We thank Professor C. E. Nordman and a referee for their helpful comments. Computational facilities were provided by the Ohio State University IRCC.

References

ALCOCK, N. W. (1970). Crystallographic Computing, edited by F. R. AHMED, pp. 271–278. Copenhagen: Munksgaard.

BANERJEE, A. (1973). Acta Cryst. B29, 2070–2074.

CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.

DIDEBERG, O., DUPONT, L. & ANDRÉ, J. M. (1972). Acta Cryst. B28, 1002–1007.

GOLDBERG, I. & SHMUELI, U. (1973). Acta Cryst. B29, 432–440.

HANSON, A. W. (1965). Acta Cryst. 19, 19–26.

HOPE, H., KNOBLER, C. & MCCULLOUGH, J. D. (1970). Acta Cryst. B26, 625–640.

HOWELLS, E. R., PHILLIPS, D. C. & ROGERS, D. (1950). Acta Cryst. 3, 210–214.

JOHNSON, C. K. (1971). ORTEPII. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee.

MCCULLOUGH, J. D. (1975). Inorg. Chem. 14, 2639–2643.

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.

SCHAFFRIN, R. M. & TROTTER, J. (1970). J. Chem. Soc. A, pp. 1561–1565.

SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.

STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. W. & HALL, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). J. Chem. Phys. 42, 3175–3187.

STEZOWSKI, J. J., BINDER, W. & KARL, N. (1982). Acta Cryst. B38, 2912–2914.

1-Phenyl-1,2-dihydro-2-quinolinone, C15H11NO

BY NORMAN C. BAENZIGER AND STANLEY WAWZONEK

Department of Chemistry, University of Iowa, Iowa City, Iowa 52242, USA

Abstract. $M_r = 221 \cdot 26$, $P2_1/c$, $a = 5 \cdot 621$ (1), $b = 16 \cdot 098$ (5), $c = 12 \cdot 368$ (3) Å, $\beta = 98 \cdot 74$ (2)°, $V = 1106 \cdot 1$ Å^3, $Z = 4$, $D_x = 1 \cdot 329$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0 \cdot 70926$ Å, $\mu = 0 \cdot 909$ cm^{-1}, $F(000) = 464$, 298 K, $R_1 = 0 \cdot 042$ based on 1595 observed reflections. The phenyl ring attached to N(1) is rotated to make a dihedral angle of $71 \cdot 9$ (1)° with the plane of the quinoline ring, orienting the ring electrons to account for the abnormal chemical shifts in the proton NMR spectrum. The C(3)—C(4) distance of $1 \cdot 333$ (3) Å indicates the localization of a double bond at this position.

Introduction. The crystal structure analysis of 1-phenyl-1,2-dihydro-2-quinolinone was undertaken primarily to verify the position of attachment of the phenyl group (both N and O sites are feasible) and to help to understand an unusual chemical shift in its proton NMR spectrum.

Experimental. The crystal was grown from ethanol solution. Intensity data collected with Picker FACS-I four-circle diffractometer, graphite-monochromatized Mo $K\alpha_1$ radiation, stepped 2θ scan (basic step size $= 0 \cdot 10°$ 2θ); $h \pm 6$, $k \pm 19$, $l \pm 14$ (Baenziger, Foster, Howells, Howells, Vander Valk & Burton, 1977); total reflections measured $= 11\,050$, $\sin \theta/\lambda_{max} = 0 \cdot 59$ Å^{-1}, 1959 unique reflections, 1595 greater than 3σ. No absorption correction $[\mu = 0 \cdot 91$ cm^{-1} and crystal dimensions $\{011\}$ $0 \cdot 22$, (100) $1 \cdot 3$ mm (φ axis approximately parallel to $\mathbf{a}^*$) small]. $R_{int} = 0 \cdot 045$ based on F^2; 3 standard reflections varied $< 3\%$. Cell dimensions determined from 12 carefully centered reflections in 2θ range 36 to 47°. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971) and electron density map methods. All hydrogen atoms located in electron density difference maps. Atom scattering factors for O, N and C from *International Tables for X-ray Crystallography* (1974); for H from Stewart, Davidson & Simpson (1965). Max. and min. electron densities in final difference map $= 0 \cdot 1$ e Å^{-3}. Full-matrix least-squares refinement (local adaption of programs from Lawrence Radiation Laboratories, Berkeley) including hydrogen atoms (isotropic) and extinction parameter gave discrepancy factors $R_1 = 0 \cdot 042$, $R_2 = 0 \cdot 070$, $S = 1 \cdot 80$ based on 1595 reflections and 199 parameters. $w = 1/(s^2_F + 0 \cdot 06\,F^2)$, where s_F is the larger of the two estimates of standard deviation in F obtained from counting statistics and agreement among equivalent reflections. Max. $\Delta/\sigma = 0 \cdot 013$ for nonhydrogen atoms and $0 \cdot 066$ for H atoms in the last cycle.

Table 1. *Atomic coordinates for* 1-*phenyl*-1,2-*dihydro-2-quinolinone*

[*B* values for the nonhydrogen atoms = $\frac{1}{3}$ trace **B**.]

	x	y	z	B
N(1)	0·0007 (3)	0·59147 (9)	0·2861 (1)	2·9
C(2)	−0·2041 (4)	0·6094 (1)	0·2114 (2)	3·3
C(3)	−0·2641 (4)	0·5503 (2)	0·1237 (2)	4·2
C(4)	−0·1342 (4)	0·4820 (2)	0·1150 (2)	4·3
C(5)	0·2163 (5)	0·3932 (1)	0·1849 (2)	4·3
C(6)	0·4178 (5)	0·3783 (1)	0·2589 (2)	4·2
C(7)	0·4850 (4)	0·4347 (1)	0·3423 (2)	3·9
C(8)	0·3486 (4)	0·5048 (1)	0·3534 (2)	3·4
C(9)	0·0759 (4)	0·4645 (1)	0·1920 (2)	3·4
C(10)	0·1433 (3)	0·5205 (1)	0·2780 (1)	2·8
O(11)	−0·3205 (3)	0·67280 (9)	0·2212 (1)	4·0
C(12)	0·0630 (3)	0·6488 (1)	0·3763 (1)	2·8
C(13)	0·2601 (3)	0·7002 (1)	0·3792 (2)	3·3
C(14)	0·3201 (4)	0·7538 (1)	0·4665 (2)	3·9
C(15)	0·1799 (4)	0·7570 (1)	0·5489 (2)	3·9
C(16)	−0·0169 (4)	0·7058 (1)	0·5450 (2)	4·1
C(17)	−0·0750 (4)	0·6512 (1)	0·4593 (2)	3·6
H(3)	−0·398 (5)	0·564 (2)	0·074 (2)	5·3 (6)
H(4)	−0·190 (6)	0·443 (2)	0·053 (2)	7·1 (8)
H(5)	0·164 (5)	0·353 (2)	0·128 (2)	5·0 (6)
H(6)	0·513 (5)	0·330 (2)	0·252 (2)	5·3 (6)
H(7)	0·628 (5)	0·424 (1)	0·399 (2)	4·9 (5)
H(8)	0·386 (5)	0·542 (1)	0·413 (2)	4·5 (5)
H(13)	0·360 (4)	0·696 (1)	0·323 (2)	3·7 (4)
H(14)	0·462 (5)	0·789 (1)	0·470 (2)	4·5 (5)
H(15)	0·226 (4)	0·798 (1)	0·608 (2)	3·9 (5)
H(16)	−0·114 (5)	0·707 (2)	0·600 (2)	5·4 (6)
H(17)	−0·206 (5)	0·612 (2)	0·454 (2)	4·9 (6)

Table 2. *Bond distances* (Å) *and angles* (°) *in* 1-*phenyl*-1,2-*dihydro-2-quinolinone*

		Corrected for thermal vibration			
N(1)—C(2)	1·392 (2)	1·394	C(2)—N(1)—C(12)	117·2 (2)
N(1)—C(12)	1·449 (2)	1·451	C(12)—N(1)—C(10)	119·9 (2)
N(1)—C(10)	1·408 (2)	1·410	C(2)—N(1)—C(10)	122·9 (2)
C(2)—C(3)	1·444 (3)	1·446	N(1)—C(2)—O(11)	120·4 (2)
C(2)—O(11)	1·228 (2)	1·230	C(3)—C(2)—O(11)	123·7 (2)
C(3)—C(4)	1·333 (3)	1·335	N(1)—C(2)—C(3)	115·9 (2)
C(3)—H(3)	0·92 (3)		C(2)—C(3)—C(4)	122·4 (2)
C(4)—C(9)	1·428 (3)	1·430	C(3)—C(4)—C(9)	121·1 (2)
C(4)—H(4)	1·00 (3)		C(4)—C(9)—C(5)	122·4 (2)
C(5)—C(6)	1·365 (3)	1·367	C(4)—C(9)—C(10)	118·7 (2)
C(5)—C(9)	1·403 (3)	1·405	C(10)—C(9)—C(5)	118·9 (2)
C(5)—H(5)	0·97 (3)		C(9)—C(5)—C(6)	121·3 (2)
C(6)—C(7)	1·383 (3)	1·385	C(5)—C(6)—C(7)	119·3 (2)
C(6)—H(6)	0·96 (3)		C(6)—C(7)—C(8)	121·1 (1)
C(7)—C(8)	1·383 (3)	1·385	C(7)—C(8)—C(10)	119·8 (2)
C(7)—H(7)	1·00 (3)		C(8)—C(10)—N(1)	121·5 (2)
C(8)—C(10)	1·392 (3)	1·394	C(8)—C(10)—C(9)	119·5 (2)
C(8)—H(8)	0·95 (2)		C(9)—C(10)—N(1)	119·0 (2)
C(9)—C(10)	1·402 (3)	1·404	N(1)—C(12)—C(13)	120·1 (2)
C(12)—C(13)	1·379 (3)	1·382	N(1)—C(12)—C(17)	119·6 (2)
C(12)—C(17)	1·378 (3)	1·381	C(13)—C(12)—C(17)	120·3 (2)
C(13)—C(14)	1·383 (3)	1·385	C(12)—C(13)—C(14)	119·6 (2)
C(13)—H(13)	0·96 (3)		C(12)—C(17)—C(16)	119·8 (2)
C(17)—C(16)	1·377 (3)	1·380	C(13)—C(14)—C(15)	120·0 (2)
C(17)—H(17)	0·96 (3)		C(17)—C(16)—C(15)	120·3 (2)
C(14)—C(15)	1·381 (3)	1·383	C(14)—C(15)—C(16)	119·9 (2)
C(14)—H(14)	0·97 (3)			
C(16)—C(15)	1·374 (3)	1·377		
C(16)—H(16)	0·94 (3)			
C(15)—H(15)	0·99 (3)			

The atom positional parameters are given in Table 1, and a list of the most important interatomic bond distances and angles is given in Table 2.*

NMR spectra determined using $CDCl_3$ as solvent and a 360 MHz Bruker spectrospin spectrometer. 1-Phenyl-1,2-dihydro-2-quinolinone (Tschitschibabin & Jeletzky, 1924) gave following values at 298 K: δ 6·64 [*d*, 1H, H(8), *J* = 8·4 Hz]; 6·78 [*d*, 1H, H(3), *J* = 9·6 Hz]; 7·16–7·34 (*m*, 4H, aromatic H's); 7·49–7·61 (*m*, 4H, aromatic H's); 7·77 [*d*, 1H, H(4), *J* = 9·5 Hz]. The value for H(8) was not affected by lowering the temperature. The unsubstituted compound, 1,2-dihydro-2-quinolinone (Einhorn & Lauch, 1886), gave the following values: δ 6·72 [*d*, 1H, H(3), *J* = 9·5 Hz]; 7·19–7·26 (*m*, 1H, aromatic H); 7·47–7·57 (*m*, 3H, aromatic H's); 7·82 [*d*, 1H, H(4), *J* = 9·6 Hz]; 12·75 (broad singlet, 1H, NH).

Discussion. A drawing of the molecule is shown in Fig. 1.

The distances observed in the molecule are typical of aromatic structures with the exception that the C(3)—C(4) bond, 1·333 (3) Å, indicates a localization of a double bond at that position in conjugation with the carbonyl group at C(2). The phenyl group is attached at the N(1) atom, and it is its orientation relative to the plane of the quinolinone ring system which provides an explanation of the chemical shift of H(8) in the proton NMR. The quinoline ring system and the phenyl group are each substantially planar. The

* Lists of structure factors, anisotropic thermal parameters, least-squares planes, and additional intermolecular distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39450 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
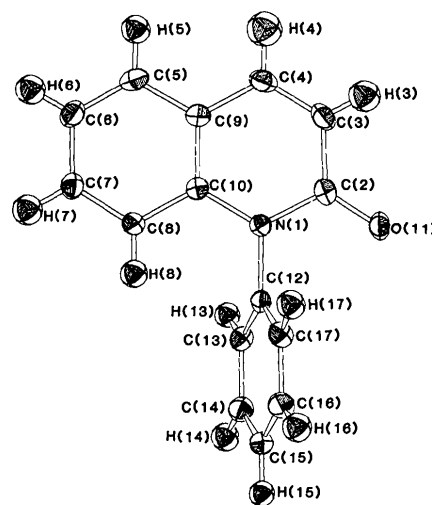

Fig. 1. *ORTEP* drawing (Johnson, 1965) of 1-phenyl-1,2-dihydro-2-quinolinone showing the atom labeling.

scatter from the mean plane of the non-hydrogen atoms is $\pm 0\cdot010$ (2) Å in the quinoline ring system, and $\pm 0\cdot006$ (2) Å for the phenyl ring. The deviations of the H atoms from the planes are not significant. The phenyl ring axis has a slight bend out of the plane of the quinoline ring, C(12) lying 0·06 and C(15) lying 0·16 Å below the plane of the quinoline ring. The angle between plane normals is 71·9 (1)°. The tilt of the phenyl group is such that C(13) [2·66 (2) Å] and H(13) [2·71 (3) Å] are near H(8). The C(12)–H(8) distance is 2·50 (2) Å, and if the dihedral angle had been 90°, the p_z orbital of C(12) would be directed nearly straight at H(8). As it is, the π-electron cloud of the phenyl group is oriented to interact strongly with H(8). This shielding of H(8) results in a shift in its proton NMR spectrum from the δ 7·47–7·57 region observed for the unsubstituted quinoline to δ 6·64 for the 1-phenyl derivative. This behavior substantiates the explanation made earlier (Wawzonek, 1982) for the unusual NMR spectra for 6-phenyl and 6-(3-pyridyl)-4,5,10,11-tetrahydro-6H-indeno[1,2-c]isoquinoline-5,11-dione. H(7) in both of these compounds is shielded by the aryl groups and appeared as a doublet at δ 5·46 and 5·52, respectively.

The thermal motion parameters of the non-hydrogen atoms were fit by a model which assumed the whole molecule behaved as a rigid body, and the components of the tensors **T, L** and **S** (Schomaker & Trueblood,

1968) were found which best reproduced the individual atomic vibrational ellipsoids. The inclusion of **S** gave a significantly better fit. The values of the tensor components are $T(11,22,33,12,13,23) = 0\cdot0349$ (14), 0·0415 (12), 0·0392 (13), −0·0038 (11), 0·0038 (12), 0·0039 (11) Å^2; L: 4·7 (7), 5·8 (1·3), 6·0 (9), −1·6 (6), 1·0 (6), 1·0 (8)°^{2}; $S(12,13,21,23,31,32,$ 11–22, 11–33): 0·02 (2) −0·00 (2) −0·00 (2), −0·00 (2), 0·08 (2), 0·12 (2), 0·09 (3), 0·04 (3) Å°. The only significant **S** components are $S31$, $S32$ and $(S11–S22)$.

References

BAENZIGER, N. C., FOSTER, B. A., HOWELLS, M., HOWELLS, R., VANDER VALK, P. & BURTON, D. J. (1977). *Acta Cryst.* B33, 2327–2329.
EINHORN, A. & LAUCH, R. (1886). *Chem. Ber.* 19, 53–55.
GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A27, 368–376.
International Tables for X-ray Crystallography (1974). Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* B24, 63–76.
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.
TSCHITSCHIBABIN, A. E. & JELETZKY, N. P. (1924). *Chem. Ber.* 57, 1158–1161.
WAWZONEK, S. (1982). *Org. Prep. Proc. Int.* 14, 163–168.

Absolute Configuration of the Brominated Product of Safracin A,* C28H35BrN4O6

BY IKUHIKO UEDA AND SHIGEAKI KAWANO

College of General Education, Kyushu University, Ropponmatsu, Chuo-ku, Fukuoka 810, Japan

AND YOSHIFUMI IKEDA, HIDEO MATSUKI AND TAKAYUKI OGAWA

Research Laboratories, Yoshitomi Pharmaceutical Ind. Ltd, 955 Koiwai, Yoshitomi-cho, Chikujo-gun, Fukuoka 871, Japan

Abstract. $M_r = 603\cdot53$, orthorhombic, $P2_12_12_1$, $a = 12\cdot25$ (1), $b = 17\cdot773$ (8), $c = 12\cdot681$ (8) Å, $V = 2760$ (3) Å^3, $Z = 4$, $D_m = 1\cdot43$, $D_x = 1\cdot45$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71069$ Å, $\mu = 1\cdot61$ mm^{-1}, $T = 298$ K, $F(000) = 1256$, $R = 0\cdot050$ for 2299 observed reflexions. The compound is an antibiotic product which has antitumor activity. The structure is similar to that of saframycin C. However, the pyruvamide side chain and
one of the p-quinone parts of the saframycins are substituted, respectively, by an alaninamide chain and a monophenol ring in the title compound. There is an unusually long C—C distance of 1·570 (9) Å.

Introduction. New antibiotic products which have antitumor activities were isolated from *Pseudomonas fluorescens* A$_2$-2 culture broth. The chemical structures of the main products were proposed to be (I) and (II) (see Fig. 1) from chemical and spectroscopic studies (Ikeda, Idemoto, Hirayama, Yamamoto, Iwao, Asao & Munakata, 1983; Ikeda, Matsuki, Ogawa & Munakata,