

Fig. 1. Perspective view of the molecule showing atomic nomenclature.

SHELX76 program. Table 1* lists the final atomic coordinates and equivalent isotropic thermal parameters of non-H atoms. The numbering scheme and molecular connectivity are given in the perspective view of the molecule (Fig. 1). The bond lengths, bond angles and selected torsion angles are listed in Table 2.

Related literature. In 2,5-bis(4-nitrophenyl)-3,4-diphenylthiophene (Meester, Maldar, Hosmane & Chu, 1986) the dihedral angle between the phenyl and thiophene rings is -72.0° and that between the nitrophenyl and thiophene rings is 26.6° . The dihedral angle between the phenyl and thiophene

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, additional torsion angles, equations to selected least-squares planes, hydrogen bonding geometry and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53451 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2-Hydroxy-2-phenyl-2-phthalidylacetophenone

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Abstract. $C_{22}H_{16}O_4$, $M_r = 344.4$, triclinic, $P\bar{1}$, $a = 10.684(2)$, $b = 9.455(1)$, $c = 9.492(1)$ Å, $\alpha = 104.18(2)$, $\beta = 90.02(2)$, $\gamma = 110.36(2)^\circ$, $V = 867.7(3)$ Å³, $Z = 2$, $D_x = 1.318$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.5184$ Å, $\mu = 6.99$ cm⁻¹, $F(000) = 360$, room

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rings is 70.23° in 2-amino-3-ethoxycarbonyl-4-phenylthiophene (Joseph, Kannan & Parthasarathi, 1990).

Biological activities of 2-aminothiophenes and thieno[2,3-*d*]pyrimidine have attracted the attention of medicinal chemists (Nakanishi, Imamura & Maruyama, 1970; Wellings, 1972; Manhas, Sharma & Amin, 1972). It is noteworthy that compounds possessing the carboxamido moiety show a variety of pharmacological effects (Kleeman & Engel, 1982).

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References

- COPE, A. C., HOFMANN, C. M., WYCKOFF, C. & HARDENBERGH, E. (1941). *J. Am. Chem. Soc.* **63**, 3452–3456.
 GEWALD, K. (1962). *Z. Chem.* **2**, 305–306.
 GEWALD, K. (1965). *Chem. Ber.* **98**, 3571–3577.
 GEWALD, K., SCHINKE, E. & BOTTCHE, H. (1966). *Chem. Ber.* **99**, 94–100.
 JOSEPH, P. S., KANNAN, S. & PARTHASARATHI, V. (1990). To be published.
 KLEEMAN, A. & ENGEL, J. (1982). Editors. *Pharmazentische Wirkstoffe*. 2nd edition. Stuttgart: George Thieme.
 MANHAS, M. S., SHARMA, S. D. & AMIN, S. G. (1972). *J. Med. Chem.* **15**, 106–107.
 MEESTER, P., MALDAR, N. N., HOSMANE, S. N. & CHU, S. S. C. (1986). *Acta Cryst.* **C42**, 363–364.
 NAKANISHI, M., IMAMURA, H. & MARUYAMA, Y. (1970). *Srzejmittel Forsch. (Drug Res.)* **20**, 998–1002.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRICK, G. M. (1986). *SHELXS86*. Program for crystal structure determination. Univ. of Göttingen, Germany.
 WELLINGS, I. (1972). US Patent Number 3644379. *Chem. Abstr.* **77**, 5440.

temperature, $R = 0.056$ for 2420 independent observed reflections. The two asymmetric centres at C(1) and C(2) respectively determine the *syn* relative configuration of the O(1) and O(2) atoms with a torsion angle O(1)—C(1)—C(2)—O(2) of $60.8(2)^\circ$.

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Table 1. Atomic fractional coordinates ($\times 10^4$) for non-H atoms and equivalent isotropic temperature factors

	x	y	z	B(\AA^2)
O(1)	5719 (2)	3382 (2)	4911 (1)	3.5 (1)
O(2)	5358 (1)	4317 (2)	7919 (1)	4.2 (1)
O(3)	3640 (2)	660 (2)	5559 (2)	6.0 (1)
O(4)	6495 (2)	5375 (2)	3888 (2)	6.9 (2)
C(1)	6194 (2)	2798 (3)	5996 (2)	5.3 (2)
C(2)	5295 (2)	2785 (2)	7269 (2)	4.5 (1)
C(3)	3838 (2)	1654 (3)	6693 (2)	4.5 (2)
C(4)	2686 (2)	1727 (2)	7569 (2)	4.4 (2)
C(5)	2844 (2)	2564 (3)	9016 (3)	4.9 (2)
C(6)	1718 (3)	2517 (3)	9769 (3)	6.1 (2)
C(7)	453 (3)	1655 (4)	9085 (3)	7.5 (2)
C(8)	291 (3)	837 (4)	7647 (4)	8.3 (2)
C(9)	1399 (3)	856 (3)	6881 (3)	7.1 (2)
C(10)	5812 (2)	2148 (2)	8378 (2)	5.2 (1)
C(11)	6232 (2)	3039 (3)	9809 (2)	5.0 (2)
C(12)	6753 (2)	2459 (3)	10770 (3)	5.5 (2)
C(13)	6892 (3)	1020 (3)	10332 (3)	6.0 (2)
C(14)	6472 (3)	143 (3)	8924 (3)	6.0 (2)
C(15)	5921 (2)	679 (3)	7953 (3)	5.4 (2)
C(16)	6673 (3)	4721 (3)	4756 (3)	5.2 (2)
C(17)	7861 (2)	5064 (3)	5722 (3)	5.0 (2)
C(18)	9091 (3)	6297 (3)	5922 (3)	5.9 (2)
C(19)	10063 (3)	6310 (3)	6854 (3)	6.9 (2)
C(20)	9855 (3)	5146 (4)	7568 (3)	7.0 (2)
C(21)	8623 (3)	3906 (3)	7356 (3)	6.3 (2)
C(22)	7617 (2)	3907 (3)	6437 (2)	5.2 (2)

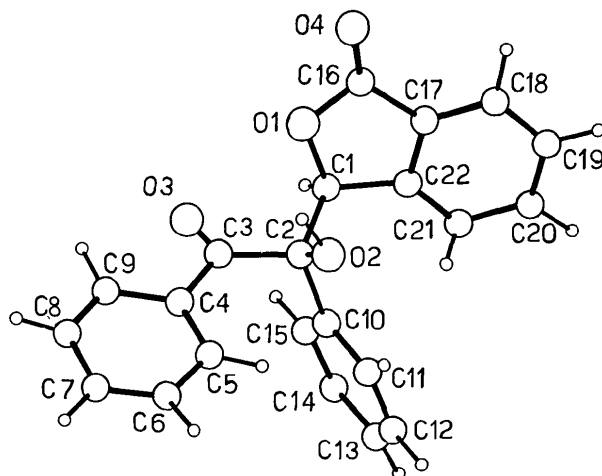


Fig. 1. A perspective view of the molecule showing the atom-numbering scheme.

The O(2) atom is *trans* to O(3) with a torsion angle O(3)—C(3)—C(2)—O(2) of $141.7(2)^\circ$. The two phenyl rings make a dihedral angle of $74.66(8)^\circ$ to each other and dihedral angles of $61.58(6)^\circ$ and $72.45(7)^\circ$ with the phthalide-system plane. Intra- and intermolecular hydrogen bonds are present.

Experimental. Colourless crystal, *ca* $0.3 \times 0.2 \times 0.2$ mm, was obtained by slow evaporation from methanol solution. Intensity data were collected on a Philips PW1100 automatic diffractometer using graphite-monochromated Cu $K\alpha$ radiation and $\theta/2\theta$ scan technique. Cell parameters were determined by least-squares refinement of 18 reflections with $18 \leq \theta$

Table 2. Bond lengths (\AA) and angles ($^\circ$)

O(1)—C(1)	1.448 (3)	C(8)—C(9)	1.386 (4)
O(1)—C(16)	1.363 (3)	C(10)—C(11)	1.390 (3)
O(2)—C(2)	1.406 (3)	C(10)—C(15)	1.395 (3)
O(3)—C(3)	1.206 (3)	C(11)—C(12)	1.378 (4)
O(4)—C(16)	1.195 (4)	C(12)—C(13)	1.383 (4)
C(1)—C(2)	1.544 (3)	C(13)—C(14)	1.368 (3)
C(1)—C(22)	1.504 (3)	C(14)—C(15)	1.375 (4)
C(2)—C(3)	1.556 (3)	C(16)—C(17)	1.458 (4)
C(2)—C(10)	1.522 (4)	C(17)—C(18)	1.396 (3)
C(3)—C(4)	1.497 (3)	C(17)—C(22)	1.375 (4)
C(4)—C(5)	1.384 (3)	C(18)—C(19)	1.359 (4)
C(4)—C(9)	1.395 (3)	C(19)—C(20)	1.384 (5)
C(5)—C(6)	1.392 (4)	C(20)—C(21)	1.400 (3)
C(6)—C(7)	1.373 (4)	C(21)—C(22)	1.384 (4)
C(7)—C(8)	1.372 (4)		
C(1)—O(1)—C(16)	110.8 (2)	C(2)—C(10)—C(15)	120.2 (2)
O(1)—C(1)—C(22)	103.9 (2)	C(2)—C(10)—C(11)	120.9 (2)
O(1)—C(1)—C(2)	109.5 (2)	C(11)—C(10)—C(15)	118.9 (2)
C(2)—C(1)—C(22)	113.3 (2)	C(10)—C(11)—C(12)	119.6 (2)
O(2)—C(2)—C(1)	109.4 (2)	C(11)—C(12)—C(13)	121.3 (2)
C(1)—C(2)—C(10)	108.1 (2)	C(12)—C(13)—C(14)	119.0 (3)
C(1)—C(2)—C(3)	109.4 (2)	C(13)—C(14)—C(15)	120.9 (3)
O(2)—C(2)—C(10)	110.0 (2)	C(10)—C(15)—C(14)	120.4 (2)
O(2)—C(2)—C(3)	112.2 (2)	O(1)—C(16)—O(4)	121.6 (3)
C(3)—C(2)—C(10)	107.6 (2)	O(4)—C(16)—C(17)	130.1 (3)
O(3)—C(3)—C(2)	119.6 (2)	O(1)—C(16)—C(17)	108.3 (2)
C(2)—C(3)—C(4)	120.8 (2)	C(16)—C(17)—C(22)	108.6 (2)
O(3)—C(3)—C(4)	119.5 (2)	C(16)—C(17)—C(18)	129.0 (2)
C(3)—C(4)—C(9)	117.1 (2)	C(18)—C(17)—C(22)	122.4 (3)
C(3)—C(4)—C(5)	123.4 (2)	C(17)—C(18)—C(19)	117.1 (3)
C(5)—C(4)—C(9)	119.5 (2)	C(18)—C(19)—C(20)	121.8 (3)
C(4)—C(5)—C(6)	119.6 (2)	C(19)—C(20)—C(21)	120.8 (3)
C(5)—C(6)—C(7)	120.6 (3)	C(20)—C(21)—C(22)	117.7 (3)
C(6)—C(7)—C(8)	120.0 (3)	C(17)—C(22)—C(21)	120.1 (3)
C(7)—C(8)—C(9)	120.4 (3)	C(1)—C(22)—C(21)	131.6 (2)
C(4)—C(9)—C(8)	119.9 (3)	C(1)—C(22)—C(17)	108.3 (2)

Table 3. Short non-bonded contacts (\AA , $^\circ$)

Donor (D)	Acceptor (A)	D...A	H...A	D—H...A	Symmetry operations
O(2)	O(4)	2.755 (3)	1.83 (3)	166 (2)	$-x + 1, -y + 1, -z + 1$
O(2)	O(1)	2.843 (2)	2.54 (3)	99 (1)	x, y, z
C(5)	O(2)	2.965 (3)	2.48 (2)	111 (2)	x, y, z
C(1)	O(3)	3.307 (3)	2.44 (2)	166 (2)	$-x + 1, -y, -z + 1$
C(15)	O(3)	3.359 (3)	2.58 (2)	140 (2)	$-x + 1, -y, -z + 1$
C(1)	O(3)	2.730 (3)	2.46 (3)	98 (2)	x, y, z

$\leq 35^\circ$. A total of 2577 unique reflections, with $-6 \leq h \leq 6$, $-13 \leq k \leq 13$, $0 \leq l \leq 9$, were collected up to $\theta = 60^\circ$. 2420 reflections with $I \geq 2\sigma(I)$ were considered observed and retained for structure determination and refinement. Three standard reflections, monitored every 90 min, showed no loss of intensity.

The structure was solved by direct methods using MULTAN80 (Main *et al.*, 1980) and subsequent difference Fourier methods. The refinement on F was carried out by two full-matrix blocks with anisotropic temperature factors for all non-H atoms using SHELX76 (Sheldrick, 1976). All H atoms were located from a difference map and refined isotropically. A total of 299 parameters were optimized. At final convergence, $R = 0.056$, $wR = 0.063$, $w^{-1} = \sigma^2(F) + 0.000092F^2$, $(\Delta/\sigma)_{\max} = 0.53$, $(\Delta/\sigma)_{\text{av}} = 0.12$, $|\Delta\rho|_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$. Atomic scattering factors were taken from *International Tables for X-ray Cryst-*

tallography (1974, Vol. IV). All geometrical calculations were performed using *PARST* (Nardelli, 1983). Fig. 1 shows the atom-labelling scheme and molecular conformation. Atomic coordinates are listed in Table 1,* bond distances and angles in Table 2. Hydrogen bonds are detailed in Table 3.

Related literature. The synthesis of the title compound forms part of a series of studies on stereoselective synthesis of α,β -dihydroxy ketones (Clerici & Porta, 1989). The ketyl radical generated by titanium(III) reduction of benzil contributes stereoselectively to the aldehydic C atom of 2-carboxy-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53462 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Nucleoside Analogue: 3'-Azido-5-chloro-2',3'-dideoxyuridine*

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Abstract. 1-(3-Azido-2,3-dideoxy- β -D-erythro-pentofuranosyl)-5-chlorouracil, $C_9H_{10}ClN_5O_4$, $M_r = 287.66$, monoclinic, $P2_1$, $a = 5.780$ (3), $b = 11.730$ (6), $c = 17.670$ (9) Å, $\beta = 93.87$ (4)°, $V = 1195$ (1) Å³, $Z = 4$, $D_m = 1.60$, $D_x = 1.598$ Mg m⁻³, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.335$ mm⁻¹, $F(000) = 592$, $T = 290$ K. Final $R = 0.021$ for 1892 unique observed reflections. The asymmetric unit contains two molecules (*A* and *B*). For molecule *A*: the *N*-glycosidic torsion angle χ has a value of -128.2 (2)° in the *anti* range; the sugar pucker is 2T_3 with $P = 173$ (1) and $\psi_m = 33$ (1)° and the C4'—C5' conformation is $+sc$ with $\gamma = 50.4$ (3)°. For molecule *B*: the *N*-glycosidic torsion

benzaldehyde, affording, after lactonization, the present phthalidyl derivative. Structural aspects of the phthalide system have been reported by Whalley, Ferguson & Roberts (1980) and by Roszak & Skrzat (1989).

References

- CLERICI, A. & PORTA, O. (1989). *J. Org. Chem.* **54**, 3872–3878.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, 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.
 NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
 ROSZAK, A. & SKRZAT, Z. (1989). *Acta Cryst.* **C45**, 1773–1776.
 SHELDRIK, G. M. (1976). Program for crystal structure determination. Univ. of Cambridge, England.
 WHALLEY, W. B., FERGUSON, G. & ROBERTS, P. J. (1980). *Acta Cryst.* **B36**, 2465–2467.

angle χ has a value of -168.9 (2)° in the *anti* range; the sugar pucker is ${}^3T^4$ with $P = 208$ (1) and $\psi_m = 36$ (1)° and the C4'—C5' conformation is *ap* with $\gamma = 170.8$ (2)°. The conformational parameters are in accordance with the IUPAC–IUB Joint Commission on Biochemical Nomenclature [*Pure Appl. Chem.* (1983), **55**, 1273–1280] guidelines. Base-pair formation occurs in the crystal structure.

Experimental. Colourless prismatic crystals obtained at room temperature from an aqueous solution, dimensions $\sim 0.55 \times 0.3 \times 0.2$ mm. D_m by flotation. STADI4 computer-controlled four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique ($2\theta_{\max} = 50^\circ$, $-7 \leq h \leq 0$, $-14 \leq k \leq 14$, $-21 \leq l \leq 21$). Cell dimensions by least-squares refinement of the setting angles of 24 reflections with $20 < 2\theta < 29^\circ$, space group $P2_1$ from

* Structural Studies of Modified Nucleosides. Part V. Part IV: Everaert, Peeters, Blaton, De Ranter, Van Aerschot & Herdewijn (1990).

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