

biphenyl groupings differ:  $52.1(2)^\circ$  between rings *a* and *b*, and  $46.8(2)^\circ$  between rings *c* and *d* [*cf.*  $\sim 30^\circ$  proposed for the molecule in solution by Olsson *et al.* (1981) and  $89.9^\circ$  observed for the tetramethoxyl-tetramethyl analog by Kaneda *et al.* (1985)]. No doubt this angle is much larger in the latter compound to accommodate the bulky methoxyl groups.

The right-hand view of the molecule in Fig. 1 shows how the protons on C(2*a*)–C(2*d*) are magnetically shielded by an aromatic ring across the large ring. Using the equations of Johnson & Bovey (1958), we have calculated the upfield shift expected for the conformation observed in the crystal to be just what is observed in solution by NMR, so the conformation is essentially the same in both media, and probably represents the lowest energy minimum for the molecule.

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## Structure of 3'-Acetamido-2,2-dichloro-4',5'-dimethylacetophenone

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**Abstract.**  $C_{12}H_{13}Cl_2NO_2$ ,  $M_r = 274.15$ , monoclinic,  $P2_1/n$ ,  $a = 17.645(2)$ ,  $b = 14.645(1)$ ,  $c = 10.5955(7)$  Å,  $\beta = 107.191(7)^\circ$ ,  $V = 2615.7(4)$  Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.41$ ,  $D_x = 1.39$  g cm<sup>-3</sup>,  $\lambda(Cu K\alpha) = 1.5418$  Å,  $\mu = 44.75$  cm<sup>-1</sup>,  $F(000) = 1136$ ,  $T = 295$  K, final  $R = 0.067$  for 3306 observed reflections. The acetyl groups in both the crystallographically independent molecules are in the *endo* conformation, whereas there is a conformational difference about the C(phenyl)–C(keto) bonds of the dichloroacetyl groups of the two molecules. The  $\pi$ -electron cloud in the keto bonds of the dichloroacetyl groups are more localized than those in related compounds. Intermolecular N–H...O hydrogen bonds link the molecules into infinite chains.

**Introduction.** The study of the structure–activity relationship of the widely used antibiotic

chloramphenicol established the importance of the dichloroacetyl group (Dennis & Berberian, 1954). Acetanilide and its various derivatives possess a variety of antifungal, antibacterial, analgesic and anaesthetic activities (Burger, 1960; Banerjee, 1983).

The structure of the title compound was analysed as part of our studies on dichloroacetophenones and acetanilides (Chattopadhyay, Banerjee, Mazumdar & Podder, 1985; Haisa, Kashino, Ueno, Shinozaki & Matsuzaki, 1980).

**Experimental.** Pale-yellow plate-shaped crystals (from ethanol); density by flotation (benzene – bromoform); crystal size:  $0.35 \times 0.27 \times 0.25$  mm; symmetry from oscillation and Weissenberg photographs,  $P2_1/n$  (systematic absences:  $0k0$ ,  $k$  odd;  $h0l$ ,  $h+l$  odd); Rigaku AFC-5 four-circle diffractometer equipped with rotating anode; Ni-filtered Cu  $K\alpha$  radiation; cell

parameters were refined from the setting angles of 20 reflections ( $6.5 \leq \theta \leq 40^\circ$ ), 4163 unique reflections ( $-20 \leq h \leq 20$ ,  $0 \leq k \leq 16$ ,  $0 \leq l \leq 11$ ,  $4 \leq 2\theta \leq 125^\circ$ ), 3306 observed [ $I \geq 2.5\sigma(I)$ ], intensity corrected for Lp, absorption ignored;  $\omega/2\theta$  scan mode used, scan speed  $4^\circ \text{ min}^{-1}$  in  $\omega$ , scan range ( $1.2 + 0.15 \tan \theta$ ) $^\circ$ , three orientation control reflections ( $\bar{2}21$ ,  $\bar{1}12$ , 600) monitored every 57 reflections, no intensity deterioration during data collection; no correction for intensity variation (<2%); background measured for 4 s on either side of peak; direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); full-matrix refinement for non-H atoms based on  $F$  with anisotropic thermal parameters (*SHELX76*; Sheldrick, 1976); 19 H atoms out of 26 located from difference synthesis, refined initially with isotropic thermal parameters and fixed in final cycles of refinement; seven H atoms, generated with fixed geometry, not refined at all;  $R = 0.067$ ,  $wR = 0.081$ ,  $S = 2.15$ ,  $w = 1/[\sigma^2 |F_o| + 0.005006 \times |F_o|^2]$ ,  $\Delta/\sigma < 0.05$ , residual  $\Delta\rho$  excursions  $-0.23$  to  $0.25 \text{ e } \text{Å}^{-3}$  in final difference synthesis, scattering factors from *International Tables for X-ray Crystallography* (1974).

**Discussion.** The molecular structure drawn by *ORTEP* (Johnson, 1965) is shown in Fig. 1 along with the numbering of atoms. The two molecules in the asymmetric unit are identified as *A* and *B* and, accordingly, the individual atoms in a molecule have been identified with the subscripts *A* and *B* whenever necessary. The fractional atomic coordinates together with the isotropic thermal parameters are listed in Table 1.\* Bond distances and bond angles are given in Table 2. The benzene rings in these molecules are nearly planar, with the atom C(2) in each molecule deviating most from the plane of the ring [ $0.006(3) \text{ Å}$  in *A* and  $0.009(3) \text{ Å}$  in *B*]. The dihedral angle between the plane of the benzene ring and the plane defined by C(1), C(7) and O(1) is  $12.6(2)^\circ$  in molecule *A* and  $6.4(3)^\circ$  in molecule *B*. The carbonyl O atom of the dichloroacetyl group, O(1), is *trans* to C(2) in molecule *A* and *cis* in *B*. The amide group in either molecule is planar. The acetyl groups in both the molecules are in an *endo* conformation with the amide O atoms, O(2), *cis* to C(3) as indicated by the C(3)–N–C(11)–O(2) torsion angles,  $2.3(5)^\circ$  and  $-1.7(6)^\circ$  in molecules *A* and *B*, respectively. The dihedral angle between the benzene ring and the amide-group plane defined by C(3), N,

C(11) and O(2) is  $75.0(1)^\circ$  in molecule *A* and  $44.8(1)^\circ$  in molecule *B*.

Christoph & Fleischer (1973) correlated the extent of  $\pi$  overlap between the benzene ring and the amide group with the dihedral angle between their planes. According to the relation given by them, the extent of  $\pi$  overlap for the present molecules *A* and *B* is 6.7% and 50% of maximum. However, the  $\pi$  character of the C(phenyl)–N bond, as indicated by its length, remains the same in both the molecules. This length is similar to the corresponding bond lengths in both the orthorhombic [ $1.422(5) \text{ Å}$ ] (Haisa, Kashino & Maeda, 1974) and the monoclinic [ $1.425(3) \text{ Å}$ ] (Haisa, Kas-

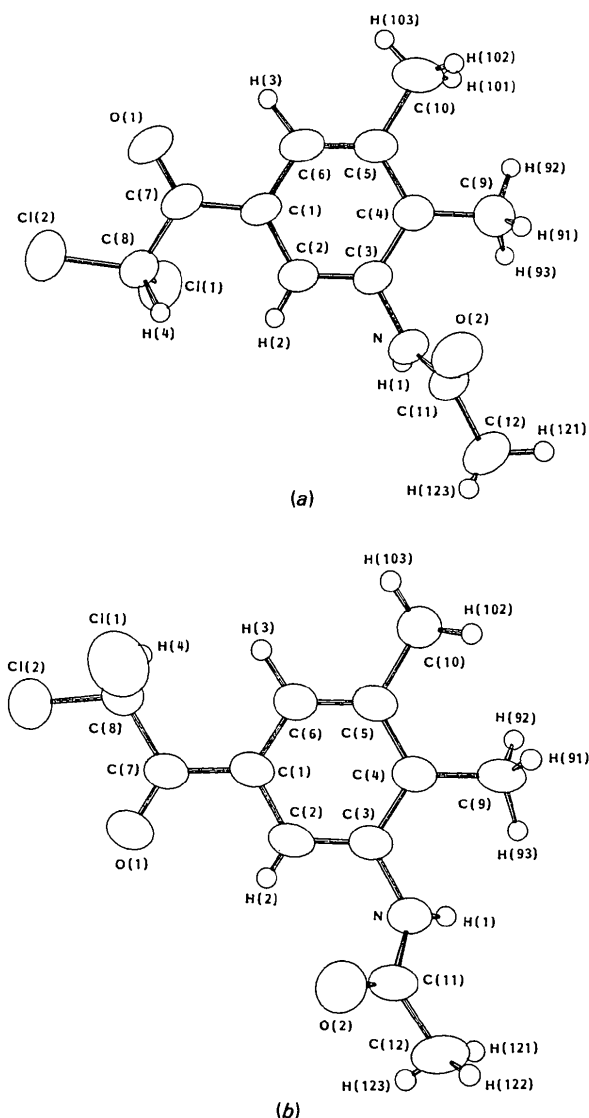


Fig. 1. Views of the molecules *A* (upper) and *B* (lower) with numbering of atoms. Ellipsoids of 50% probability are used for the non-H atoms: the H atoms are represented as spheres equivalent to  $B = 1.0 \text{ Å}^2$ .

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances and angles involving H atoms, selected torsion angles, and deviations from least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42950 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates of the atoms and their isotropic temperature factors ( $\text{\AA}^2$ ) with e.s.d.'s in parentheses

	x	y	z	$U_{eq}$
<b>Molecule A</b>				
Cl(1)	0.0784 (1)	0.7401 (1)	-0.1467 (1)	0.09
Cl(2)	-0.0302 (1)	0.8794 (1)	-0.2867 (1)	0.08
O(1)	0.1035 (2)	0.8946 (2)	-0.3834 (2)	0.08
O(2)	0.3910 (2)	1.0145 (2)	0.1742 (3)	0.05
N	0.3497 (2)	0.8685 (2)	0.1477 (3)	0.07
C(1)	0.2121 (2)	0.8752 (2)	-0.1922 (3)	0.05
C(2)	0.2415 (2)	0.8745 (2)	-0.0549 (3)	0.05
C(3)	0.3221 (2)	0.8713 (2)	0.0070 (3)	0.05
C(4)	0.3770 (2)	0.8699 (2)	-0.0660 (3)	0.05
C(5)	0.3468 (2)	0.8721 (2)	-0.2051 (3)	0.06
C(6)	0.2668 (2)	0.8744 (2)	-0.2647 (3)	0.06
C(7)	0.1272 (2)	0.8782 (2)	-0.2668 (3)	0.05
C(8)	0.0677 (2)	0.8572 (3)	-0.1920 (3)	0.06
C(9)	0.4645 (2)	0.8645 (3)	0.0028 (4)	0.07
C(10)	0.4036 (3)	0.8715 (4)	-0.2884 (4)	0.09
C(11)	0.3836 (2)	0.9402 (2)	0.2224 (3)	0.05
C(12)	0.4127 (3)	0.9226 (3)	0.3676 (4)	0.08
<b>Molecule B</b>				
Cl(1)	-0.1019 (1)	1.2256 (1)	-0.3559 (2)	0.11
Cl(2)	-0.1879 (1)	1.0659 (1)	-0.3158 (1)	0.09
O(1)	-0.0581 (2)	1.0904 (2)	-0.0842 (3)	0.08
O(2)	0.1746 (2)	1.2216 (2)	0.1893 (3)	0.08
N	0.2403 (2)	1.1186 (2)	0.1003 (3)	0.05
C(1)	0.0507 (2)	1.1084 (2)	-0.1697 (3)	0.05
C(2)	0.1053 (2)	1.1102 (2)	-0.0444 (3)	0.05
C(3)	0.1853 (2)	1.1166 (2)	-0.0289 (3)	0.05
C(4)	0.2141 (2)	1.1190 (2)	-0.1386 (3)	0.05
C(5)	0.1590 (2)	1.1161 (2)	-0.2658 (3)	0.05
C(6)	0.0790 (2)	1.1108 (2)	-0.2794 (3)	0.05
C(7)	-0.0345 (2)	1.1011 (2)	-0.1790 (3)	0.05
C(8)	-0.0947 (2)	1.1085 (3)	-0.3141 (3)	0.06
C(9)	0.3019 (2)	1.1233 (2)	-0.1234 (3)	0.06
C(10)	0.1868 (2)	1.1185 (3)	-0.3870 (3)	0.06
C(11)	0.2309 (2)	1.1701 (3)	0.2012 (3)	0.06
C(12)	0.2943 (3)	1.1596 (3)	0.3302 (4)	0.08

 Table 2. Bond distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with e.s.d.'s in parentheses

	Molecule A	Molecule B
Cl(1)—C(8)	1.776 (4)	1.766 (5)
Cl(2)—C(8)	1.752 (3)	1.754 (4)
O(1)—C(7)	1.205 (4)	1.206 (5)
O(2)—C(11)	1.225 (4)	1.224 (5)
N—C(3)	1.425 (4)	1.425 (4)
N—C(11)	1.344 (4)	1.358 (5)
C(1)—C(2)	1.393 (4)	1.391 (4)
C(2)—C(3)	1.380 (4)	1.375 (5)
C(3)—C(4)	1.407 (5)	1.400 (5)
C(4)—C(5)	1.412 (4)	1.410 (4)
C(5)—C(6)	1.366 (4)	1.378 (5)
C(6)—C(1)	1.400 (5)	1.394 (5)
C(1)—C(7)	1.473 (4)	1.481 (5)
C(7)—C(8)	1.522 (5)	1.513 (4)
C(9)—C(4)	1.502 (4)	1.511 (5)
C(5)—C(10)	1.519 (7)	1.504 (5)
C(11)—C(12)	1.493 (5)	1.496 (5)
Cl(1)—C(8)—Cl(2)	110.0 (2)	110.2 (2)
Cl(1)—C(8)—C(7)	107.5 (3)	106.6 (3)
Cl(2)—C(8)—C(7)	112.3 (2)	112.2 (2)
O(1)—C(7)—C(1)	123.1 (3)	123.2 (3)
O(1)—C(7)—C(8)	119.4 (3)	118.6 (3)
O(2)—C(11)—N	122.1 (3)	123.1 (3)
O(2)—C(11)—C(12)	122.4 (3)	121.5 (3)
N—C(11)—C(12)	115.4 (3)	115.4 (3)
N—C(3)—C(2)	118.9 (3)	120.0 (3)
N—C(3)—C(4)	119.8 (3)	119.1 (3)
C(2)—C(1)—C(6)	118.0 (3)	118.5 (3)
C(2)—C(1)—C(7)	124.5 (3)	117.9 (3)
C(7)—C(1)—C(6)	117.6 (3)	123.6 (3)
C(1)—C(2)—C(3)	120.7 (3)	120.8 (3)
C(2)—C(3)—C(4)	121.3 (3)	121.0 (3)
C(3)—C(4)—C(5)	117.7 (3)	118.4 (3)
C(3)—C(4)—C(9)	120.7 (3)	121.7 (3)
C(5)—C(4)—C(9)	121.6 (3)	119.9 (3)
C(4)—C(5)—C(6)	120.2 (3)	119.8 (3)
C(4)—C(5)—C(10)	119.8 (3)	120.6 (3)
C(6)—C(5)—C(10)	120.1 (3)	119.6 (3)
C(5)—C(6)—C(1)	122.2 (3)	121.5 (3)
C(8)—C(7)—C(1)	117.6 (3)	118.2 (3)
C(3)—N—C(11)	123.3 (3)	124.1 (3)

hino, Kawai & Maeda, 1976) forms of *p*-hydroxyacetanilide, and in *p*-aminoacetanilide [1.421 (4)  $\text{\AA}$ ] (Haisa, Kashino, Matsuzaki, Kawai & Kunitomi, 1977). The extent of delocalization of the carbonyl  $\pi$ -electron cloud of the amide moieties in molecules *A* and *B* is comparable with that in *p*-hydroxyacetanilide, orthorhombic and monoclinic forms, and in *p*-aminoacetanilide; this carbonyl bond is similar in acetanilide [1.219 (3)  $\text{\AA}$ ] (Brown, 1966). Also the average of the two N—C(11) bond lengths in the present structure is comparable with the corresponding bond lengths in these structures [1.341 (6)  $\text{\AA}$  in *p*-hydroxyacetanilide, orthorhombic form; 1.340 (3)  $\text{\AA}$  in *p*-hydroxyacetanilide, monoclinic form; 1.344 (4)  $\text{\AA}$  in *p*-aminoacetanilide].

The Cl(1)—C(8)—Cl(2) bond angle and the Cl(1)—C(8) and Cl(2)—C(8) bond lengths in the present structure are comparable to the corresponding values in chloroamphenicol [110.2 (6) $^\circ$ , 1.789 (11) and 1.753 (11)  $\text{\AA}$ ] (Ravindra Acharya, Sake Gowda & Post, 1979), in 4'-amino-2,2,3'-trichloroacetophenone [110.0 (4) $^\circ$ , 1.774 (7) and 1.757 (7)  $\text{\AA}$ ] (De, 1984) and in 4-methyl-3-chloro-6-dichloroacetylacetanilide [109.6 (1) $^\circ$ , 1.783 (2) and 1.754 (2)  $\text{\AA}$ ] (Banerjee, 1983). The keto bond lengths [1.205 (4), 1.206 (5)  $\text{\AA}$ ]

in the dichloroacetyl moieties of each of the molecules are significantly shorter than the similar bonds in 4'-amino-2,2,3'-trichloroacetophenone [1.234 (8)  $\text{\AA}$ ] (De, 1984) and in 2,2-dichloro-2'-hydroxy-4'-methoxyacetophenone [1.229 (7)  $\text{\AA}$ ] (Chattopadhyay *et al.*, 1985), but similar to that in 4-methyl-3-chloro-6-dichloroacetylacetanilide [1.207 (2)  $\text{\AA}$ ] (Banerjee, 1983). The average C—C(phenyl) bond length in the present structure is similar to the corresponding bond length in 4-methyl-3-chloro-6-dichloroacetylacetanilide [1.485 (2)  $\text{\AA}$ ] (Banerjee, 1983) but longer than that in 2,2-dichloro-2'-hydroxy-4'-methoxyacetophenone [1.457 (7)  $\text{\AA}$ ] (Chattopadhyay *et al.*, 1985). This lengthening of the C( $sp^2$ )—C( $sp^2$ ) bond indicates a smaller extent of conjugation between the benzene ring and the dichloroacetyl group and is due to the more localized nature of the adjacent carbonyl  $\pi$ -electron cloud in the present structure.

Molecular packing in the crystal is shown in Fig. 2. The molecules in the asymmetric units are linked through N<sub>B</sub>—H(1)<sub>B</sub>...O(2)<sub>A</sub> hydrogen bonds [N—H(1) 0.84, H(1)...O(2) 2.13, N...O(2) 2.963 (5)  $\text{\AA}$ ; N—H(1)...O(2) = 170 $^\circ$ ]. Additional N<sub>A</sub>—H(1)...O(2)<sub>B</sub> hydrogen bonds [N—H(1) 0.72, H(1)...O(2) 2.18, N...O(2) 2.869 (5)  $\text{\AA}$ ; N—H(1)...O(2) = 163 $^\circ$ ] be-

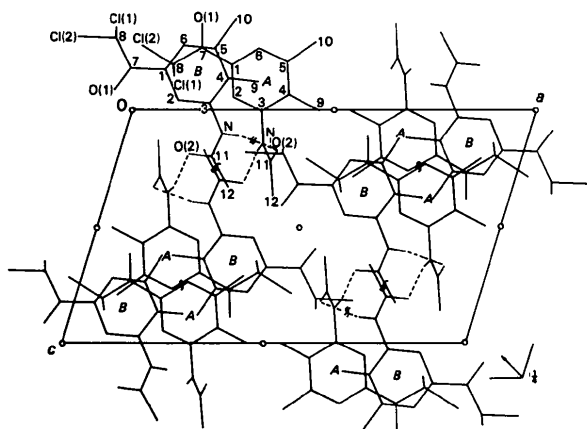


Fig. 2. Perspective view of the molecular packing.

tween the asymmetric units related by the twofold screw axis at  $x = \frac{1}{4}$  and  $z = \frac{1}{4}$  result in the formation of infinite chains. In the chains the benzene-ring planes of the molecules *A* and *B* lie nearly perpendicular to *b* and parallel with each other, the dihedral angle being  $4.8(1)^\circ$ . The Cl(2) atoms of molecules *A* and *B* lie in the respective ring planes, and the Cl(1)—C(8) bonds in the molecules *A* and *B* extend nearly parallel to the twofold screw axis, but in opposite directions, resulting in compact packing of the molecules in the chain. The chains are held together by van der Waals interactions to form a sheet in (100). In the sheet the methyl groups at C(4) and C(5) of molecule *B* are sandwiched between the benzene rings of molecules *A* related by the twofold screw axis at  $x = \frac{1}{4}$  and  $z = \frac{3}{4}$ . The sheets are stacked along *a* by weak van der Waals interactions to complete the structure.

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## Structure of 14 $\alpha$ ,17 $\alpha$ -Epoxy-3-methoxy-14-methyl-14 $\alpha$ -estra-1,3,5(10)-trien-17 $\beta$ -ol

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**Abstract.** C<sub>20</sub>H<sub>26</sub>O<sub>3</sub>,  $M_r = 314.4$ , trigonal,  $P3_221$ ,  $a = b = 7.1677(5)$ ,  $c = 55.370(5)$  Å,  $V = 2463.6$  Å<sup>3</sup>,  $Z = 6$ ,  $D_x = 1.27$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54184$  Å,  $\mu = 5.8$  cm<sup>-1</sup>,  $F(000) = 1020$ ,  $T = 293$  K,  $R = 0.0387$  for 2098 unique reflections. The atoms forming ring *A* do not deviate significantly from the least-squares plane. Ring *B* is found to adopt a <sup>2</sup>H<sub>3</sub> half-chair conformation, whereas ring *C* is in the expected <sup>4</sup>C<sub>1</sub> chair form. The molecules are linked by O—H...O hydrogen bonds.

**Introduction.** A novel cycloaddition approach to the synthesis of estrone analogues bearing functionalized 14 $\alpha$ -alkyl groups has recently been described (Bull & Thomson, 1986). Selective reduction of 3-methoxy-17-oxo-1,3,5(10)-estratriene-14 $\alpha$ -carbaldehyde results in formation of the corresponding 14 $\alpha$ -hydroxymethyl compound (I), the spectroscopic properties of which revealed that it exists in solution as an equimolar mixture of (I) and the title hemiacetal (II).

An X-ray crystallographic structure determination

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