References

ADAMS, R. D., COLLINS, D. M. & COTTON, F. A. (1974). *Inorg. Chem.* **13**, 1086–1090.

BREEZE, R., RICALTON, A. & WHITELEY, M. W. (1987). *J. Organomet. Chem.* **327**, C29–C32.

CHURCHILL, M. R. & O'BRIEN, T. A. (1969). *J. Chem. Soc. A*, pp. 1110–1115.

CLEGG, W., COMPTON, N. A., ERRINGTON, R. J. & NORMAN, N. C. (1988). *Acta Cryst.* C**44**, 568–570.

JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

ZIEGLER, M. L., SASSE, H. E. & NUBER, B. (1975). *Z. Naturforsch. Teil B*, **30**, 26–29.
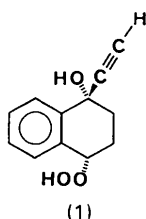
1-Ethynyl-4-hydroperoxy-1,2,3,4-tetrahydro-1-naphthol

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Abstract. $C_{12}H_{12}O_3$, $M_r = 204.2$, monoclinic, $P2_1/c$, $a = 11.815$ (2), $b = 7.673$ (2), $c = 12.580$ (3) Å, $\beta = 115.06$ (2)°, $V = 1033.1$ (9) Å³, $Z = 4$, $D_x = 1.313$ g cm⁻³, $\lambda(\text{Cu }K\alpha) = 1.54184$ Å, $\mu = 7.34$ cm⁻¹, $F(000) = 432$, $T = 299$ K, $R = 0.065$ for 1420 observations (of 2131 unique data). The stereochemistry of the hydroxyl and hydroperoxyl groups is *cis*. The hydroperoxyl O—O bond distance is 1.476 (2) Å. The hydrogen-bonding pattern consists of two intermolecular interactions: a hydroperoxyl donor to the hydroxyl group, with an O···O distance of 2.818 (2) Å and an O—H···O angle of 168 (2)°, and a hydroxyl donor to O(3) of the hydroperoxyl group, with a distance and angle of 2.670 (2) Å and 170 (2)°, respectively.

Experimental. Colorless needles, m.p. 426–428 K, of 1-ethynyl-4-hydroperoxy-1,2,3,4-tetrahydro-1-naphthol (1) were isolated

(1)

from a mixture of 1-tetralone and 1-ethynyl-1,2,-3,4-tetrahydro-1-naphthol in benzene, which was allowed to evaporate slowly over a period of 3 weeks. The rate of autooxidation of tetralins is increased by the

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac\beta_{13}\cos\beta).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O(1)	0.1313 (2)	−0.0616 (3)	0.1309 (2)	5.26 (6)
O(2)	0.1673 (2)	0.2560 (3)	0.4530 (2)	4.83 (5)
O(3)	0.0869 (2)	0.2583 (3)	0.5163 (2)	5.29 (6)
C(1)	0.2937 (2)	0.0282 (4)	0.3128 (2)	3.22 (6)
C(2)	0.3753 (2)	0.0570 (5)	0.2595 (2)	3.83 (7)
C(3)	0.4782 (2)	0.1620 (5)	0.3112 (2)	4.42 (8)
C(4)	0.5045 (2)	0.2409 (5)	0.4170 (2)	4.21 (8)
C(5)	0.4244 (3)	0.2145 (5)	0.4706 (2)	3.94 (7)
C(6)	0.3190 (2)	0.1100 (4)	0.4190 (2)	3.33 (6)
C(7)	0.2333 (2)	0.0902 (4)	0.4803 (2)	4.00 (7)
C(8)	0.1440 (3)	−0.0599 (5)	0.4339 (3)	5.18 (8)
C(9)	0.0840 (2)	−0.0611 (5)	0.3019 (3)	5.01 (8)
C(10)	0.1830 (2)	−0.0931 (4)	0.2550 (2)	3.72 (7)
C(11)	0.2239 (2)	−0.2761 (4)	0.2748 (2)	3.80 (7)
C(12)	0.2504 (3)	−0.4217 (5)	0.2874 (3)	5.52 (9)

presence of a ketone (Robertson & Waters, 1948). Crystal size 0.12 × 0.16 × 0.40 mm, space group from systematic absences $h0l$ with l odd and $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0.59–3.28° min⁻¹. Data having $2 < \theta < 75°$, $0 \le h \le 14$, $0 \le k \le 9$, $−15 \le l \le 15$ measured. Data corrected for background, Lorentz, polarization, decay and absorption effects. Absorption corrections were based on ψ scans, with a minimum relative transmission coefficient of 69.90%. Standard reflections 200, 060, 004 indicated a 14.1% decay and a linear correction was applied. $R_{int} = 0.033$ for averaging $0kl$ and $0k\bar{l}$ data, 2131 unique data, 1420

Table 2. Bond distances (Å), angles (°) and selected torsion angles (°)

O(1)		C(10)	1·436 (2)	C(4)		C(5)	1·390 (2)
O(2)		O(3)	1·476 (2)	C(5)		C(6)	1·389 (2)
O(2)		C(7)	1·455 (2)	C(6)		C(7)	1·518 (2)
C(1)		C(2)	1·406 (2)	C(7)		C(8)	1·503 (3)
C(1)		C(6)	1·390 (2)	C(8)		C(9)	1·504 (3)
C(1)		C(10)	1·516 (2)	C(9)		C(10)	1·536 (2)
C(2)		C(3)	1·371 (3)	C(10)		C(11)	1·471 (3)
C(3)		C(4)	1·373 (3)	C(11)		C(12)	1·153 (3)
O(3)	O(2)	C(7)	106·6 (1)	O(2)	C(7)	C(8)	111·2 (2)
C(2)	C(1)	C(6)	118·4 (2)	C(6)	C(7)	C(8)	112·8 (2)
C(2)	C(1)	C(10)	119·6 (2)	C(7)	C(8)	C(9)	111·1 (2)
C(6)	C(1)	C(10)	121·9 (1)	C(8)	C(9)	C(10)	110·2 (2)
C(1)	C(2)	C(3)	121·1 (2)	O(1)	C(10)	C(1)	107·6 (1)
C(2)	C(3)	C(4)	120·7 (2)	O(1)	C(10)	C(9)	110·3 (2)
C(3)	C(4)	C(5)	119·0 (2)	O(1)	C(10)	C(11)	107·6 (2)
C(4)	C(5)	C(6)	121·2 (2)	C(1)	C(10)	C(9)	111·1 (2)
C(1)	C(6)	C(5)	119·7 (1)	C(1)	C(10)	C(11)	110·5 (1)
C(1)	C(6)	C(7)	121·7 (2)	C(9)	C(10)	C(11)	109·6 (2)
C(5)	C(6)	C(7)	118·7 (2)	C(10)	C(11)	C(12)	176·9 (2)
O(2)	C(7)	C(6)	101·5 (1)				

H(10)	O(1)	C(10)	C(1)	179 (3)
H(10)	O(1)	C(10)	C(9)	−60 (3)
H(10)	O(1)	C(10)	C(11)	60 (3)
C(7)	O(2)	O(3)	H(30)	127 (2)
O(3)	O(2)	C(7)	C(6)	−175·7 (2)
O(3)	O(2)	C(7)	C(8)	64·1 (2)
O(2)	C(7)	C(8)	C(9)	67·0 (3)
C(6)	C(7)	C(8)	C(9)	−46·2 (4)
C(7)	C(8)	C(9)	C(10)	64·7 (4)
C(8)	C(9)	C(10)	O(1)	−169·0 (3)
C(8)	C(9)	C(10)	C(1)	−49·8 (4)
C(8)	C(9)	C(10)	C(11)	72·6 (3)

Fig. 1. *ORTEP* (Johnson, 1965) drawing of title compound.

Related literature. Autooxidation of tetralin: Hock & Susemihl (1933). Structural and steric effects on oxidation of alkylarenes: Voronenkov, Vinogradov & Belyaev (1970). Kinetics of tetralin oxidation: Woodward & Mesrobian (1953). Intermolecular hydrogen bonding of hydrocarbon hydroperoxides: Yablonskii, Belyaev & Vinogradov (1972). Oxidation of 1-substituted tetralins: Yeomans & Young (1958).

observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN*78 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), 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 Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Nonhydrogen atoms refined anisotropically; H atoms located by ΔF and refined isotropically, with the isotropic thermal parameter of the peroxyl hydrogen fixed at $B = 8$ Å². Final $R = 0·065$, $wR = 0·078$, $R = 0·095$ for all data, $S = 3·444$ for 184 variables. Maximum shift 0·07σ in the final cycle, max. residual density 0·22, min. −0·29 e Å⁻³, extinction coefficient $g = 6·5$ (6) × 10⁻⁶, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. Coordinates are given in Table 1;* bond distances, angles, and torsion angles are given in Table 2. The molecule is illustrated in Fig. 1.

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* Tables of H-atom coordinates, distances and angles involving H-atoms, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51189 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
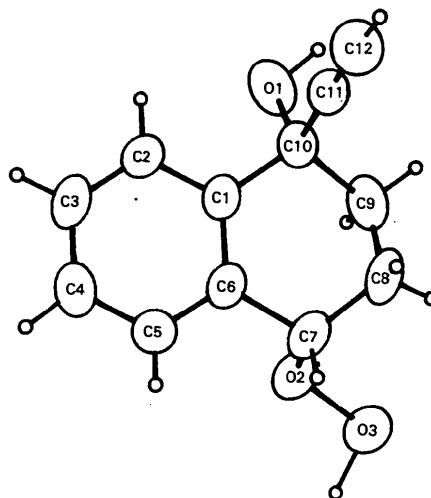
References

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

HOCK, H. & SUSEMIHL, W. (1933). *Chem. Ber.* **66**, 61–68.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA.

MAIN, P., FISKE, J. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

ROBERTSON, A. & WATERS, W. A. (1948). *J. Chem. Soc.* pp. 1574–1578.

VORONENKOV, V. V., VINOGRADOV, A. N. & BELYAEV, V. A. (1970). *Russ. Chem. Rev.* **39**(11), 944–953.

WOODWARD, A. E. & MESROBIAN, R. B. (1953). *J. Am. Chem. Soc.* **75**(24), 2189–2195.

YABLONSKII, O. P., BELYAEV, V. A. & VINOGRADOV, A. N. (1972). *Russ. Chem. Rev.* **41**(7), 565–573.

YEOMANS, B. & YOUNG, D. P. (1958). *J. Chem. Soc.* pp. 2288–2293.