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Methyl 3,4-Dihydro-1-oxo-2(1*H*)-naphthylidenehydroxyacetate

By Philippe Prince, Frank R. Fronczek and Richard D. Gandour*

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

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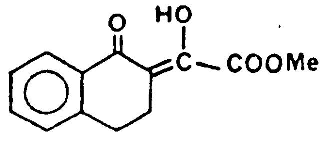
Abstract. $C_{13}H_{12}O_4$, $M_r = 232{\cdot}2$, monoclinic, $P2_1/c$, $a = 8{\cdot}499\,(2)$, $b = 13{\cdot}798\,(4)$, $c = 9{\cdot}652\,(2)$ Å, $\beta = 98{\cdot}25\,(2)°$, $V = 1120{\cdot}1\,(9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}377$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54184$ Å, $\mu = 8{\cdot}12$ cm^{-1}, $F(000) = 488$, $T = 295$ K, $R = 0{\cdot}042$ for 1916 observations (of 2296 unique data). The molecule contains an enol subunit, the hydroxy group of which forms an intramolecular hydrogen bond to the cyclic carbonyl group, with an O···O distance of 2·4782 (14) Å and an O—H···O angle of 143 (3)°. The refined position of the hydrogen atom is 1·16 (4) Å from the hydroxy oxygen and 1·44 (4) Å from the carbonyl oxygen, and the isotropic thermal parameter is large; $B = 13\,(1)$ Å^2. The enol C=C bond distance is 1·371 (1) Å and its C—OH distance is 1·312 (1) Å.

Experimental. Pale yellow crystals of (1), m.p. 337 K, were isolated, by two successive recrystallizations in methanol, from the crude reaction product of dimethyl oxalate and 1-tetralone with sodium methoxide in toluene at room temperature (Brown, Touet & Ragault, 1972). Crystal size 0·30 × 0·35 × 0·50 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 $I = 25\sigma(I)$, subject to max. scan time = 60 s, scan rates varied 1·27–3·28° min^{-1}. One hemisphere of data having $2 < \theta < 75°$, $-10 \le h \le 10$, $0 \le k \le 17$, $-12 \le l \le 12$ 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 87·13%. Standard reflections $\bar{4}23$, 060, 002 indicated an 8·4% decay, for which a linear correction was applied. The two octants (4264 measurements) merged, $R_{\mathrm{int}} = 0{\cdot}015$, to yield 2296 unique data, 1916 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ using *Enraf–Nonius SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-hydrogen atoms refined anisotropically; hydrogen atoms located by ΔF and refined isotropically. Final $R = 0{\cdot}042$, $wR = 0{\cdot}058$, $S = 2{\cdot}582$ for 203 variables. Maximum shift $0{\cdot}15\sigma$ in the final cycle, max. and min. residual density 0·20 and $-0{\cdot}16$ e Å^{-3} respectively, extinction coefficient $g = 2{\cdot}6\,(2) \times 10^{-6}$ 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.



(1)

Table 1. *Coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}(Å^2)*
O(1)	−0·0044 (1)	0·40046 (8)	0·80170 (9)	4·29 (2)
O(2)	0·1998 (1)	0·4010 (1)	0·64240 (9)	5·00 (2)
O(3)	0·4971 (1)	0·40727 (8)	0·6050 (1)	4·46 (2)
O(4)	0·5620 (1)	0·30791 (9)	0·7871 (1)	5·04 (2)
C(1)	0·1026 (2)	0·38634 (9)	0·9048 (1)	3·33 (2)
C(2)	0·0575 (2)	0·37556 (9)	1·0455 (1)	3·47 (2)
C(3)	−0·1020 (2)	0·3584 (1)	1·0609 (2)	4·30 (3)
C(4)	−0·1435 (2)	0·3482 (1)	1·1932 (2)	5·29 (3)
C(5)	−0·0286 (2)	0·3547 (1)	1·3098 (2)	5·27 (3)
C(6)	0·1289 (2)	0·3722 (1)	1·2959 (1)	4·65 (3)
C(7)	0·1731 (2)	0·3833 (1)	1·1636 (1)	3·73 (2)
C(8)	0·3413 (2)	0·4072 (1)	1·1433 (1)	4·56 (3)
C(9)	0·3881 (2)	0·3575 (1)	1·0150 (1)	4·42 (3)
C(10)	0·2680 (2)	0·3771 (1)	0·8863 (1)	3·30 (2)
C(11)	0·3044 (2)	0·3818 (1)	0·7527 (1)	3·50 (2)
C(12)	0·4696 (2)	0·3612 (1)	0·7194 (1)	3·60 (2)
C(13)	0·6508 (2)	0·3892 (1)	0·5592 (2)	5·08 (3)

* The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $\frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac\beta_{13}\cos\beta]$.

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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 51562 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* To whom correspondence should be addressed.

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

O(1)—C(1)	1·263 (1)	C(3)—C(4)	1·380 (1)
O(2)—C(11)	1·312 (1)	C(4)—C(5)	1·383 (2)
O(3)—C(12)	1·324 (1)	C(5)—C(6)	1·385 (2)
O(3)—C(13)	1·459 (1)	C(6)—C(7)	1·390 (1)
O(4)—C(12)	1·199 (1)	C(7)—C(8)	1·507 (1)
C(1)—C(2)	1·471 (1)	C(8)—C(9)	1·517 (1)
C(1)—C(10)	1·447 (1)	C(9)—C(10)	1·515 (1)
C(2)—C(3)	1·404 (1)	C(10)—C(11)	1·371 (1)
C(2)—C(7)	1·398 (1)	C(11)—C(12)	1·511 (1)
O(2)—H(20)	1·16 (3)		

C(12)—O(3)—C(13)	116·39 (8)	C(6)—C(7)—C(8)	122·00 (9)
O(1)—C(1)—C(2)	119·30 (8)	C(7)—C(8)—C(9)	111·82 (8)
O(1)—C(1)—C(10)	121·21 (8)	C(8)—C(9)—C(10)	111·34 (8)
C(2)—C(1)—C(10)	119·45 (7)	C(1)—C(10)—C(9)	117·81 (8)
C(1)—C(2)—C(3)	119·88 (8)	C(1)—C(10)—C(11)	117·80 (7)
C(1)—C(2)—C(7)	119·94 (8)	C(9)—C(10)—C(11)	124·31 (8)
C(3)—C(2)—C(7)	120·18 (8)	O(2)—C(11)—C(10)	123·70 (8)
C(2)—C(3)—C(4)	119·6 (1)	O(2)—C(11)—C(12)	114·04 (7)
C(3)—C(4)—C(5)	120·1 (1)	C(10)—C(11)—C(12)	122·22 (7)
C(4)—C(5)—C(6)	120·8 (1)	O(3)—C(12)—O(4)	124·64 (8)
C(5)—C(6)—C(7)	120·0 (1)	O(3)—C(12)—C(11)	111·61 (7)
C(2)—C(7)—C(6)	119·29 (9)	O(4)—C(12)—C(11)	123·72 (8)
C(2)—C(7)—C(8)	118·67 (8)		
C(11)—O(2)—H(20)	104 (1)		

C(13)—O(3)—C(12)—O(4)	1·0 (2)	C(8)—C(9)—C(10)—C(1)	−34·0 (2)
C(13)—O(3)—C(12)—C(11)	−177·29 (12)	C(8)—C(9)—C(10)—C(11)	149·46 (14)
O(1)—C(1)—C(2)—C(7)	−164·59 (13)	C(1)—C(10)—C(11)—O(2)	5·1 (2)
C(10)—C(1)—C(2)—C(7)	17·6 (2)	C(1)—C(10)—C(11)—C(12)	−172·59 (12)
O(1)—C(1)—C(10)—C(9)	−178·37 (13)	C(9)—C(10)—C(11)—O(2)	−178·36 (14)
O(1)—C(1)—C(10)—C(11)	−1·6 (2)	C(9)—C(10)—C(11)—C(12)	3·95 (21)
C(2)—C(1)—C(10)—C(9)	−0·6 (2)	O(2)—C(11)—C(12)—O(3)	28·87 (17)
C(2)—C(1)—C(10)—C(11)	176·15 (12)	O(2)—C(11)—C(12)—O(4)	−149·47 (14)
C(1)—C(2)—C(7)—C(8)	2·74 (20)	C(10)—C(11)—C(12)—O(3)	−153·24 (13)
C(2)—C(7)—C(8)—C(9)	−38·0 (2)	C(10)—C(11)—C(12)—O(4)	28·42 (21)
C(7)—C(8)—C(9)—C(10)	52·2 (2)		

Related literature. Structure of 2-acetyl-1-tetralone: Geoffroy, Jain, Celalyan & Bernardinelli (1983); structures of several tetracycline derivatives: Stezowski (1976).

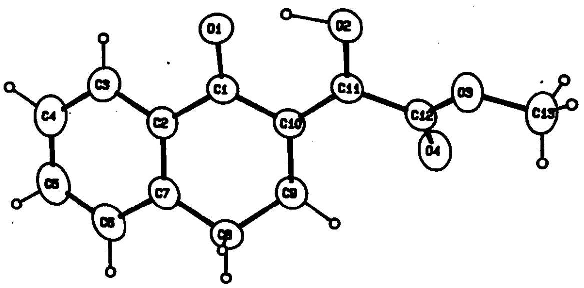

Fig. 1. Numbering scheme of title compound; thermal ellipsoids are drawn at the 40% probability level. H atoms are drawn as circles with the same arbitrary radius.

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References

Brown, E., Touet, J. & Ragault, M. (1972). *Bull. Soc. Chim. Fr.* pp. 212–220.

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.

Geoffroy, M., Jain, A., Celalyan, A. & Bernardinelli, G. (1983). *Z. Naturforsch. Teil B,* **38**, 830–834.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Stezowski, J. J. (1976). *J. Am. Chem. Soc.* **98**, 6012–6018.

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Structure of *N-tert*-Butoxycarbonyl-L-tyrosine 4-Bromophenacyl Ester

By J. Matsoukas, M. Semertzidis, J. Hondrelis, V. Nastopoulos and S. Voliotis*

*Department of Chemistry, University of Patras, GR-*26110 *Patras, Greece*

and Ivan Leban

Department of Chemistry and Chemical Technology, Edvard Kardelj University, Murnikova 6, *PO Box* 537, 61001 *Ljubljana, Yugoslavia*

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Abstract. $C_{22}H_{24}BrNO_6$, $M_r = 478{\cdot}3$, monoclinic, $P2_1$, $a = 5{\cdot}456\ (1)$, $b = 10{\cdot}158\ (1)$, $c = 20{\cdot}239\ (5)$ Å, $\beta = 95{\cdot}83\ (2)°$, $V = 1115{\cdot}9\ (6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}423$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 19{\cdot}89$ cm^{-1}, $F(000) = 492$, room temperature, $R = 0{\cdot}085$ for 962 unique observed reflections. The urethane amide bond

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* To whom all correspondence should be addressed.