

resulting in three intermolecular contacts $<3.2 \text{ \AA}$ involving C and O atoms [O(6)···C(14) = $3.168 (5)$, C(2)···O(16) = $3.136 (5)$ and C(3)···O(16) = $3.132 (5) \text{ \AA}$].

One of us (MMB) thanks the Council of Scientific and Industrial Research, India, for the financial assistance of this work.

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Acta Cryst. (1982). **B38**, 1857–1859

Structure of 1-(4-Chlorophenoxy)-3,3-dimethyl-1-(1,2,4-triazol-1-yl)-2-butanone

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(Received 30 November 1981; accepted 22 January 1982)

Abstract. $C_{14}H_{16}ClN_3O_2$, $M_r = 293.8$, $P2_1/n$, $a = 8.076 (3)$, $b = 20.317 (8)$, $c = 9.307 (3) \text{ \AA}$, $\beta = 97.43 (5)^\circ$, $U = 1514.2 \text{ \AA}^3$, $Mo K\alpha$, $\lambda = 0.71069 \text{ \AA}$, $Z = 4$, $D_m = 1.31 (2)$, $D_c = 1.29 \text{ Mg m}^{-3}$, $\mu = 0.21 \text{ mm}^{-1}$, $F(000) = 616$. The structure was solved by direct methods and refined to $R = 0.058$ for 1023 counter reflections classed as observed. The triazolyl ring, which shows significant delocalization, is planar and inclined at angles of $73.1 (9)$ and $61.8 (9)^\circ$ to the *p*-chlorophenyl ring and to the C–C(O)–C grouping respectively. The exocyclic angles at N(1) of the triazolyl ring are very asymmetric with C–N–N $119.5 (6)$ and C–N–C $130.7 (6)^\circ$.

Introduction. 1-(4-Chlorophenoxy)-3,3-dimethyl-1-(1,2,4-triazol-1-yl)-2-butanone, triadimefon, is well established as an effective systemic fungicide (Martin & Morris, 1979). While the exact mode of action of the fungicide has not been established, treatment of fungi with the compound results in the inhibition of ergosterol biosynthesis (Buchenaer, 1976). From the study of structure–activity relationships in related compounds, it was believed that the triazolyl group and the overall conformation of the molecule played important roles. It is to establish the conformation, albeit in the solid state, that we have determined the crystal structure of the title compound.

A crystal $0.20 \times 0.24 \times 0.35 \text{ mm}$ was mounted about the crystallographic *a* axis and data were collected on a Stoe Stadi-2 two-circle diffractometer in layers of constant *h* up to $h = 9$. The data were

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Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$) calculated by reference to Willis & Pryor (1975); *e.s.d.*'s are in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} (Å ²)
Cl	-2894 (4)	9666 (2)	5907 (3)	10 (3)
O(1)	1799 (8)	8481 (3)	-1736 (5)	76 (5)
O(2)	186 (7)	8950 (2)	718 (5)	45 (4)
N(1)	-52 (9)	7815 (3)	150 (6)	39 (4)
N(2)	107 (11)	7226 (3)	818 (7)	71 (6)
N(3)	-1929 (10)	7193 (4)	-1075 (7)	62 (5)
C(1)	-1261 (12)	7787 (4)	-966 (7)	52 (6)
C(2)	-1047 (16)	6880 (4)	33 (10)	76 (7)
C(31)	4079 (11)	8674 (3)	147 (7)	46 (5)
C(32)	4097 (14)	9155 (4)	1423 (9)	65 (6)
C(33)	5000 (13)	8961 (4)	-1056 (9)	66 (6)
C(34)	4908 (12)	8017 (4)	670 (8)	60 (6)
C(4)	2292 (10)	8505 (3)	-468 (7)	43 (5)
C(5)	1076 (10)	8345 (3)	654 (7)	40 (5)
C(71)	-525 (11)	9081 (4)	1989 (7)	45 (5)
C(72)	-802 (12)	8613 (4)	2992 (8)	54 (6)
C(73)	-1521 (13)	8797 (4)	4222 (8)	63 (7)
C(74)	-1963 (12)	9438 (5)	4370 (8)	61 (6)
C(75)	-1677 (12)	9909 (4)	3367 (10)	61 (6)
C(76)	-964 (12)	9727 (4)	2160 (8)	52 (5)

processed to give 2773 planes of which 1023 had $I \geq 2.5\sigma(I)$ and were classed as observed. Systematic absences were $h0l$, $h + l = 2n + 1$, and $0k0$, $k = 2n + 1$, corresponding to space group $P2_1/n$. The structure was solved by multiresolution direct methods (Sheldrick, 1976). The H atoms were included in positions calculated from the geometry of the molecule (C-H = 1.08 Å). A final isotropic $U = 0.049 (20) \text{ \AA}^2$ was refined for H(5) while the common isotropic temperature factors applied to the methyl and to the remaining H atoms refined to $U = 0.081 (7)$ and $0.082 (10) \text{ \AA}^2$ respectively. Scattering factors were calculated using an analytical approximation (*International Tables for X-ray Crystallography*, 1974) and the weighting scheme adopted was $w = 0.8614/[\sigma^2(F_o) + 0.0016(F_o)^2]$. Full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms gave the final $R = 0.058$ and $R' = 0.063$. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.*

Discussion. Bond lengths and angles are given in Table 2, torsion angles in Table 3, and mean planes in Table 4. The molecule is found to adopt a conformation such that the triazolyl ring is inclined at an angle of $73.1 (9)^\circ$ to the aromatic ring and at an angle of

Table 2. Bond distances (Å) and angles ($^\circ$) with *e.s.d.*'s in parentheses

N(1)-N(2)	1.348 (8)	C(31)-C(33)	1.537 (12)
N(1)-C(1)	1.332 (10)	C(31)-C(34)	1.544 (10)
N(2)-C(2)	1.311 (13)	O(2)-C(71)	1.406 (9)
C(2)-N(3)	1.336 (12)	C(71)-C(72)	1.371 (11)
N(3)-C(1)	1.319 (11)	C(71)-C(76)	1.374 (11)
N(1)-C(5)	1.449 (9)	C(72)-C(73)	1.399 (12)
C(5)-C(4)	1.558 (11)	C(73)-C(74)	1.363 (14)
C(5)-O(2)	1.429 (9)	C(74)-C(75)	1.377 (13)
C(4)-O(1)	1.196 (8)	C(75)-C(76)	1.378 (13)
C(4)-C(31)	1.521 (11)	C(74)-Cl	1.761 (9)
C(31)-C(32)	1.537 (10)		
C(5)-N(1)-N(2)	119.5 (6)	C(4)-C(31)-C(34)	105.9 (6)
C(5)-N(1)-C(1)	130.7 (6)	C(32)-C(31)-C(33)	111.4 (6)
C(1)-N(1)-N(2)	109.7 (6)	C(32)-C(31)-C(34)	110.2 (6)
N(1)-N(2)-C(2)	101.6 (6)	C(33)-C(31)-C(34)	109.1 (7)
N(2)-C(2)-N(3)	116.3 (8)	C(5)-O(2)-C(71)	117.4 (5)
C(1)-N(3)-C(2)	101.8 (7)	O(2)-C(71)-C(72)	124.2 (7)
N(1)-C(1)-N(3)	110.5 (7)	O(2)-C(71)-C(76)	114.7 (6)
N(1)-C(5)-C(4)	111.3 (5)	C(72)-C(71)-C(76)	121.1 (7)
N(1)-C(5)-O(2)	110.7 (6)	C(71)-C(72)-C(73)	119.5 (8)
C(4)-C(5)-O(2)	102.3 (5)	C(72)-C(73)-C(74)	118.7 (8)
C(5)-C(4)-O(1)	119.6 (7)	C(73)-C(74)-C(75)	121.8 (8)
C(5)-C(4)-C(31)	116.4 (6)	C(73)-C(74)-Cl	118.5 (7)
O(1)-C(4)-C(31)	124.0 (7)	Cl-C(74)-C(75)	119.6 (7)
C(4)-C(31)-C(32)	110.4 (7)	C(74)-C(75)-C(76)	119.3 (8)
C(4)-C(31)-C(33)	109.7 (6)	C(75)-C(76)-C(71)	119.5 (7)

Table 3. Torsion angles ($^\circ$) with *e.s.d.*'s in parentheses

C(5)-O(2)-C(71)-C(72)	-17.6 (10)	O(2)-C(5)-C(4)	C(31)	100.1 (7)
C(5)-O(2)-C(71)-C(76)	163.4 (7)	N(1)-C(5)-C(4)-O(1)		37.3 (9)
C(71)-O(2)-C(5)-N(1)	86.0 (7)	N(1)-C(5)-C(4)-C(31)		-141.6 (6)
C(71)-O(2)-C(5)-C(4)	-155.4 (6)	O(1)-C(4)-C(31)-C(32)		136.3 (8)
O(2)-C(5)-N(1)-N(2)	-136.4 (7)	O(1)-C(4)-C(31)-C(33)		13.3 (10)
O(2)-C(5)-N(1)-C(1)	47.1 (10)	O(1)-C(4)-C(31)-C(34)		-104.3 (8)
C(4)-C(5)-N(1)-N(2)	110.6 (7)	C(5)-C(4)-C(31)-C(32)		44.8 (8)
C(4)-C(5)-N(1)-C(1)	-66.0 (10)	C(5)-C(4)-C(31)-C(33)		167.8 (6)
O(2)-C(5)-C(4)-O(1)	-80.9 (8)	C(5)-C(4)-C(31)-C(34)		74.6 (8)

Table 4. Equations of least-squares planes referred to orthogonal axes with distances (Å) of atoms from the planes in square brackets

Plane A: N(1), N(2), N(3), C(1), C(2)	$0.7512X - 0.2781Y - 0.5986Z + 4.5433 = 0.0000$
[N(1), 0.0001 (66); N(2), -0.0000 (79); N(3), 0.0002 (76); C(1), -0.0002 (86); C(2), -0.0001 (114); C(5), 0.061 (7)]	
Plane B: C(71)-C(76)	$-0.8494X - 0.1808Y - 0.4958Z + 3.6807 = 0.0000$
[C(71), -0.001 (8); C(72), 0.004 (9); C(73), -0.007 (10); C(74), 0.008 (9); C(75), -0.005 (10); C(76), 0.002 (9); Cl, 0.017 (3); O(2), 0.011 (5)]	
Plane C: C(5), C(4), O(1), C(31)	$0.2561X - 0.9664Y - 0.0202Z + 16.1969 = 0.0000$
[C(5), 0.002 (6); C(4), -0.006 (6); O(1), 0.002 (6); C(31), 0.002 (6)]	

Angles between planes ($^\circ$), *e.s.d.*'s 0.9-1.5 $^\circ$

A-B	73.1	A-C	61.8	B-C	88.1
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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36692 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

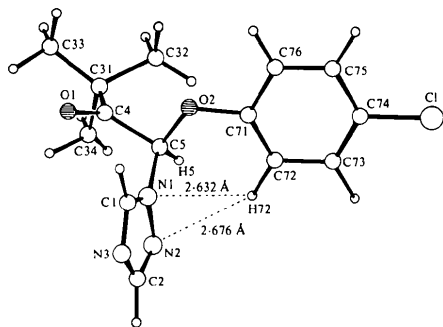


Fig. 1. The conformation of 1-(4-chlorophenoxy)-3,3-dimethyl-1-(1,2,4-triazol-1-yl)-2-butanone.

61.8 (9)° to the C(31), C(4), O(1), C(5) grouping (Fig. 1). The resulting arrangement leads to the close approach of the *ortho*-H, H(72), to the triazolyl atoms N(1) and N(2), such that both N...H distances lie within the sum of the van der Waals radii of N and H (Fig. 1). There is an accompanying distortion of the exocyclic angles at C(71), with the O(2)–C(71)–C(72) bond angle of 124.2 (7)° being considerably larger than the value found for O(2)–C(71)–C(76) [114.7 (6)°].

The triazolyl ring is planar, with C(5) lying only 0.061 (7) Å from the mean plane (Table 4). Although the C(1)–N(1) and C(2)–N(3) distances are somewhat larger than those found for C(1)–N(3) and C(2)–N(2), in keeping with the uncharged canonical valence form, all four C–N distances are shorter than a normal single bond (1.47 Å). The N(1)–N(2) bond is also shorter than a normal single bond (1.45 Å) and

the three atoms bonded to N(1) are almost coplanar with it. Taken together these data indicate extensive delocalization within the heterocyclic ring. The most noteworthy feature of the heterocyclic ring is the asymmetry of the exocyclic angles at N(1) [119.5, 130.7 (6)°]. We have observed a similar pattern in related triazole systems (Anderson, Branch, Mann, Nowell & Walker, 1971, unpublished results) and it appears to be a function of the triazolyl ring itself rather than the influence of any inter- or intramolecular interactions.

The C(31), C(4), C(5), O(2), C(71) backbone is rather compressed resulting in the main from the orientation of the *tert*-butyl group, the C(31)–C(4)–C(5)–O(2) torsion angle being only 100.1 (7)°.

We thank the SRC for an equipment grant and computing facilities (IWN), Imperial Chemical Industries Limited for financial support and W. Krämer (Bayer AG) for helpful discussions.

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Acta Cryst. (1982). **B38**, 1859–1862

Structure of the Benzylammonium Perchlorate Complex of 3,6,9,12,15,18,21,26-Octaoxabicyclo[21.2.1]hexacosal(25),23-diene-2,22-dione

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(Received 21 October 1981; accepted 15 January 1982)

Abstract. C₁₈H₂₆O₁₀·C₇H₁₀N⁺·ClO₄⁻, *M_r* = 610.01, *F*(000) = 1288, monoclinic, *P*2₁/*c*, *a* = 11.710 (2), *b* = 15.087 (5), *c* = 17.574 (3) Å, β = 105.68 (2)° at *T* = 173 K, *Z* = 4, *V* = 2989.2 (13) Å³, *D_x* = 1.355, *D_m* (at

295 K) = 1.334 Mg m⁻³ (measured by flotation in CCl₄/*n*-hexane), colorless, m.p. 366.5–368 K. *R* = 0.040, *R_w* = 0.037 for 3645 reflections. The macrocyclic ligand is hydrogen-bonded to the organic cation