

Triclinic	Cell parameters from 25 reflections
$P\bar{1}$	$\theta = 4.1\text{--}65.0^\circ$
$a = 11.119(1)\text{ \AA}$	$\mu = 3.22\text{ mm}^{-1}$
$b = 6.368(1)\text{ \AA}$	$T = 292(1)\text{ K}$
$c = 5.582(1)\text{ \AA}$	Prismatic
$\alpha = 101.60(1)^\circ$	$0.410 \times 0.205 \times 0.130\text{ mm}$
$\beta = 90.43(1)^\circ$	Pale yellow
$\gamma = 102.40(1)^\circ$	
$V = 377.6(1)\text{ \AA}^3$	
$Z = 2$	
$D_x = 1.383\text{ Mg m}^{-3}$	

Data collection

Rigaku AFC diffractometer	$R_{\text{int}} = 0.029$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 65.04^\circ$
Absorption correction: <i>SHELX76</i> Gaussian (Sheldrick, 1976)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.460$, $T_{\text{max}} = 0.685$	$k = -7 \rightarrow 7$
1182 measured reflections	$l = 0 \rightarrow 6$
844 independent reflections	3 standard reflections monitored every 100 reflections
797 observed reflections [$F > 3\sigma(F)$]	intensity decay: $>1.5\%$

Refinement

Refinement on F^2	$\Delta\rho_{\text{max}} = 0.254\text{ e \AA}^{-3}$
$R = 0.047$	$\Delta\rho_{\text{min}} = -0.245\text{ e \AA}^{-3}$
$wR = 0.066$	Extinction correction: $F_c = F(1 - \chi/\sin\theta)$
$S = 3.01$	Extinction coefficient: $\chi = 0.049(16) \times 10^{-6}$
847 reflections	Atomic scattering factors from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
136 parameters	
All H-atom parameters refined	
$w = 1/[\sigma^2(F) + 0.00036F^2]$	
$(\Delta/\sigma)_{\text{max}} = 0.003$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N1	0.8949 (2)	0.4911 (4)	0.7407 (5)	0.032 (1)
C2	0.9108 (3)	0.2847 (5)	0.7003 (6)	0.033 (1)
O2	0.9751 (2)	0.2123 (4)	0.8316 (5)	0.044 (1)
C3	0.8397 (4)	0.1571 (6)	0.4696 (8)	0.043 (1)
C3a	0.7706 (3)	0.3128 (6)	0.3763 (7)	0.036 (1)
C4	0.6305 (4)	0.2380 (6)	0.3769 (8)	0.047 (1)
S5	0.5905 (1)	0.3370 (1)	0.6862 (2)	0.0456 (5)
C6	0.6946 (3)	0.5965 (5)	0.6854 (7)	0.037 (1)
C6'	0.7174 (4)	0.7511 (7)	0.9336 (8)	0.050 (1)
C6a	0.8104 (3)	0.5350 (6)	0.5665 (7)	0.032 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

N1—C2	1.338 (4)	C3a—C6a	1.564 (5)
N1—C6a	1.454 (4)	C4—S5	1.808 (4)
C2—O2	1.237 (5)	S5—C6	1.803 (4)
C2—C3	1.494 (5)	C6—C6'	1.514 (5)
C3—C3a	1.541 (6)	C6—C6a	1.542 (5)
C3a—C4	1.528 (5)		
C2—N1—C6a	115.4 (2)	C3a—C4—S5	105.7 (3)
O2—C2—N1	125.6 (2)	C6—S5—C4	88.8 (2)
O2—C2—C3	125.6 (3)	C6'—C6—C6a	115.4 (3)
N1—C2—C3	109.0 (2)	C6'—C6—S5	114.1 (3)
C2—C3—C3a	106.9 (3)	C6a—C6—S5	104.9 (2)

C4—C3a—C3	113.0 (3)	N1—C6a—C6	112.7 (2)
C4—C3a—C6a	107.7 (3)	N1—C6a—C3a	104.4 (2)
C3—C3a—C6a	104.3 (3)	C6—C6a—C3a	108.8 (3)

Data collection: Rigaku AFC diffractometer software. Cell refinement: Rigaku AFC diffractometer software. Data reduction: Rigaku AFC diffractometer software. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*. Computer: VAX 8800.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1030). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Altona, C., Geise, H. J. & Romers, C. (1968). *Tetrahedron*, **24**, 13–32.
- DeTitta, G. T., Edmonds, J. W., Stallings, W. & Donohue, J. (1976). *J. Am. Chem. Soc.* **98**, 1920–1926.
- Duax, W. L. & Norton, D. A. (1975). *An Atlas of Steroid Structure*, Vol. 1, pp. 18–199. New York: Plenum Press.
- Etta, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
- Green, N. M. (1963). *Biochem. J.* **89**, 585–591.
- Henderson, S. A., O'Connor, J., Rendina, A. R., Savage, G. P. & Simpson, G. W. (1995). *Aust. J. Chem.* In the press.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Knowles, J. R. (1989). *Ann. Rev. Biochem.* **58**, 195–221.
- Kuroda, Y., Taira, Z., Uno, T. & Osaki, K. (1975). *Cryst. Struct. Commun.* **4**, 325–328.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). **C51**, 2674–2676

1,1-Diphenyl-3-propyn-1-ol

J. GABRIEL GARCIA,* BETHZAIDA RAMOS AND AUGUSTO RODRIGUEZ

Clark Atlanta University, Atlanta, GA 30314, USA

FRANK R. FRONCZEK

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA

(Received 7 April 1995; accepted 20 June 1995)

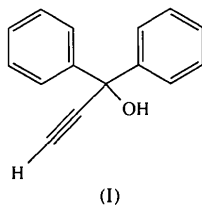
Abstract

The aromatic ring planes in the title compound, C₁₅H₁₂O, are almost mutually orthogonal, forming a dihedral angle of magnitude 94.15 (5)°. The C≡C triple

bond distance is 1.169 (2) Å and the C—O distance is 1.430 (2) Å. There is no O—H···O hydrogen bonding; however, molecules form dimers by O—H··· π interactions about inversion centres.

Comment

The title compound, (I), bears two aromatic rings forming a dihedral angle of magnitude 94.15 (5)°. The acetylenic unit is almost coplanar with one of the aromatic rings, with atoms C2 and C3 lying 0.187 (2) and 0.339 (2) Å, respectively, away from the best plane of C4—C5—C6—C7—C8—C9. Torsion angles C2—C1—C4—C9 and O1—C1—C10—C11 are 11.2 and -6.7 (2)°, respectively. The bond lengths C2—C3 and C1—O1 of the title molecule [1.169 (2) and 1.430 (2) Å, respectively] are comparable with those of 1,1,6,6-tetra-phenylhexa-2,4-diyne-1,6-diol [1.195 (6) and 1.436 (5) Å] (Johnson, Nassimbeni & Toda, 1992), 1,1,6,6-tetrakis(2,4-dimethylphenyl)-2,4-hexadiyne-1,6-diol [1.208 (5) and 1.447 (7) Å], 1,1-bis-(2,4-dimethylphenyl)-2-butyn-1-ol [1.189 (6) and 1.449 (5) Å] and 9-(1-propynyl)-9-fluoreneol [1.16 (2) and 1.47 (1) Å] (Toda, Tanaka & Mak, 1985), and 1-ethynyl-4-hydroperoxy-1,2,3,4-tetrahydro-1-naphthol [1.153 (3) and 1.436 (2) Å] (Moore, Fronczek & Gandour, 1988).



No O—H···O hydrogen bonding is present; the shortest intermolecular O···O distance is 4.907 (2) Å. Lack of such interactions is fairly common for monoalcohols, particularly those in which the OH group is sterically hindered (Brock & Duncan, 1994), an example being 1,1,5,5-tetraphenyl-3,3-dimethyl-1-penten-5-ol, which also has two phenyl groups on the C atom carrying the OH group (Zimmerman & Zuraw, 1989). In the absence of O—H···O interactions, the title compound forms dimers by O—H··· π interactions (Jeffrey & Saenger, 1991) about the inversion centre at $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$. The phenyl group involved in this intermolecular interaction is that of C4—C9. The O1···centroid distance is 3.473 (2) Å, the H···centroid distance is 2.65 (2) Å, and the O—H···centroid angle is 172 (2)°. The propyne C≡CH group forms long intermolecular interactions with two hydroxy O atoms: C3···O1' ($x, y - 1, z$) 3.420 (2) and C3···O1'' ($\frac{3}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$) 3.523 (2) Å. In this bifurcated contact, the H atom lies approximately midway between the two O atoms, with H···O' 3.16 (2) and H···O'' 3.01 (2) Å, and an angle about H of 162 (2)°. These interactions are illustrated in Fig. 2.

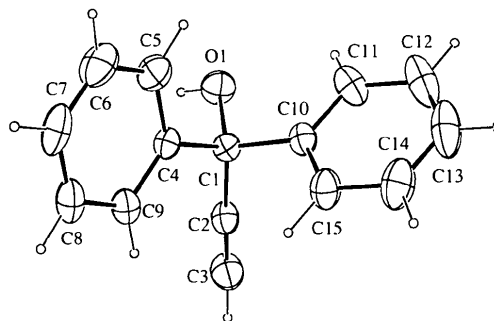


Fig. 1. Molecular structure showing 30% probability ellipsoids. H atoms are illustrated by circles of arbitrary radii.

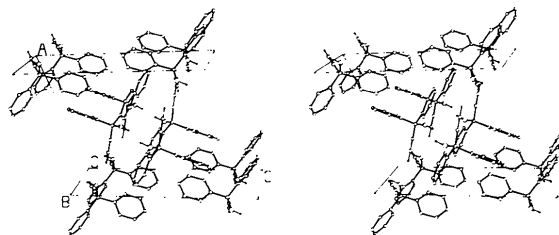


Fig. 2. Stereoview of the unit cell viewed slightly obliquely to b. Phenyl H atoms are omitted.

Experimental

The title compound was prepared by K₂CO₃/methanol deprotection (Austin, Bilow, Kelleghan & Lau, 1981) of the corresponding trimethylsilyl protected derivative.

Crystal data

C₁₅H₁₂O
M_r = 208.26
 Monoclinic
*P*2₁/*n*
a = 12.131 (1) Å
b = 6.1670 (5) Å
c = 16.507 (2) Å
 β = 108.69 (1)°
V = 1169.8 (4) Å³
Z = 4
D_x = 1.182 Mg m⁻³

Cu K α radiation
 λ = 1.54184 Å
 Cell parameters from 25 reflections
 θ = 26–30°
 μ = 0.534 mm⁻¹
T = 296 K
 Needle fragment
 0.20 × 0.20 × 0.13 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\theta/2\theta$ scans
 Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
 T_{\min} = 0.932, T_{\max} = 0.996
 2750 measured reflections
 2240 independent reflections

1712 observed reflections [$I > 3.0\sigma(I)$]
 R_{int} = 0.031
 θ_{max} = 74.92°
 $h = 0 \rightarrow 15$
 $k = 0 \rightarrow 7$
 $l = -20 \rightarrow 19$
 3 standard reflections
 frequency: 120 min
 intensity decay: 11.5%

Refinement

Refinement on F	$\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$
R = 0.045	$\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$
wR = 0.054	Extinction correction:
S = 2.566	isotropic (Zachariasen, 1963)
1712 reflections	Extinction coefficient:
194 parameters	$0.232(2) \times 10^{-4}$
H atoms refined isotropically	Atomic scattering factors
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$	from <i>International Tables for X-ray Crystallography</i> (1974, Vol. IV)
$(\Delta/\sigma)_{\max} = 0.021$	

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms; $U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^*$ for all others.

	x	y	z	$U_{\text{eq}}/U_{\text{iso}}$
O1	0.55026 (9)	0.6516 (2)	0.64473 (7)	0.0624 (3)
C1	0.5015 (1)	0.4492 (3)	0.65840 (9)	0.0428 (4)
C2	0.5938 (1)	0.2828 (3)	0.68246 (9)	0.0501 (4)
C3	0.6678 (1)	0.1537 (4)	0.7050 (1)	0.0717 (6)
C4	0.4016 (1)	0.3851 (3)	0.57852 (8)	0.0443 (4)
C5	0.3183 (1)	0.5401 (3)	0.5415 (1)	0.0674 (6)
C6	0.2248 (2)	0.4888 (4)	0.4706 (1)	0.0858 (7)
C7	0.2133 (2)	0.2834 (4)	0.4365 (1)	0.0798 (6)
C8	0.2951 (2)	0.1297 (3)	0.4724 (1)	0.0695 (5)
C9	0.3902 (1)	0.1803 (3)	0.54372 (9)	0.0547 (5)
C10	0.4565 (1)	0.4806 (3)	0.73422 (8)	0.0471 (4)
C11	0.4766 (1)	0.6685 (3)	0.7816 (1)	0.0715 (6)
C12	0.4373 (2)	0.6863 (4)	0.8516 (1)	0.0987 (7)
C13	0.3783 (2)	0.5217 (5)	0.8736 (1)	0.1005 (8)
C14	0.3572 (2)	0.3347 (5)	0.8266 (1)	0.0903 (7)
C15	0.3965 (1)	0.3132 (3)	0.7571 (1)	0.0662 (5)
H10H	0.582 (2)	0.641 (3)	0.608 (1)	0.121 (8)
H3	0.722 (2)	0.039 (4)	0.722 (1)	0.126 (8)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O1—C1	1.430 (2)	C1—C4	1.530 (2)
O1—H10H	0.83 (2)	C1—C10	1.530 (2)
C1—C2	1.476 (2)	C2—C3	1.169 (2)
C1—O1—H10H	111 (1)	C2—C1—C10	107.9 (1)
O1—C1—C2	109.5 (1)	C4—C1—C10	110.2 (1)
O1—C1—C4	110.1 (1)	C1—C2—C3	177.2 (2)
O1—C1—C10	107.0 (1)	C2—C3—H3	174 (1)
C2—C1—C4	112.1 (1)		

The 12 phenyl C—C distances have a mean value of 1.377 \AA ; individual e.s.d.'s are 0.002–0.004 \AA , and the e.s.d. of their distribution is 0.010 \AA . Isotropic displacement parameters for the H atoms are in the range 5.9 (4)–11.2 (7) \AA^2 , and C—H distances are 0.90 (2)–1.04 (2) \AA .

Data collection: CAD-4 (Enraf–Nonius, 1977). Cell refinement: CAD-4. Data reduction: PROCESS in MolEN (Fair, 1990). Program(s) used to solve structure: MULTAN (Main *et al.*, 1980) (direct methods). Program(s) used to refine structure: LSFM in MolEN (Fair, 1990). Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: CIF in MolEN (Fair, 1990).

The purchase of the diffractometer was made possible by a National Science Foundation chemical instrumentation grant, which we gratefully acknowledge.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1146). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Austin, W. B., Bilow, N., Kelleghan, W. J. & Lau, K. S. Y. (1981). *J. Org. Chem.* **46**, 2280–2286.
- Brock, C. P. & Duncan, L. L. (1994). *Chem. Mater.* **6**, 1307–1312.
- Enraf–Nonius (1977). *CAD-4 Operations Manual*. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*. Berlin: Springer-Verlag.
- Johnson, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Johnson, L., Nassimbeni, L. R. & Toda, F. (1992). *Acta Cryst.* **B48**, 827–832.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Moore, J. A., Fronczek, F. R. & Gandour, R. D. (1988). *Acta Cryst.* **C44**, 2027–2028.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Toda, F., Tanaka, K. & Mak, T. C. W. (1985). *Bull. Chem. Soc. Jpn.* **58**, 2221–2227.
- Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.
- Zimmerman, H. E. & Zuraw, M. J. (1989). *J. Am. Chem. Soc.* **111**, 7974–7989.

Acta Cryst. (1995). **C51**, 2676–2680

Absolute Configuration of Mutilin, 5-Acetylmutilin and 5-Bromoacetylmutilin

TULLIO PILATI

CNR - Centro per lo Studio delle Relazioni fra Struttura e Reattività Chimica, Via Golgi 19, 20133 Milan, Italy

GIANCARLO CRAVOTTO AND GIOVANNI PALMISANO

Dipartimento di Scienza e Tecnologia del Farmaco, Università di Torino, Via P. Giuria 9, 10125 Turin, Italy

(Received 18 May 1995; accepted 26 June 1995)

Abstract

The structures of the tricyclic diterpenoids mutilin, C₂₀H₃₂O₃, (1), 5-acetylmutilin, C₂₂H₃₄O₄, (2), and 5-bromoacetylmutilin, C₂₂H₃₃BrO₄, (3), are reported (mu-