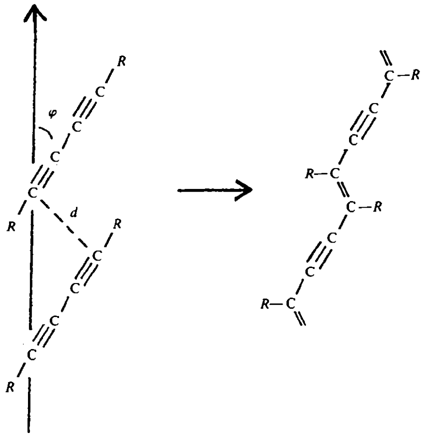
using an Acorn Workstation ACW443. Final atomic parameters are listed in Table 1 and bond lengths and angles in Table 2.* Fig. 1 shows the atomic labelling scheme and illustrates the packing of molecules in the y direction. Fig. 2 shows the unit-cell contents viewed in projection down the b axis.

Related literature. The molecules are arranged in the y direction to form a close, inclined stack. The stack repeat distance is the length of the b axis, 4·269 Å. The intermolecular separation d between the diacetylene C atoms C2 and C2(at $1-x$, $-1-y$, $1-z$) is 4·00 Å; these are the C atoms which are potentially reactive in respect of solid-state *trans*-1,4′-polymerization (see scheme). The stacking angle, φ, is defined by the intermolecular angle C1—C2···C2(at x, $1+y$, z) and is 59·4°. These distance and angle parameters are at the limit of the range of values considered appropriate for crystal-state polymerization reactivity (Schmidt, 1967; Baughman, 1974; Wegner, 1977).



The only other 4-alkoxybenzoate diester whose crystal structure has been reported is hexa-2,4-diyne-1,6-diyl bis(4-hexyloxybenzoate), determined at room temperature by Shklover, Struchkov, Bagirov, Konstantinov & Amerik (1985) and at 185 K by Barrow, Lough, Milburn, Hardy, Nyitrai & Horvath (1989). The crystal structure of a sulfonate diester analogue, hexa-2,4-diyne-1,6-diyl bis(4-methoxybenzenesulfonate), has been determined by Fisher, Ando, Bloor & Hursthouse (1979). Both the above compounds exhibit polymerization activity but not in the crystal forms whose structures were determined.

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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 51918 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *anti*-2-(2-Cyano-3-methoxy-6-methylphenyl)-1,2-diphenyl-1-ethanol

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Abstract. $C_{23}H_{21}NO_2$, $M_r = 343·4$, monoclinic, $P2_1/c$, $a = 10·873\,(5)$, $b = 9·131\,(5)$, $c = 19·009\,(7)$ Å, $\beta = 104·54\,(4)°$, $V = 1826\,(1)$ Å^3, $Z = 4$, $D_x = 1·25$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·74$ cm^{-1}, $F(000) = 728$, $T = 295$ K. Final $R = 0·046$ for 1597 observed reflections. The bond distances are C—CN = 1·433 (5), C≡CN = 1·146 (5), O—C(benz.) = 1·360 (4), O—CH$_3$ = 1·428 (4), C—C(benz., av.) =

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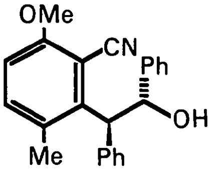
1·380 (7), C—C(aliphatic—aliphatic) = 1·536 (5), and C—C(benz.—aliphatic, av.) = 1·523 (5) Å. The X-ray structure confirms the *anti* disposition of the two phenyl substituents and the diastereoselectivity of the reaction.

Experimental. The title compound (1) was prepared by the reaction of α-lithio-2-cyanodiarylmethane (obtained *in situ, via* an aryne generated from the reaction of 2-bromo-4-methylanisole and lithiophenylacetonitrile, or alternatively by the metallation of 2-cyanodiarylmethane) and benzaldehyde at 273 K for 15 min followed by acidic work-up (Crenshaw, Khanapure, Siriwardane & Biehl, 1988).



(1)

Crystals of (1) are colorless plates, unit-cell parameters by least-squares fit of 25 reflections in the range $10 < 2\theta < 25°$, crystal dimensions 0·30 × 0·40 × 0·10 mm, space group $P2_1/c$ from systematic absences (0*k*0, *k* odd, *h*0*l*, *l* odd); automatic Nicolet *R*3*m*/V diffractometer, graphite-monochromated Mo *Kα* radiation, θ/2θ scan mode (scan rate 3·0–15·0° min^{-1}, depending on intensity), 2801 measured reflections, 2399 independent reflections in the range $3 < 2\theta < 50°$, $R_{\text{int}} = 0{\cdot}012$, *hkl* range *h* −11→11, *k* 0→9, *l* 0→20, 1597 observed reflections with $I > 3{\cdot}0\sigma(I)$, σ(*I*) from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change in intensity (1·0%) during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Structure solved by direct methods (*SHELXTL-Plus*, Sheldrick, 1988) and subsequent difference Fourier methods. H positions were calculated and were

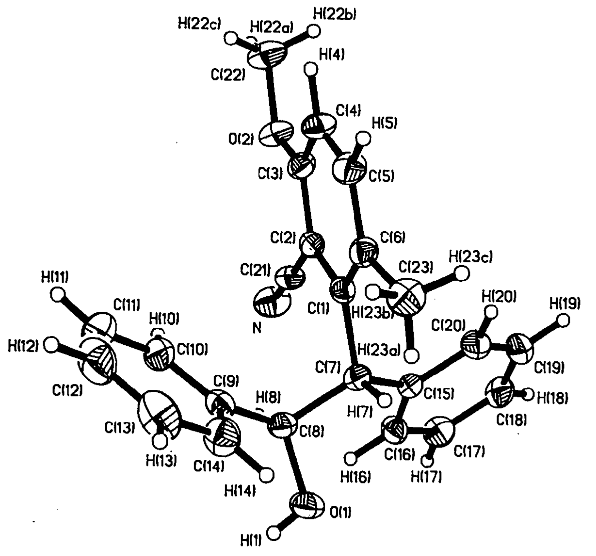


Fig. 1. *ORTEP* (Johnson 1965) drawing of the molecule. Thermal ellipsoids are scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3)

	x	*y*	*z*	U_{eq}*
O(1)	249 (2)	2154 (3)	597 (1)	59 (1)
O(2)	−5015 (2)	1358 (3)	−2173 (1)	53 (1)
N	−2136 (3)	−38 (4)	−1568 (2)	64 (1)
C(1)	−2704 (3)	3294 (4)	−755 (2)	37 (1)
C(2)	−3304 (3)	2258 (4)	−1280 (2)	36 (1)
C(3)	−4576 (3)	2404 (4)	−1662 (2)	43 (1)
C(4)	−5279 (3)	3561 (4)	−1501 (2)	53 (2)
C(5)	−4693 (3)	4555 (4)	−984 (2)	52 (2)
C(6)	−3419 (3)	4475 (4)	−609 (2)	41 (1)
C(7)	−1288 (3)	3141 (4)	−381 (2)	39 (1)
C(8)	−1000 (3)	1867 (4)	163 (2)	43 (1)
C(9)	−1962 (3)	1752 (4)	620 (2)	43 (1)
C(10)	−2937 (3)	758 (5)	448 (2)	57 (2)
C(11)	−3829 (4)	686 (6)	857 (2)	76 (2)
C(12)	−3730 (5)	1580 (6)	1441 (3)	83 (2)
C(13)	−2759 (5)	2547 (5)	1621 (2)	79 (2)
C(14)	−1878 (4)	2641 (5)	1214 (2)	63 (2)
C(15)	−498 (3)	3157 (4)	−941 (2)	37 (1)
C(16)	413 (3)	2124 (4)	−970 (2)	46 (1)
C(17)	1067 (3)	2159 (5)	−1503 (2)	56 (2)
C(18)	829 (3)	3230 (5)	−2022 (2)	57 (2)
C(19)	−62 (3)	4278 (5)	−2003 (2)	58 (2)
C(20)	−715 (3)	4242 (4)	−1462 (2)	51 (1)
C(21)	−2645 (3)	992 (4)	−1443 (2)	43 (1)
C(22)	−6295 (3)	1474 (5)	−2600 (2)	67 (2)
C(23)	−2860 (3)	5691 (4)	−89 (2)	60 (2)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

O(1)—C(8)	1·425 (3)	O(2)—C(3)	1·360 (4)
O(2)—C(22)	1·428 (4)	N—C(21)	1·146 (5)
C(1)—C(7)	1·532 (4)	C(2)—C(21)	1·433 (5)
C(6)—C(23)	1·510 (5)	C(7)—C(8)	1·536 (5)
C(7)—C(15)	1·527 (5)	C(8)—C(9)	1·521 (5)
C(3)—O(2)—C(22)	118·1 (3)	C(2)—C(1)—C(7)	120·0 (3)
C(6)—C(1)—C(7)	121·5 (3)	C(1)—C(2)—C(21)	121·7 (3)
C(3)—C(2)—C(21)	116·6 (3)	O(2)—C(3)—C(2)	115·3 (3)
O(2)—C(3)—C(4)	125·5 (3)	C(1)—C(6)—C(23)	123·0 (3)
C(5)—C(6)—C(23)	118·9 (3)	C(1)—C(7)—C(8)	113·1 (3)
C(1)—C(7)—C(15)	110·5 (2)	C(8)—C(7)—C(15)	115·0 (3)
O(1)—C(8)—C(7)	104·7 (3)	O(1)—C(8)—C(9)	112·0 (3)
C(7)—C(8)—C(9)	112·5 (3)	C(8)—C(9)—C(10)	120·7 (3)
C(8)—C(9)—C(14)	121·0 (3)	C(7)—C(15)—C(16)	123·8 (3)
C(7)—C(15)—C(20)	118·9 (3)	N—C(21)—C(2)	178·6 (4)
C(2)—C(1)—C(7)—C(8)	−71·9 (0·4)	C(2)—C(1)—C(7)—C(15)	58·6 (0·4)
C(6)—C(1)—C(7)—C(8)	110·6 (0·4)	C(6)—C(1)—C(7)—C(15)	−118·9 (0·4)
C(1)—C(7)—C(8)—O(1)	−163·6 (0·3)	C(1)—C(7)—C(8)—C(9)	−41.7 (0·4)
C(15)—C(7)—C(8)—O(1)	68·2 (0·3)	C(15)—C(7)—C(8)—C(9)	−170·0 (0·3)
C(1)—C(7)—C(15)—C(16)	−129·7 (0·3)	C(1)—C(7)—C(15)—C(20)	48·9 (0·4)
C(8)—C(7)—C(15)—C(16)	−0·2 (0·4)	C(8)—C(7)—C(15)—C(20)	178·4 (0·3)
O(1)—C(8)—C(9)—C(10)	−142·9 (0·3)	O(1)—C(8)—C(9)—C(14)	37·1 (0·4)
C(7)—C(8)—C(9)—C(10)	99·5 (0·4)	C(7)—C(8)—C(9)—C(14)	−80·5 (0·4)

included in the refinement in a riding model with fixed isotropic *U*'s. Final full-matrix least-squares refinement (*SHELXTL-Plus*) converged to $R = 0{\cdot}046$, $wR = 0{\cdot}048$, $S = 2{\cdot}21$ for 235 parameters; $w = 1/[\sigma^2(F) + 0{\cdot}00018F^2]$, $\sum w(|F_o| - |F_c|)^2$ minimized; $(\Delta/\sigma)_{\max} = 0{\cdot}003$, $\Delta\rho_{\max,\,\min} = 0{\cdot}32$, $-0{\cdot}26$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, O and N were those stored in *SHELXTL-Plus* which were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* bond lengths, angles and selected torsion angles are given in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1.

Related literature. Recently we observed (Khanapure, Crenshaw, Reddy & Biehl, 1988) that the reactions of arynes generated from 2-bromoanisoles and lithium diisopropylamide with lithioarylnitriles led to the formation of polysubstituted benzenes *via* a tandem addition rearrangement involving an initial addition (Siriwardane, Khanapure, Chu & Biehl, 1988*b*), a formation of a benzocyclobutenimine intermediate (Siriwardane, Reddy, Chu & Biehl, 1988) and ring opening of the cyclobutenimine (Siriwardane, Khanapure, Chu & Biehl, 1988*a*). The diastereoselective formation (Crenshaw *et al.*, 1988) of (1) in the electrophilic quenching using benzaldehyde is consistent with Cram's rule (Cram & Wilson, 1963).

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* Anisotropic temperature factors, bond lengths, bond angles, torsion angles, H parameters and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51827 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1989). C**45**, 1465–1467

Structure of a Bryonolic Acid Derivative, Bis(3β-acetoxy-*D*:*C*-friedoolean-8-en-29-oic) Anhydride

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(*Received* 27 *February* 1989; *accepted* 29 *March* 1989)

Abstract. $C_{64}H_{98}O_7$, $M_r = 979{\cdot}47$, monoclinic, $C2$, $a = 13{\cdot}574\,(1)$, $b = 6{\cdot}403\,(1)$, $c = 32{\cdot}504\,(2)$ Å, $\beta = 98{\cdot}57\,(1)°$, $V = 2793{\cdot}4\,(5)$ Å^3, $Z = 2$, $D_x = 1{\cdot}164$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu(\text{Cu } K\alpha) = 0{\cdot}58$ mm^{-1}, $F(000) = 1076$, $T = 295$ K, $R = 0{\cdot}062$ for 2268 observed reflections $[F_o > 3\sigma(F_o)]$. The molecule lies on a twofold rotational axis. The ring junctions *A*/*B*, *C*/*D* and *D*/*E* are *trans, trans* and *cis*, respectively. The conformations of rings *A*–*E* are chair, half-chair, half-chair, boat and boat, respectively.

Experimental. Colorless plates obtained from methanol. Crystal of dimensions 0·25 × 0·15 × 0·05 mm. Rigaku AFC-5 diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell dimensions determined from 2θ angles for 25 reflections in the range $30 < 2\theta < 45°$. Intensities measured up to $2\theta = 140°$ in h −16/16, k 0/7 and l 0/39, ω–2θ scans, ω-scan width $(1{\cdot}0 + 0{\cdot}2\tan\theta)°$. 2834 unique reflections measured, 2268 intensities observed $[F_o \le 3\sigma(F_o)$ and two very strong reflections rejected], no absorption corrections. Three standard reflections moni-

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