organic papers

Di-*tert*-butyl 4-oxocyclohexane-1,1-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.150
Data-to-parameter ratio = 21.4

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

In the title compound, $C_{16}H_{26}O_5$, the cyclohexanone ring has a flattened chair conformation, with absolute values of the endocyclic torsion angles lying in the range 39.7 (3)–58.0 (2)°.

Comment

The title compound, (I), was synthesized as a precursor in the synthesis of an α,α-disubstituted amino acid having the potential to stabilize the 3_{10}-helical secondary structure of a peptide in aqueous media. The crystal structure was determined in order to confirm its identity. Cooling the sample for data collection was limited to 190 K, as crystals were destroyed at lower temperatures.

(I)
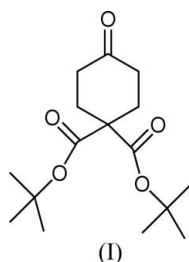
The six-membered ring exists in a flattened chair conformation, as indicated by the torsion angles in Table 1. This flattening, which results from the presence of the ketone group, is similar to that seen in 4,4-dimethylcyclohexanone [Cambridge Structural Database (CSD; Version 5.27; Allen, 2002) refcode DMCYHX (Lichanot *et al.*, 1977)] and 4,4-
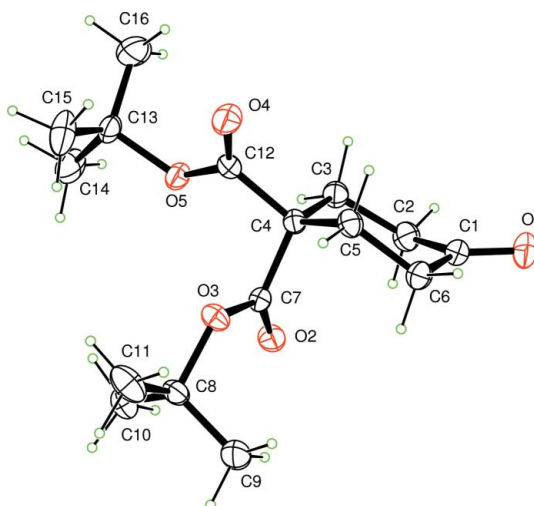

Figure 1
View of (I), showing displacement ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary radius.

diphenylcyclohexanone (CSD refcode DPHCX10; Lambert *et al.*, 1969). The cyclohexanone ring in (I), however, is more distorted from local mirror symmetry than in either of those compounds. The mean difference of three torsion angle magnitudes across the local mirror is 7.9° in (I), 4.6° in DPHCX10, and zero for DMCYHX, which lies on a crystallographic mirror. The *tert*-butoxycarbonyl groups in (I) have conformations such that their carbonyl groups are approximately eclipsed with the C3—C4 and C4—C7 bonds (Table 1).

Experimental

Compound (I) was prepared by the method of Sanchez *et al.* (1985) from 2-methoxycarbonyl-4,4-di-*tert*-butoxycarbonylcyclohexanone. Crystals were grown by slow evaporation of a dimethyl sulfoxide solution.

Crystal data

$C_{16}H_{26}O_5$	$Z = 4$
$M_r = 298.37$	$D_x = 1.136$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.3412$ (3) Å	$\mu = 0.08$ mm^{-1}
$b = 16.186$ (2) Å	$T = 190$ K
$c = 17.211$ (2) Å	Lath, colorless
$\beta = 99.096$ (6)°	$0.43 \times 0.28 \times 0.13$ mm
$V = 1744.3$ (3) Å^3	

Data collection

Enraf–Nonius CAD-4 diffract-ometer with an Oxford Cryosystems Cryostream cooler	2173 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.038$
ω–2θ scans	$\theta_{max} = 28.0°$
Absorption correction: none	3 standard reflections
6463 measured reflections	frequency: 60 min
4198 independent reflections	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$+ 0.2515P]$
$wR(F^2) = 0.150$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
4198 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
196 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C1—C2—C3—C4	−43.1 (3)	C5—C6—C1—C2	−45.9 (3)
C2—C3—C4—C5	52.4 (2)	C6—C1—C2—C3	39.7 (3)
C3—C4—C5—C6	−58.0 (2)	C3—C4—C7—O2	8.8 (3)
C4—C5—C6—C1	55.1 (2)	C5—C4—C12—O4	12.9 (3)

All H atoms were placed in idealized positions (C—H = 0.98–0.99 Å) and refined as riding, with the constraint $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). A torsional parameter was refined for each methyl group.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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