Table 2. *Torsion angles of interest* (°)

C(3)–O(2)–C(1)–C(18)	97·4 (3)	C(12)–C(11)–C(10)–O(10)	−8·6 (3)
O(2)–C(1)–C(18)–C(19)	−171·4 (3)	C(12)–C(11)–C(10)–C(9)	173·6 (3)
C(1)–C(18)–C(19)–C(15)	0·3 (3)	C(11)–C(10)–C(9)–C(8)	−158·8 (3)
C(18)–C(19)–C(15)–C(14)	166·1 (3)	C(10)–C(9)–C(8)–C(7)	78·4 (3)
C(19)–C(15)–C(14)–C(13)	−88·4 (3)	C(9)–C(8)–C(7)–C(20)	−28·6 (4)
C(15)–C(14)–C(13)–C(12)	63·7 (3)	C(8)–C(7)–C(20)–C(3)	−176·6 (3)
C(14)–C(13)–C(12)–C(11)	−116·7 (3)	C(7)–C(20)–C(3)–O(2)	179·9 (2)
C(14)–C(13)–C(12)–O(12)	61·3 (3)	C(20)–C(3)–O(2)–C(1)	−14·2 (3)
C(13)–C(12)–C(11)–C(10)	0·1 (3)		

the plane. The angle between these planes is 95·3 (3)°. The least-squares plane through C(12), C(11), C(10) and O(10) also shows deviation from planarity (χ^2 = 244); C(9) and C(13) are 0·105 (2) and 0·135 (3) Å from this plane whereas O(12) is essentially on the plane [deviation 0·005 (3) Å].

The torsion angles around the macrocylic ring are given in Table 2. Some of the unusual geometry observed in the present study may be related to the electronic properties of the C(9)⋯C(13) segment. However, further analysis will only become possible when more structures in this class of antibiotics are

known. The structure is stabilized by van der Waals forces.

References

BRUFANI, M., FEDELI, W., GIACOMELLO, G. & VACIAGO, A. (1964). *Experientia*, **20**, 339–342.
CAMERMAN, N. & TROTTER, J. (1964). *Acta Cryst.* **17**, 384–391.
GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
GRANT, D. F. & GABE, E. J. (1978). *J. Appl. Cryst.* **11**, 114–120.
HARRIS, D. R., MCGEACHIN, S. G. & MILLS, H. H. (1965). *Tetrahedron Lett.* pp. 679–685.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
LARSON, A. C. & GABE, E. J. (1978). *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, p. 81. Delft Univ. Press.
LE PAGE, Y., GABE, E. J. & CALVERT, L. D. (1979). *J. Appl. Cryst.* **12**, 25–26.
STOECKLI-EVANS, H. (1979). *Acta Cryst.* **B35**, 2798–2800.

Structure of 4-(p-Chlorobenzoyl)pyridine,* C₁₂H₈ClNO

BY ASHFAQUZZAMAN SYED,† LINDA I. FOSS AND EDWIN D. STEVENS

Department of Chemistry, University of New Orleans, New Orleans, LA 70148, USA

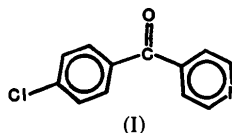**Abstract.** $M_r = 217·66$, monoclinic, $P2_1$, $a = 21·585$ (4), $b = 5·762$ (1), $c = 3·843$ (1) Å, $\beta = 90·27$ (2)°, $V = 477·99$ Å³, $Z = 2$, $D_x = 1·512$ g cm⁻³, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3·6$ cm⁻¹, $F(000) = 224$, $T = 110$ K. Final $R = 0·0472$ for 1853 observed reflections. The molecular structure shows a twist in the essentially planar aromatic rings giving a dihedral angle of 52·2°; the repulsions between the non-bonded atoms are thus minimized. The carbonyl portion of the molecule exhibits a high degree of planarity and an increase in the endocyclic angle opposite the chlorine substituent is observed.

Introduction. The title compound (I) is one of a series of substituted benzophenones being studied in our laboratory (Syed, Stevens & Klein, 1984; Syed & Stevens, 1984). Structure determination was undertaken as a continuation of a program to determine the geometry of compounds containing carbonyl groups.

---

* p-Chlorophenyl 4-pyridyl ketone.
† On leave from Tata Iron & Steel Company, Jamshedpur, India.

The effect of an electronegative group substituted for an H atom on a phenyl ring has been observed.

(I)

Experimental. Sample recrystallized from equal volumes acetone and tetrahydrofuran, approximate dimensions 0·28 × 0·13 × 0·40 mm, Enraf–Nonius CAD-4 diffractometer, graphite-crystal monochromator, Mo $K\alpha$ radiation, temperature maintained at constant 110 K, lattice parameters from 25 reflections with $20 < 2\theta < 44°$, systematic absences: 0k0, $k = 2n + 1$, absorption as a function of ψ observed to be insignificant and no correction applied, reflections measured within ranges $2 \leq 2\theta < 70°$, $−34 \leq h \leq 34$, $0 \leq k \leq 9$, $0 \leq l \leq 6$, $\omega:2\theta$ scans; intensities of three standard reflections decreased by 1·2% and were used to correct data; 2315 total reflections measured, 1853

reflections considered observed $[F > 3\sigma(F)]$, corrected for Lorentz and polarization effects. Cl atom located from a three-dimensional Patterson map, peaks corresponding to 14 non-H atoms from a three-dimensional Fourier map computed using phases from Cl-atom position, and seven H atoms from subsequent difference Fourier syntheses. H thermal parameters correspond to their respective C atoms. Structure refined by full-matrix least squares, $\sum w(|F_o| - |F_c|)^2$ minimized. One H-atom position calculated on the basis of standard geometry, H positions and thermal parameters fixed during refinement, other atoms refined anisotropically. Final $R = 0.0472$, $R_w = 0.0664$ for 137 variables, $w = 1/\sigma(F)^2$, where $\sigma(F^2)^2 = \sigma_{CS}^2 + (0.03F^2)^2$, $S = 2.48$, $(\Delta/\sigma)_{max} = 0.01$, max. peak in final difference Fourier map 0.8 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974), all computer programs from *SDP* system (Frenz, 1982).

Discussion. Final atomic and corresponding isotropic thermal parameters for the non-H atoms are given in Table 1 and the bond lengths and angles in Table 2.* The corresponding e.s.d.'s are shown in parentheses. Fig. 1 shows the molecular structure and atomic-numbering scheme in an *ORTEP*II (Johnson, 1976) plot.

The two rings are essentially planar with atom C(7) deviating as much as -0.022 (5) and -0.117 (5) Å with respect to the chloro-substituted and pyridine rings respectively. However, a least-squares plane through C(4), C(7), O and C(8) reveals a high degree of planarity $(\chi^2 = 2)$ for the central part of the structure. Thus the molecule adopts a twisted conformation in order to minimize the repulsions between the nonbonded atoms: torsion angles C(5)—C(4)—C(7)—C(8) and C(9)—C(8)—C(7)—C(4) are -178.5 and $53.6°$ respectively and the dihedral angle between the two rings is $52.2°$. The molecular dimensions are usual for such structures and compare well with related structures (Syed, Stevens & Klein, 1984; Greenhough & Trotter, 1980; Acton, Roth, Katz, Frank, Maier & Paul, 1972).

The C=O bond distance is in accord with various other carbonyl bond lengths (Brown & Colclough, 1983; Syed, Stevens & Klein, 1984). The C—Cl bond length, 1.745 (2) Å, agrees well with that obtained for related structures (Syed & Pant, 1979; Syed, Umrigar,

---

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and C—H distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39157 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and equivalent isotropic thermal parameters*

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as:
$$\tfrac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (ab\cos\gamma)\beta_{12} + (ac\cos\beta)\beta_{13} + (bc\cos\alpha)\beta_{23}].$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Cl	0.45174 (3)	0.4996	0.0942 (2)	1.60 (1)
O	0.2421 (1)	1.2756 (4)	0.6243 (6)	1.52 (4)
N	0.0425 (1)	0.9103 (5)	0.2991 (7)	1.47 (4)
C(1)	0.3883 (1)	0.6713 (5)	0.2064 (7)	1.12 (4)
C(2)	0.3301 (2)	0.5948 (5)	0.1165 (8)	1.19 (4)
C(3)	0.2791 (1)	0.7304 (5)	0.2026 (7)	1.05 (4)
C(4)	0.2869 (1)	0.9403 (5)	0.3802 (7)	0.98 (4)
C(5)	0.3473 (1)	1.0143 (6)	0.4634 (7)	1.16 (4)
C(6)	0.3978 (1)	0.8818 (5)	0.3740 (8)	1.18 (4)
C(7)	0.2337 (1)	1.0884 (5)	0.4812 (7)	1.01 (4)
C(8)	0.1683 (1)	1.0143 (6)	0.4063 (6)	0.95 (4)
C(9)	0.1439 (1)	0.8008 (5)	0.5085 (7)	1.13 (4)
C(10)	0.0818 (2)	0.7587 (5)	0.4536 (8)	1.37 (5)
C(11)	0.0674 (2)	1.1126 (6)	0.1949 (8)	1.41 (5)
C(12)	0.1288 (1)	1.1728 (5)	0.2471 (8)	1.13 (4)

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

Cl—C(1)	1.745 (2)	C(4)—C(5)	1.407 (3)
O—C(7)	1.224 (2)	C(4)—C(7)	1.485 (3)
N—C(10)	1.353 (3)	C(7)—C(8)	1.501 (3)
N—C(11)	1.346 (3)	C(5)—C(6)	1.376 (3)
C(1)—C(2)	1.374 (3)	C(8)—C(9)	1.395 (3)
C(1)—C(6)	1.388 (3)	C(8)—C(12)	1.389 (3)
C(2)—C(3)	1.392 (3)	C(9)—C(10)	1.378 (3)
C(3)—C(4)	1.399 (2)	C(11)—C(12)	1.385 (3)

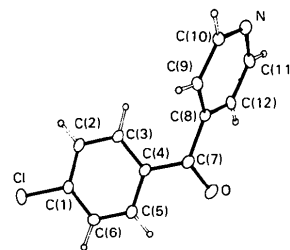|			
C(10)—N—C(11)	116.0 (2)	O—C(7)—C(4)	120.7 (2)
Cl—C(1)—C(2)	118.3 (2)	O—C(7)—C(8)	118.3 (2)
Cl—C(1)—C(6)	119.7 (2)	C(4)—C(7)—C(8)	121.0 (2)
C(2)—C(1)—C(6)	122.0 (2)	C(7)—C(8)—C(9)	123.6 (2)
C(1)—C(2)—C(3)	118.9 (2)	C(7)—C(8)—C(12)	118.1 (2)
C(2)—C(3)—C(4)	120.5 (2)	C(9)—C(8)—C(12)	118.2 (2)
C(3)—C(4)—C(5)	118.9 (2)	C(8)—C(9)—C(10)	118.7 (2)
C(3)—C(4)—C(7)	122.1 (2)	N—C(10)—C(9)	124.2 (2)
C(5)—C(4)—C(7)	119.0 (2)	N—C(11)—C(12)	123.9 (2)
C(4)—C(5)—C(6)	120.6 (2)	C(8)—C(12)—C(11)	118.9 (2)
C(1)—C(6)—C(5)	119.0 (2)		

Fig. 1. Plot of the 4-(*p*-chlorobenzoyl)pyridine molecule showing the atomic-labeling scheme. Thermal ellipsoids are plotted at the 50% probability level. H atoms have been plotted with arbitrary radii. Selected torsion angles: C(5)—C(4)—C(7)—C(8) = −178.5, C(3)—C(4)—C(7)—C(8) = 2.1, C(9)—C(8)—C(7)—C(4) = 53.6, C(12)—C(8)—C(7)—C(4) = −130.8, C(9)—C(8)—C(7)—O = −127.2, C(12)—C(8)—C(7)—O = 48.4, C(3)—C(4)—C(7)—O = −177.1, C(5)—C(4)—C(7)—O = 2.4° (e.s.d.'s are in the range 0.4–0.5°).
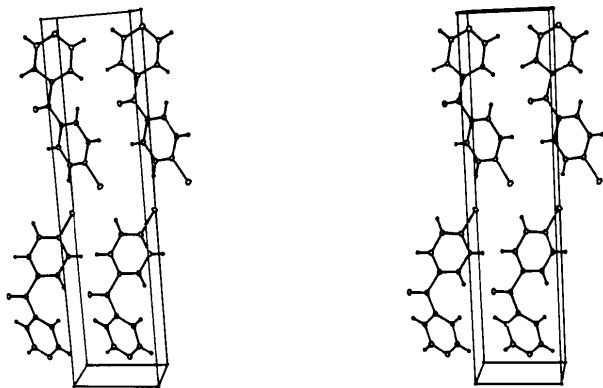
Fig. 2. Stereoview of the molecular packing in the unit cell of the 4-(p-chlorobenzoyl)pyridine molecule.

Griffin, Stevens & Majeste, 1984). The shortening of the bond C(1)—C(2) and an increase in the endocyclic angle C(2)—C(1)—C(6) [122·0 (2)°] occur due to the substitution of an electron-withdrawing group in place of an H atom (Domenicano, Vaciago & Coulson, 1975a,b; Syed & Pant, 1979).

A stereoview of the contents of the unit cell is shown in Fig. 2 (Johnson, 1976). The molecules are packed in parallel stacks at normal van der Waals distances. No unusually short intermolecular contacts were noted.

References

ACTON, N., ROTH, R. J., KATZ, T. J., FRANK, J. K., MAIER, C. A. & PAUL, I. C. (1972). J. Am. Chem. Soc. 94, 5446–5456.

BROWN, C. J. & COLCLOUGH, M. L. (1983). Acta Cryst. C39, 300–302.

DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975a). Acta Cryst. B31, 221–234.

DOMENICANO, A., VACIAGO, A. & COULSON, C. A. (1975b). Acta Cryst. B31, 1630–1641.

FRENZ, B. A. (1982). The Enraf–Nonius CAD-4 Structure Determination Package, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft Univ. Press.

GREENHOUGH, T. J. & TROTTER, J. (1980). Acta Cryst. B36, 478–480.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

SYED, A. & PANT, A. K. (1979). Acta Cryst. B35, 1394–1399.

SYED, A. & STEVENS, E. D. (1984). Acta Cryst. Submitted for publication.

SYED, A., STEVENS, E. D. & KLEIN, C. L. (1984). Acta Cryst. Submitted for publication.

SYED, A., UMRIGAR, P., GRIFFIN, G. W., STEVENS, E. D. & MAJESTE, R. J. (1984). Acta Cryst. Submitted for publication.

β-Modifikation von Naphtho[2,3-c][1,2,5]thiadiazol-4,9-dion, C₁₀H₄N₂O₂S*

VON ALFRED GIEREN, THOMAS HÜBNER UND VIKTOR LAMM

Max-Planck-Institut für Biochemie, Abteilung Strukturforschung I, 8033 Martinsried bei München, Bundesrepublik Deutschland

Abstract. $M_r = 216·2$, monoclinic, $I2/a$, $a = 8·416$ (4), $b = 10·952$ (4), $c = 9·504$ (4) Å, $\beta = 86·57$ (4)°, $U = 874·4$ Å³, $Z = 4$, $D_m = 1·64$ (1), $D_x = 1·642$ Mg m⁻³, Cu $K\alpha$, $\lambda = 1·54178$ Å, $\mu = 2·97$ mm⁻¹, $F(000) = 440$, 295 K, final $R = 0·098$ for 548 unique reflections. The S—N— [1·619 (4) Å] and the C—N— [1·337 (6) Å] bond lengths indicate a strong π interaction between the sulfur diimide group and the naphthoquinone system inducing a quasi-aromatic character of the 1,2,5-thiadiazole ring. The molecular structure is not significantly different from that in the α modification and the 2:1 complex with tetrathiafulvalene (TTF). In the crystal structure the molecules form layers with coplanar molecular planes and also columnar stacks with an interplanar distance of 3·33 Å.

* Synthesen, Konstitutionsaufklärung, Röntgenstrukturanalyse und elektrische Eigenschaften von Mono-, Bis- und Polychalkogendiimiden 4. 3. Mitteilung: Gieren, Lamm, Hübner, Rabben, Neidlein & Droste (1984).

0108-2701/84/050836-04$01.50