$\omega/2\theta$ scans
Absorption correction: none
1284 measured reflections
721 independent reflections
522 observed reflections $[I > 2\sigma(I)]$

$h = -4 \rightarrow 4$
$k = 0 \rightarrow 16$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 120 min
intensity variation: none

Refinement

Refinement on F
$R = 0.071$
$wR = 0.040$
$S = 3.00$
522 reflections
127 parameters
H-atom parameters not refined
Weights as default in *TEXSAN*

$(\Delta/\sigma)_{\max} = 0.04$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: CAD-4 *Software* (Enraf-Nonius, 1989). Cell refinement: CAD-4 *Software*. Data reduction: *SHELX76* (Sheldrick, 1976). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation,1985). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *CIF* (Hall, Allen & Brown, 1991).

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

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

	x	y	z	$B_{\rm eq}$
F1	0.4080 (13)	0.1649 (4)	0.6144 (5)	6.1 (4)
F2	0.0379 (13)	0.1554 (4)	0.7181 (6)	6.5 (4)
F3	−0.0007 (13)	0.1211 (4)	0.5448 (5)	6.9 (4)
O1	0.425 (2)	0.2980 (4)	0.7814 (7)	6.4 (5)
O2	0.329 (2)	0.4275 (5)	0.7495 (6)	5.8 (5)
N1	−0.189 (2)	0.3563 (6)	0.4962 (7)	4.0 (5)
N2	−0.028 (2)	0.3941 (5)	0.5784 (8)	4.2 (5)
C3	0.101 (2)	0.3325 (7)	0.6307 (9)	3.8 (7)
C4	0.029 (2)	0.2585 (7)	0.5834 (9)	3.7 (7)
C5	−0.159 (2)	0.2769 (6)	0.4951 (9)	4.0 (7)
C6	0.126 (3)	0.1765 (8)	0.6155 (11)	4.8 (8)
C7	0.303 (3)	0.3503 (8)	0.7312 (10)	4.6 (8)
C8	0.536 (3)	0.4523 (7)	0.8382 (10)	7.5 (9)
C9	−0.379 (2)	0.4066 (6)	0.4156 (9)	5.3 (7)

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

F1—C6	1.32 (1)	N1—C5	1.33 (1)
F2—C6	1.33 (1)	N1—C9	1.51 (1)
F3—C6	1.35 (1)	N2—C3	1.32 (1)
O1—C7	1.18 (1)	C3—C4	1.38 (1)
O2—C7	1.30 (1)	C3—C7	1.50 (1)
O2—C8	1.44 (1)	C4—C5	1.35 (1)
N1—N2	1.35 (1)	C4—C6	1.48 (1)
C7—O2—C8	116.9 (9)	N1—C5—C4	106 (1)
N2—N1—C5	114.4 (9)	F1—C6—F2	108 (1)
N2—N1—C9	118.5 (9)	F1—C6—F3	107 (1)
C5—N1—C9	127 (1)	F1—C6—C4	115 (1)
N1—N2—C3	101.2 (8)	F2—C6—F3	103 (1)
N2—C3—C4	114.1 (9)	F2—C6—C4	112 (1)
N2—C3—C7	118 (1)	F3—C6—C4	111 (1)
C4—C3—C7	128 (1)	O1—C7—O2	127 (1)
C3—C4—C5	103.8 (9)	O1—C7—C3	121 (1)
C3—C4—C6	131 (1)	O2—C7—C3	112 (1)
C5—C4—C6	125 (1)		

The CAD-4 diffractometer was funded by the SERC which is also thanked for funding the Chemical Databank Services used for cystallographic literature searches.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry including torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71507 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1068]

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Acta Cryst. (1994). C**50**, 116–120

meso- and *rac*-2,7-Diphenyl-3,5-octadiyne-2,7-diol, $C_{20}H_{18}O_2$

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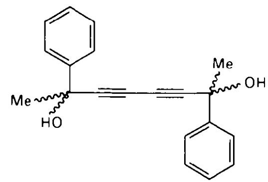
Abstract

The stereoisomeric forms of 2,7-diphenyl-3,5-octadiyne-2,7-diol (I) have been identified; *meso*- and *rac*-(I) exhibit different crystal packing, reflecting very different hydrogen-bonding networks.

Comment

Copper(I)-induced oxidative coupling of terminal acetylenes, the so-called Glaser reaction, provides a synthetically useful method for the preparation of conjugated diynes. In the case of propargylic alcohols of the type (±)-RR'C(OH)C≡CH, both *meso* and racemic products are possible. Yet, typically, these stereoisomeric forms are not distinguishable, either spectroscopically or chemically, and structures have not been assigned nor have analytical methods been developed to differentiate them. This note describes the stereoisomeric coupling products obtained from (±)-2-phenyl-3-butyn-1-ol. Previously reported structural studies on similar conjugated diyne diols are limited to inclusion compounds of tetraaryl derivatives (see below); in no instance have the diastereomeric forms of such coupling products been identified. The structures described herein were determined as a prelude to studies aimed at effecting directed coupling of terminal acetylenes.



(I)

Air oxidation of (±)-2-phenyl-3-butyn-1-ol in the presence of copper(I) halides produces 2,7-diphenyl-3,5-octadiyne-2,7-diol (I) as a mixture of stereoisomers (Strauss, Kollek & Hauptmann, 1930); the *meso* and racemic forms of (I) were separated by fractional crystallization and crystals of each suitable for X-ray analysis were obtained from benzene. The less-soluble form was identified as *meso*-(I), while *rac*-(I) was obtained from the mother liquors; the molecular structures and numbering used are shown in Fig. 1. The structure of the *meso* isomer contains two independent molecules, one of which (molecule *A*) lies on a center of inversion. In contrast, the asymmetric unit for the racemic form contains a single molecule. Noteworthy, are the differing geometries for the conjugated triple bond. For *rac*-(I), the four atoms in the diacetylene linkage [C(4), C(5), C(6) and C(7)] adopt an almost linear arrangement; one of the substituents [C(1)] lies along this line, with the other [C(8)] deviating significantly. In *meso*-(I), molecule *A* also has an almost linear diacetylene linkage [C(24), C(25), C(25*a*) and C(24*a*)], which is, however, slightly bent at both ends [*i.e.* neither C(21) nor C(21*a*) are collinear with the acetylene C atoms], while all the angles for the corresponding six atoms of molecule *B* deviate significantly from linearity, giving an overall bow of greater than 20°. The reasons for these differences are evident on examination of the crystal packing for each diastereomeric form. The intermolecular hydrogen-bonding network for *meso*-(I) yields cyclic dimers of those molecules with distorted acetylene linkages (*B* molecules); these dimers being linked by *A* molecules (*cf.* Fig. 2). In contrast, *rac*-(I) crystallizes as hydrogen-bonded chains of molecules with alternating molecules having opposite absolute configurations. These chains are in turn paired through additional hydrogen bonding, which, however, involves only one hydroxyl group of each diyne-diol unit, producing in effect cyclic tetramers (*cf.* Fig. 3).

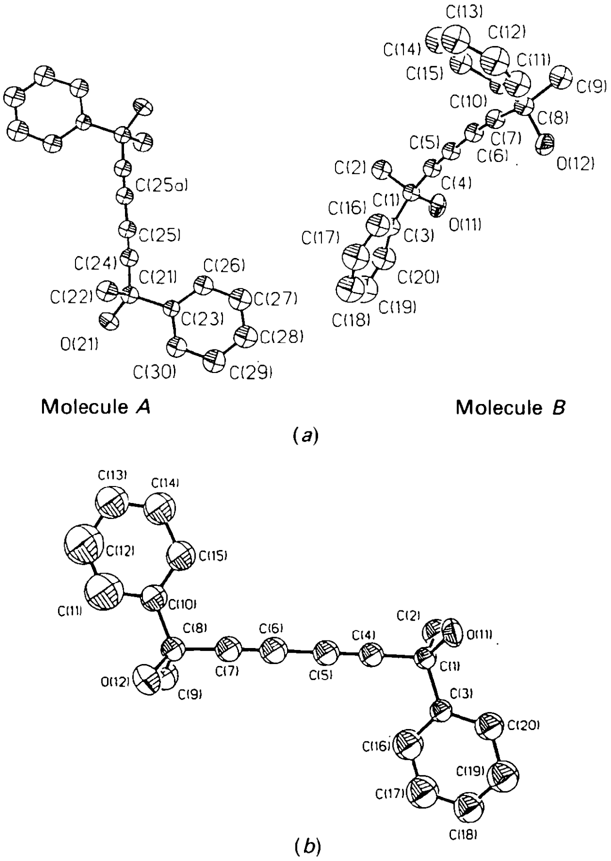


Fig. 1. Perspective view of (*a*) *meso*-2,7-diphenyl-3,5-octadiyne-2,7-diol and (*b*) the racemic form, showing ellipsoids at the 50% probability level and the atom-numbering scheme.

Structures of diyne-diols related to (I) have been reported by Toda and co-workers and have mainly involved clathrates of the three tetraaryl derivatives: 1,1,6,6-tetraphenylhexa-2,4-diyne-1,6-diol (Toda, Ward & Hart, 1981; Kaftory, Tanaka & Toda, 1985; Kaftory, 1987; Fukawa *et al.*, 1989; Kaftory, Yagi, Tanaka & Toda, 1988; Toda, Tanaka, Kai *et al.*, 1988; Toda, Tanaka, Asao *et al.*, 1988), 1,1,6,6-tetrakis(3,4-dimethylphenyl)hexa-2,4-diyne-1,6-diol (Toda, Tanaka & Mak, 1985) and chiral

1,6-bis(2-chlorophenyl)-1,6-diphenylhexa-2,4-diyne-1,6-diol (Kaftory, 1987; Fujiwara, Tanaka, Tanaka & Toda, 1989; Toda, Tanaka & Mak, 1989; Fujiwara, Nanba, Hamada, Toda & Tanaka, 1990).

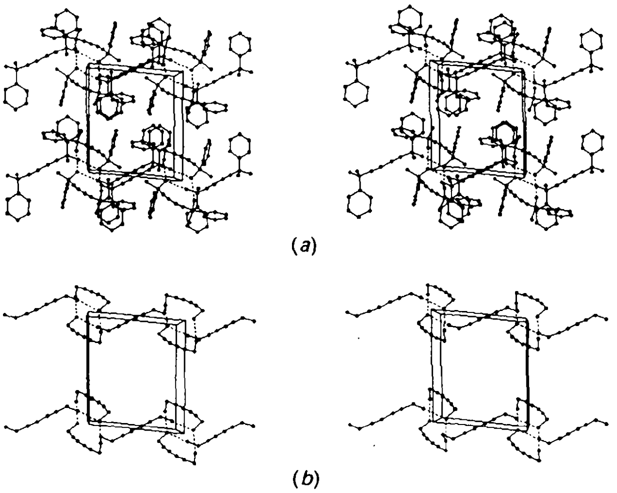


Fig. 2. Stereoscopic view of the crystal packing of *meso*-(I): (*a*) only H atoms omitted and (*b*) methyl and phenyl groups also omitted. Intermolecular O···O proximities suitable for O—H···O hydrogen bonding are indicated by broken lines.

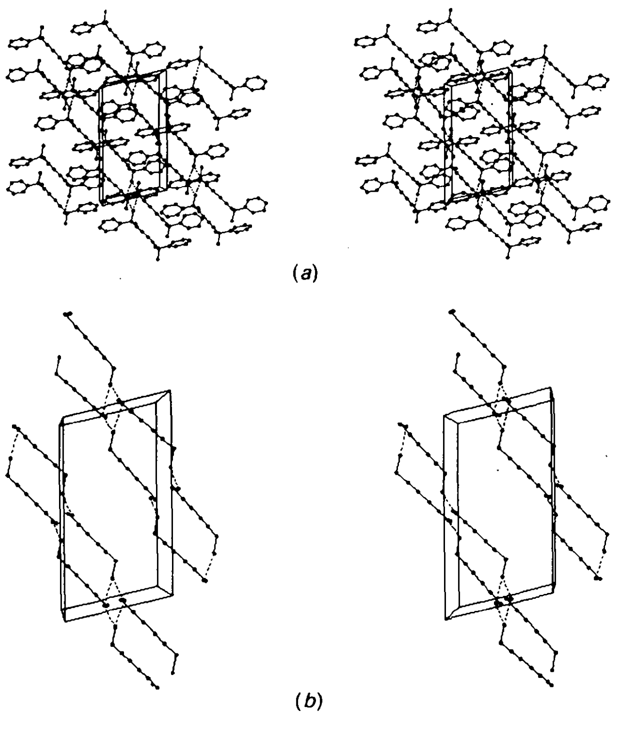


Fig. 3. Stereoscopic view of the crystal packing of *rac*-(I): (*a*) only H atoms omitted and (*b*) methyl and phenyl groups also omitted. Intermolecular O···O proximities suitable for O—H···O hydrogen bonding are indicated by broken lines.

Experimental

meso-(I)

Crystal data

$C_{20}H_{18}O_2$
$M_r = 290.3$
Triclinic
$P\bar{1}$
$a = 10.194$ (2) Å
$b = 10.224$ (2) Å
$c = 11.985$ (2) Å
$\alpha = 95.32$ (2)°
$\beta = 95.93$ (1)°
$\gamma = 97.03$ (1)°
$V = 1226.1$ (4) Å^3
$Z = 3$
$D_x = 1.180$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10 - 12.5°$
$\mu = 0.075$ mm^{-1}
$T = 298$ K
Rectangular plates
0.3 × 0.3 × 0.1 mm
Colorless

Data collection

Siemens *R3m/V* diffractometer
2θ-θ scans
Absorption correction: none
2436 measured reflections
2286 independent reflections
1451 observed reflections [$F > 6.0\sigma(F)$]
$R_{\rm int} = 0.59$
$\theta_{\rm max} = 20°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 50 reflections intensity variation: <4%

Refinement

Refinement on F
$R = 0.0677$
$wR = 0.0654$
$S = 1.26$
1451 reflections
148 parameters
H-atom parameters not refined
Unit weights applied
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.32$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.38$ e Å^{-3}
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) *for meso*-(I)

$U_{\rm iso}$ for C atoms; $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for O atoms.

	x	y	z	$U_{\rm eq}/U_{\rm iso}$
O(11)	0.1016 (4)	0.1259 (5)	0.8292 (4)	0.052 (2)
O(12)	0.7486 (4)	−0.1160 (4)	0.9708 (4)	0.052 (2)
O(21)	0.1456 (4)	0.8826 (4)	0.1044 (4)	0.048 (2)
C(1)	0.1801 (6)	0.1191 (6)	0.7369 (5)	0.039 (2)
C(2)	0.0815 (7)	0.0711 (7)	0.6321 (6)	0.050 (2)
C(3)	0.2536 (6)	0.2548 (6)	0.7241 (5)	0.038 (2)
C(4)	0.2781 (6)	0.0243 (6)	0.7566 (6)	0.040 (2)
C(5)	0.3609 (6)	−0.0431 (7)	0.7800 (6)	0.040 (2)
C(6)	0.4647 (6)	−0.1134 (6)	0.8089 (6)	0.040 (2)
C(7)	0.5596 (6)	−0.1641 (6)	0.8386 (6)	0.040 (2)
C(8)	0.6825 (6)	−0.2152 (7)	0.8831 (6)	0.039 (2)
C(9)	0.6476 (7)	−0.3414 (7)	0.9388 (6)	0.057 (2)
C(10)	0.7730 (6)	−0.2384 (6)	0.7927 (5)	0.036 (2)
C(11)	0.9083 (7)	−0.2358 (7)	0.8245 (7)	0.062 (2)
C(12)	0.9922 (9)	−0.2630 (8)	0.7435 (7)	0.077 (3)
C(13)	0.9440 (9)	−0.2925 (8)	0.6343 (7)	0.075 (3)
C(14)	0.8130 (8)	−0.2957 (8)	0.6000 (7)	0.067 (2)
C(15)	0.7257 (7)	−0.2686 (7)	0.6800 (6)	0.051 (2)
C(16)	0.3741 (8)	0.2698 (8)	0.6836 (6)	0.062 (2)
C(17)	0.4363 (9)	0.3959 (9)	0.6653 (7)	0.083 (3)

C(18)	0.3754 (9)	0.5034 (10)	0.6930 (7)	0.089 (3)
C(19)	0.2580 (10)	0.4908 (10)	0.7333 (8)	0.095 (3)
C(20)	0.1932 (8)	0.3666 (8)	0.7491 (7)	0.070 (2)
C(21)	0.2041 (6)	0.7658 (6)	0.1303 (5)	0.040 (2)
C(22)	0.3339 (7)	0.7702 (7)	0.0779 (6)	0.054 (2)
C(23)	0.2278 (6)	0.7651 (6)	0.2583 (5)	0.036 (2)
C(24)	0.1128 (6)	0.6481 (7)	0.0759 (6)	0.041 (2)
C(25)	0.0418 (6)	0.5543 (7)	0.0284 (6)	0.042 (2)
C(26)	0.1970 (7)	0.6533 (7)	0.3119 (6)	0.049 (2)
C(27)	0.2230 (7)	0.6578 (8)	0.4285 (6)	0.064 (2)
C(28)	0.2810 (7)	0.7741 (8)	0.4910 (7)	0.065 (2)
C(29)	0.3115 (7)	0.8850 (8)	0.4394 (6)	0.062 (2)
C(30)	0.2864 (7)	0.8832 (7)	0.3229 (6)	0.053 (2)

Table 2. *Geometric parameters* (Å, °) *for meso*-(I)

O(11)—C(1)	1.432 (8)	O(12)—C(8)	1.441 (7)
O(21)—C(21)	1.443 (8)	C(1)—C(2)	1.527 (8)
C(1)—C(3)	1.523 (9)	C(1)—C(4)	1.492 (10)
C(3)—C(16)	1.363 (10)	C(3)—C(20)	1.386 (11)
C(4)—C(5)	1.181 (10)	C(5)—C(6)	1.383 (10)
C(6)—C(7)	1.190 (10)	C(7)—C(8)	1.483 (9)
C(8)—C(9)	1.528 (10)	C(8)—C(10)	1.515 (9)
C(10)—C(11)	1.390 (10)	C(10)—C(15)	1.378 (9)
C(11)—C(12)	1.389 (12)	C(12)—C(13)	1.343 (12)
C(13)—C(14)	1.351 (11)	C(14)—C(15)	1.406 (11)
C(16)—C(17)	1.412 (12)	C(17)—C(18)	1.359 (14)
C(18)—C(19)	1.332 (14)	C(19)—C(20)	1.397 (13)
C(21)—C(22)	1.520 (10)	C(21)—C(23)	1.529 (9)
C(21)—C(24)	1.481 (8)	C(23)—C(26)	1.381 (10)
C(23)—C(30)	1.403 (9)	C(24)—C(25)	1.189 (8)
C(25)—C(25*A*)	1.389 (12)	C(26)—C(27)	1.390 (10)
C(27)—C(28)	1.377 (10)	C(28)—C(29)	1.361 (11)
C(29)—C(30)	1.391 (10)		
O(11)—C(1)—C(2)	105.6 (5)	O(11)—C(1)—C(3)	111.0 (5)
C(2)—C(1)—C(3)	109.9 (5)	O(11)—C(1)—C(4)	109.3 (5)
C(2)—C(1)—C(4)	111.5 (5)	C(3)—C(1)—C(4)	109.5 (5)
C(1)—C(3)—C(16)	122.2 (6)	C(1)—C(3)—C(20)	119.0 (6)
C(16)—C(3)—C(20)	118.7 (7)	C(1)—C(4)—C(5)	174.2 (6)
C(4)—C(5)—C(6)	175.7 (7)	C(5)—C(6)—C(7)	174.4 (7)
C(6)—C(7)—C(8)	174.4 (7)	O(12)—C(8)—C(7)	106.0 (5)
O(12)—C(8)—C(9)	106.9 (5)	C(7)—C(8)—C(9)	110.4 (5)
O(12)—C(8)—C(10)	110.2 (5)	C(7)—C(8)—C(10)	112.1 (6)
C(9)—C(8)—C(10)	111.0 (6)	C(8)—C(10)—C(11)	119.0 (6)
C(8)—C(10)—C(15)	122.7 (6)	C(11)—C(10)—C(15)	118.3 (7)
C(10)—C(11)—C(12)	119.9 (7)	C(11)—C(12)—C(13)	120.8 (8)
C(12)—C(13)—C(14)	121.0 (9)	C(13)—C(14)—C(15)	119.5 (7)
C(10)—C(15)—C(14)	120.5 (7)	C(3)—C(16)—C(17)	121.3 (8)
C(16)—C(17)—C(18)	118.3 (9)	C(17)—C(18)—C(19)	121.3 (9)
C(18)—C(19)—C(20)	121.2 (10)	C(3)—C(20)—C(19)	119.1 (8)
O(21)—C(21)—C(22)	107.4 (5)	O(21)—C(21)—C(23)	109.4 (5)
C(22)—C(21)—C(23)	111.3 (5)	O(21)—C(21)—C(24)	108.1 (5)
C(22)—C(21)—C(24)	108.5 (5)	C(23)—C(21)—C(24)	111.9 (5)
C(21)—C(23)—C(26)	123.0 (5)	C(21)—C(23)—C(30)	117.9 (6)
C(26)—C(23)—C(30)	119.1 (6)	C(21)—C(24)—C(25)	177.3 (7)
C(24)—C(25)—C(25*A*)	179.2 (8)	C(23)—C(26)—C(27)	120.5 (6)
C(26)—C(27)—C(28)	119.9 (7)	C(27)—C(28)—C(29)	120.2 (7)
C(28)—C(29)—C(30)	121.0 (7)	C(23)—C(30)—C(29)	119.2 (7)

***rac*-(I)**

Crystal data

$C_{20}H_{18}O_2$
$M_r = 290.3$
Monoclinic
$P2_1/n$
$a = 8.548\ (7)$ Å
$b = 12.546\ (8)$ Å
$c = 15.809\ (10)$ Å
$\beta = 103.54\ (2)°$
$V = 1648\ (2)$ Å^3
$Z = 4$
$D_x = 1.170$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$ - $12.5°$
$\mu = 0.069$ mm^{-1}
$T = 298$ K
Needle
0.3 × 0.2 × 0.2 mm
Colorless

Data collection

Siemens *R*3*m/V* diffractometer
2θ-θ scans
Absorption correction: none
4036 measured reflections
2159 independent reflections
642 observed reflections $[F > 4.0\sigma(F)]$

$R_{\text{int}} = 1.31$
$\theta_{\max} = 22.5°$
$h = 0 \rightarrow 9$
$k = -12 \rightarrow 13$
$l = -17 \rightarrow 16$
3 standard reflections monitored every 50 reflections intensity variation: <5%

Refinement

Refinement on *F*
$R = 0.069$
$wR = 0.0863$
$S = 1.12$
642 reflections
99 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F) + 0.0027F^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for rac*-(I)

U_{iso} for C atoms; $U_{\text{eq}} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for O atoms.

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
O(11)	−0.4131 (8)	−0.3593 (5)	−0.0093 (4)	0.052 (3)
O(12)	−0.0076 (8)	0.0288 (5)	0.3746 (3)	0.049 (3)
C(1)	−0.4535 (13)	−0.2494 (8)	−0.0218 (6)	0.038 (3)
C(2)	−0.4253 (13)	−0.2153 (7)	−0.11063 (6)	0.064 (4)
C(3)	−0.6273 (12)	−0.2292 (8)	−0.0201 (6)	0.039 (3)
C(4)	−0.3448 (13)	−0.1895 (8)	0.0487 (6)	0.046 (3)
C(5)	−0.2586 (14)	−0.1407 (8)	0.1063 (7)	0.050 (3)
C(6)	−0.1575 (14)	−0.0818 (9)	0.1722 (7)	0.055 (3)
C(7)	−0.0743 (13)	−0.0303 (8)	0.2267 (7)	0.050 (3)
C(8)	0.0246 (14)	0.0452 (8)	0.2900 (7)	0.045 (3)
C(9)	−0.0353 (13)	0.1574 (8)	0.2670 (6)	0.070 (4)
C(10)	0.1990 (14)	0.0317 (9)	0.2922 (7)	0.054 (3)
C(11)	0.3084 (19)	0.1002 (11)	0.3449 (9)	0.112 (5)
C(12)	0.4731 (20)	0.0917 (11)	0.3491 (9)	0.121 (6)
C(13)	0.5328 (17)	0.0189 (9)	0.3008 (8)	0.081 (4)
C(14)	0.4267 (15)	−0.0438 (9)	0.2495 (8)	0.076 (4)
C(15)	0.2635 (15)	−0.0397 (9)	0.2452 (7)	0.064 (4)
C(16)	−0.6750 (16)	−0.1325 (9)	0.0097 (7)	0.074 (4)
C(17)	−0.8349 (16)	−0.1126 (10)	0.0091 (7)	0.086 (4)
C(18)	−0.9473 (16)	−0.1882 (9)	−0.0197 (7)	0.071 (4)
C(19)	−0.9063 (15)	−0.2809 (10)	−0.0488 (7)	0.075 (4)
C(20)	−0.7450 (13)	−0.3035 (9)	−0.0496 (6)	0.059 (4)

Table 4. *Geometric parameters* (Å, °) *for rac*-(I)

O(11)—C(1)	1.423 (12)	O(12)—C(8)	1.442 (13)
C(1)—C(2)	1.540 (15)	C(1)—C(3)	1.513 (15)
C(1)—C(4)	1.478 (13)	C(3)—C(16)	1.397 (16)
C(3)—C(20)	1.371 (14)	C(4)—C(5)	1.197 (14)
C(5)—C(6)	1.399 (15)	C(6)—C(7)	1.175 (14)
C(7)—C(8)	1.489 (14)	C(8)—C(9)	1.512 (14)
C(8)—C(10)	1.493 (17)	C(10)—C(11)	1.393 (17)
C(10)—C(15)	1.360 (17)	C(11)—C(12)	1.398 (24)
C(12)—C(13)	1.364 (21)	C(13)—C(14)	1.324 (16)
C(14)—C(15)	1.381 (19)	C(16)—C(17)	1.386 (20)
C(17)—C(18)	1.351 (17)	C(18)—C(19)	1.328 (17)
C(19)—C(20)	1.411 (17)		
O(11)—C(1)—C(2)	108.1 (8)	O(11)—C(1)—C(3)	111.6 (8)
C(2)—C(1)—C(3)	109.5 (8)	O(11)—C(1)—C(4)	107.4 (7)
C(2)—C(1)—C(4)	109.9 (9)	C(3)—C(1)—C(4)	110.3 (9)
C(1)—C(3)—C(16)	121.2 (9)	C(1)—C(3)—C(20)	121.7 (9)
C(16)—C(3)—C(20)	117.2 (10)	C(1)—C(4)—C(5)	179.1 (14)
C(4)—C(5)—C(6)	178.6 (13)	C(5)—C(6)—C(7)	178.5 (11)

C(6)—C(7)—C(8)	173.8 (11)	O(12)—C(8)—C(7)	109.3 (9)
O(12)—C(8)—C(9)	103.3 (8)	C(7)—C(8)—C(9)	108.8 (8)
O(12)—C(8)—C(10)	111.6 (8)	C(7)—C(8)—C(10)	110.5 (9)
C(9)—C(8)—C(10)	113.0 (9)	C(8)—C(10)—C(11)	118.1 (11)
C(8)—C(10)—C(15)	126.4 (10)	C(11)—C(10)—C(15)	115.6 (12)
C(10)—C(11)—C(12)	120.6 (13)	C(11)—C(12)—C(13)	121.9 (13)
C(12)—C(13)—C(14)	116.6 (14)	C(13)—C(14)—C(15)	123.2 (12)
C(10)—C(15)—C(14)	122.1 (10)	C(3)—C(16)—C(17)	121.2 (11)
C(16)—C(17)—C(18)	119.9 (12)	C(17)—C(18)—C(19)	120.5 (13)
C(18)—C(19)—C(20)	121.1 (11)	C(3)—C(20)—C(19)	120.1 (10)

2,7-Diphenyl-3,5-octadiyne-2,7-diol (I) was prepared by treatment of (±)-2-phenyl-3-butyn-2-ol dissolved in methanol containing pyridine with cuprous chloride in an oxygen-containing atmosphere. Diastereomeric (I) was isolated in near quantitative yield as an off-white solid melting over a wide range (313-473 K). Recrystallization from benzene gave the less-soluble diastereomer as colorless needles; m.p. 472-473 K (literature value 465 K; Strauss, Kollek & Hauptmann, 1930). Suitable crystals for X-ray analysis were obtained from the same solvent by slow evaporation.

The mother liquors from *meso*-(I) were systematically pooled and, following several recrystallizations from benzene, produced the more-soluble diastereomer; m.p. 420-421 K (literature value 415-416 K; Popova, 1959). Suitable crystals for X-ray analysis were slowly grown from benzene.

In both cases, a crystal was mounted on a glass fiber in a random orientation and a preliminary examination was carried out. Final cell parameters and an orientation matrix were obtained by least-squares refinement. Axial photographs confirmed the cell dimensions and lattice symmetry; ω scans of representative reflections indicated acceptable crystal quality. No absorption correction was applied. Data reduction, structure solution and refinement were performed using *SHELXTL-Plus* (Sheldrick, 1991). Both structures were solved by direct methods and the remaining non-H atoms were located from subsequent difference Fourier maps. Full-matrix least-squares refinement was carried out by minimizing the function $\Sigma w(F_o - F_c)^2$. H atoms were included in their idealized positions. O atoms were refined to convergence anisotropically and C atoms isotropically.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71404 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1054]

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Di-*tert*-butyl 4-[2-(*tert*-Butoxycarbonyl)ethyl]-4-nitroheptanedioate, $C_{22}H_{39}NO_8$

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Abstract

The molecule has an extended conformation with approximate mirror symmetry. 15 of the 16 *anti* torsion angles are in the range 171.7 (3)–179.1 (4)° in magnitude, with a mean value of 176.6°. The major deviation from mirror symmetry is the C—C—C—C torsion angle in the *tert*-butoxycarbonylethyl group, which is nearly coplanar with the nitro group, having a magnitude of 163.0 (2)°. The C—N bond has a length of 1.555 (3) Å.

Comment

During the synthesis of cascade polymers (Newkome, Nayak, Behera, Moorefield & Baker,