3,5-Bis(3-chlorophenyl)-4-formyl-2,6-dimethyl-cyclohexan-1-one

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.007 Å
R factor = 0.042
wR factor = 0.100
Data-to-parameter ratio = 8.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title molecule, $C_{21}H_{20}Cl_2O_2$, reveals a chair conformation for the cyclohexane ring in which all the substituents occupy equatorial positions.

Comment

During attempts to prepare a pyran derivative by the Michael condensation of diethyl ketone and o-chlorobenzaldehyde, an unexpected product, the title compound, (I) (Fig. 1), was obtained as a minor product. The present investigation was undertaken to ascertain the structure and conformation of (I), since they cannot be assigned conclusively from ^{1}H NMR data.

(I)
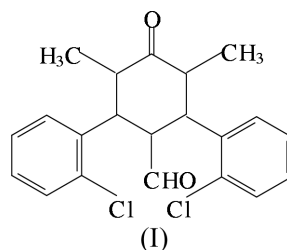
The cyclohexane ring is in a chair conformation, as shown by the torsion angles (Table 1) around the C—C bonds involving the six C atoms (C1–C6). The torsion angles deviate from the angle of 56° expected for a perfect chair conformation (Kalsi, 1997) as a result of the sp^2-hybridized atom C1. The bond lengths and angles within the cyclohexane ring are in the ranges 1.503 (6)–1.551 (6) Å and 106.6 (3)–116.9 (4)°, respectively. The equatorial orientations of all the substituents are confirmed by the torsion angles of about 180° formed by the external atom and the other three ring atoms. The dihedral angle between the planes of the phenyl rings is 61.9 (2)°. The chair conformation observed in (I) is comparable to that observed in the thioketal of N-methyl-2,6-diphenyl-3-iso-
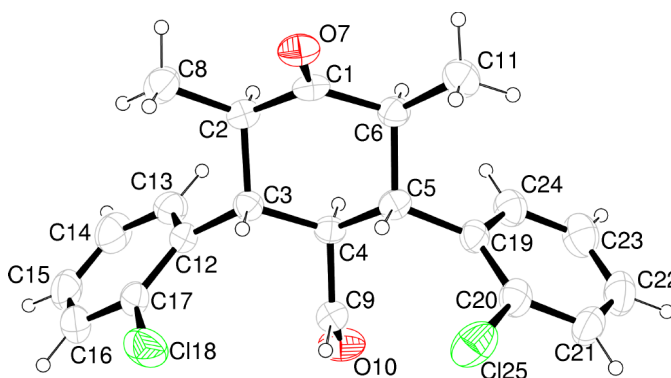

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

propylpiperdin-4-one (Sujatha, 1995), but contrasts with the twist-boat conformation of the *N*-nitroso derivative of 2,6-bis(2-chlorophenyl-3,5-dimethylpiperidin-4-one (Sukumar *et al.*, 1993). Atoms O10, C9, C4, O7 and C1 are coplanar, the maximum deviation being 0.014 (2) Å for O10; the molecule is nearly symmetrical with respect to this plane.

The crystal structure is stabilized by C—H···O interactions, the most significant of which are (C9)H9···O7($-1 + x$, y, z) and (C16)H16···O7($1 - x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$) with H···O distances of 2.45 and 2.42 Å, and C—H···O angles of 131 and 147°, respectively. The former interactions contribute to chain formation along the *a* axis; the chains are interlinked by the latter interactions. Another intermolecular interaction exists between atoms C4 and O10($\frac{1}{2} + x$, $\frac{1}{2} - y$, $-z$) through atom H4, with an H···O distance of 2.56 Å and an angle of 161° at H4. The formation of (I), although unexpected, can be explained by the condensation of the dibenzylideneacetone derivative formed *in situ* with the acetaldehyde present in ethanol.

Experimental

The title compound was obtained by the reaction of diethyl ketone with *o*-chlorobenzaldehyde in 80% ethanol. Diffraction quality crystals were obtained by recrystallization from ethanol. ^{1}H NMR (CDCl$_3$, p.p.m.): δ 0.92 (*d*, 6H, $J = 6.3$), 2.87 (*m*, 2H), 3.93 (*t*, 2H, $J = 12.0$), 3.38 (*dt*, 1H, $^3J = 4.8$, $^2J = 12.2$), 7.1–7.8 (*m*, 8H), 9.21 (*d*, 1H, $J = 4.5$).

Crystal data

C$_{21}$H$_{20}$Cl$_2$O$_2$	Mo $K\alpha$ radiation
$M_r = 375.27$	Cell parameters from 25
Orthorhombic, $P2_12_12_1$	reflections
$a = 7.635$ (2) Å	$\theta = 2$–12°
$b = 12.1814$ (15) Å	$\mu = 0.36$ mm^{-1}
$c = 20.175$ (4) Å	$T = 293$ (2) K
$V = 1876.4$ (7) Å^3	Translucent block, colourless
$Z = 4$	$0.2 \times 0.2 \times 0.1$ mm
$D_x = 1.328$ Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	1399 reflections with $I > 2\sigma(I)$
ω–2θ scans	$\theta_{max} = 25.0°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 9$
	$k = 0 \rightarrow 14$
$T_{min} = 0.982$, $T_{max} = 0.999$	$l = 0 \rightarrow 23$
1904 measured reflections	3 standard reflections
1904 independent reflections	every 100 reflections
	intensity decay: negligible

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$+ 0.6059P]$
$wR(F^2) = 0.100$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} < 0.001$
1904 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
226 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C6—C1—C2—C3	57.4 (5)	C3—C4—C5—C6	−61.8 (4)
C1—C2—C3—C4	−56.5 (4)	C2—C1—C6—C5	−58.4 (5)
C2—C3—C4—C5	61.5 (4)	C4—C5—C6—C1	57.9 (4)

Since the Friedel pairs were not measured during the data collection, we were unable to determine the absolute structure; in any case, the molecule is achiral. All the H atoms were fixed geometrically and allowed to ride on the parent C atoms, with aromatic C—H = 0.93 Å, tertiary C—H = 0.97 Å and methyl C—H = 0.96 Å. The displacement parameters U_{iso}(H) were set at 1.5U_{eq}(C) for the methyl H atoms and at 1.2U_{eq}(C) for other H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL*97.

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