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Diisopropyl 3-methyl-5-oxocyclohex-3-ene-1,1-dicarboxylate

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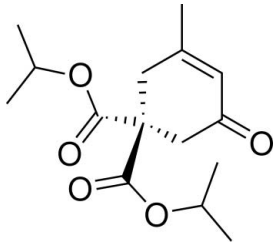
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.123; data-to-parameter ratio = 19.6.

In the title compound, $C_{15}H_{22}O_5$, the cyclohexenone ring has an envelope conformation; the flap atom (with the ester groups attached) is displaced by 0.624 (2) Å from the plane of the other five ring atoms. The crystal structure contains weak intra- and intermolecular C—H···O hydrogen bonds.

Related literature

For related literature, see: Hu *et al.* (2003); Li & Strobel (2001); Luu *et al.* (2004).



Experimental

Crystal data

$C_{15}H_{22}O_5$
M_r = 282.34
Monoclinic, $P2_1/n$
a = 12.302 (3) Å
b = 8.909 (3) Å
c = 14.409 (6) Å
β = 96.859 (14)°
V = 1568.0 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (1) K
0.32 × 0.28 × 0.26 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: none
15048 measured reflections
3564 independent reflections
2299 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.123
S = 1.01
3564 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.98	2.47	3.333 (2)	147
C12—H12···O5	0.98	2.35	2.707 (2)	101

Symmetry code: (i) $x, y-1, z$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

Mr Jianming Gu of the X-ray crystallography facility of Zhejiang University is acknowledged for assistance with the crystal structural analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2144).

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supplementary materials

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Diisopropyl 3-methyl-5-oxocyclohex-3-ene-1,1-dicarboxylate

B.-S. Wang, C. Zhang and L.-Y. Yao

Comment

The cyclohex-2-enone ring system is highly reactive and derivatives can be used to synthesize some complex compounds, such as vitamin E, amino acids, terpenes *etc*. (Hu *et al.*, 2003). In addition, cyclohex-2-enone derivatives have shown a wide range of biological activities such as antimicrobial (Li *et al.*, 2001) and the protection of cerebral neurocytes (Luu *et al.*, 2004). We are interested in their pharmaceutical activity. In this paper, we present an X-ray crystallographic analysis of the title compound. The plane which is composed of C1,C2,C6 and the plane which is composed of C2,C3, C4,C5,C6 form a dihedral angle of 44.9 (2)°.

Experimental

A solution of 4,4-di(isopropyloxycarbonyl)-2,6-heptanedione (300 mg, 1 mmol) and sodium methoxide (54 mg, 1 mmol) in methanol (10 ml) was heated at 323 K for 4 h. The reaction mixture was acidified with dilute aqueous HCl, then concentrated and partitioned between water and dichloromethane. The pure product was obtained through silica gel chromatography (eluant petroleum ether/ethyl acetate, 5:1), and diffraction quality crystals were obtained by slow evaporation of a diethyl ether/hexane (1:3) solu- tion at room temperature.

Refinement

All H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.98 Å and included in the final cycles of refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

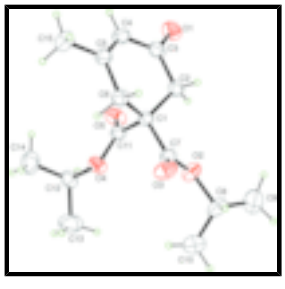

Fig. 1. The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

Diisopropyl 3-methyl-5-oxocyclohex-3-ene-1,1-dicarboxylate

Crystal data

$C_{15}H_{22}O_5$	$F_{000} = 608.00$
$M_r = 282.34$	$D_x = 1.196$ Mg m^{-3}

Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71075$ Å
Hall symbol: -P 2yn	Cell parameters from 11337 reflections
$a = 12.302$ (3) Å	$\theta = 3.1–27.5°$
$b = 8.909$ (3) Å	$\mu = 0.09$ mm^{-1}
$c = 14.409$ (6) Å	$T = 298$ (1) K
$\beta = 96.859$ (14)°	Block, colorless
$V = 1568.0$ (9) Å^3	0.32 × 0.28 × 0.26 mm
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID diffractometer	2299 reflections with $F^2 > 2\sigma(F^2)$
Detector resolution: 10.00 pixels mm^{-1}	$R_{int} = 0.025$
ω scans	$\theta_{max} = 27.5°$
Absorption correction: none	$h = -14\rightarrow15$
15048 measured reflections	$k = -11\rightarrow11$
3564 independent reflections	$l = -18\rightarrow18$

Refinement

Refinement on F^2	$w = 1/[0.0011F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.123$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.19$ e Å^{-3}
3564 reflections	Extinction correction: Larson (1970), equation 22
182 parameters	Extinction coefficient: 160 (35)
H-atom parameters constrained	

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (S) are based on F^2. *R*-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.81333 (10)	0.99331 (12)	0.68321 (9)	0.0752 (3)
O2	0.84308 (6)	0.45934 (11)	0.57521 (8)	0.0587 (3)
O3	0.67627 (8)	0.37808 (11)	0.59798 (9)	0.0669 (3)
O4	0.67440 (8)	0.60283 (11)	0.41615 (6)	0.0556 (2)
O5	0.76178 (9)	0.81440 (12)	0.46250 (8)	0.0694 (3)
C1	0.70689 (10)	0.64352 (13)	0.57863 (9)	0.0425 (3)
C2	0.78210 (11)	0.73547 (14)	0.64992 (10)	0.0497 (3)
C3	0.74691 (12)	0.89631 (16)	0.65636 (10)	0.0542 (4)

C4	0.63088 (12)	0.92857 (17)	0.63461 (11)	0.0596 (4)
C5	0.55626 (11)	0.82376 (16)	0.60862 (10)	0.0528 (4)
C6	0.58809 (10)	0.66294 (13)	0.59738 (10)	0.0489 (3)
C7	0.73776 (10)	0.47728 (14)	0.58521 (9)	0.0456 (3)
C8	0.88911 (12)	0.30794 (17)	0.58361 (12)	0.0648 (4)
C9	1.00390 (16)	0.3266 (2)	0.62810 (16)	0.1043 (7)
C10	0.88125 (19)	0.2399 (2)	0.48880 (17)	0.1116 (8)
C11	0.71947 (10)	0.69858 (14)	0.48006 (9)	0.0443 (3)
C12	0.67282 (12)	0.64383 (17)	0.31749 (10)	0.0546 (4)
C13	0.69216 (19)	0.5012 (2)	0.26693 (13)	0.1015 (7)
C14	0.56709 (14)	0.7170 (2)	0.28566 (12)	0.0875 (6)
C15	0.43618 (12)	0.8564 (2)	0.58951 (13)	0.0768 (5)
H4	0.6076	1.0273	0.6391	0.071*
H8	0.8479	0.2470	0.6240	0.078*
H12	0.7326	0.7142	0.3109	0.066*
H21	0.8556	0.7336	0.6318	0.060*
H22	0.7824	0.6894	0.7109	0.060*
H61	0.5408	0.6203	0.5454	0.059*
H62	0.5778	0.6093	0.6543	0.059*
H91	1.0468	0.3727	0.5845	0.125*
H92	1.0342	0.2301	0.6460	0.125*
H93	1.0047	0.3890	0.6825	0.125*
H101	0.8065	0.2419	0.4607	0.134*
H102	0.9257	0.2960	0.4508	0.134*
H103	0.9065	0.1379	0.4937	0.134*
H131	0.6329	0.4329	0.2722	0.122*
H132	0.6964	0.5227	0.2022	0.122*
H133	0.7596	0.4565	0.2940	0.122*
H141	0.5603	0.8071	0.3211	0.105*
H142	0.5082	0.6499	0.2946	0.105*
H143	0.5641	0.7417	0.2206	0.105*
H151	0.4104	0.8301	0.5261	0.092*
H152	0.3974	0.7987	0.6312	0.092*
H153	0.4237	0.9614	0.5990	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0967 (8)	0.0489 (6)	0.0753 (8)	−0.0152 (5)	−0.0089 (5)	−0.0077 (5)
O2	0.0551 (5)	0.0444 (5)	0.0785 (7)	0.0083 (4)	0.0159 (4)	0.0110 (4)
O3	0.0670 (6)	0.0405 (5)	0.0966 (9)	−0.0047 (4)	0.0245 (5)	−0.0005 (5)
O4	0.0761 (6)	0.0495 (5)	0.0398 (5)	−0.0127 (4)	0.0018 (4)	0.0014 (4)
O5	0.0998 (8)	0.0551 (6)	0.0553 (6)	−0.0250 (5)	0.0176 (5)	−0.0011 (5)
C1	0.0492 (6)	0.0361 (6)	0.0420 (7)	0.0012 (5)	0.0042 (5)	−0.0006 (5)
C2	0.0589 (7)	0.0423 (7)	0.0458 (8)	−0.0011 (5)	−0.0023 (5)	0.0001 (5)
C3	0.0772 (9)	0.0417 (7)	0.0423 (7)	−0.0039 (6)	0.0015 (6)	−0.0014 (5)
C4	0.0816 (9)	0.0409 (7)	0.0554 (9)	0.0117 (7)	0.0045 (7)	−0.0032 (6)
C5	0.0647 (8)	0.0499 (8)	0.0441 (7)	0.0105 (6)	0.0076 (5)	−0.0004 (6)

C6	0.0531 (7)	0.0451 (7)	0.0494 (7)	0.0003 (5)	0.0095 (5)	−0.0004 (5)
C7	0.0538 (7)	0.0408 (7)	0.0427 (7)	0.0006 (5)	0.0073 (5)	0.0008 (5)
C8	0.0699 (9)	0.0486 (8)	0.0783 (11)	0.0186 (7)	0.0192 (7)	0.0141 (7)
C9	0.0969 (13)	0.1152 (17)	0.0959 (16)	0.0267 (12)	−0.0085 (11)	0.0057 (13)
C10	0.1302 (17)	0.0905 (15)	0.1088 (18)	0.0378 (13)	−0.0072 (13)	−0.0317 (13)
C11	0.0492 (6)	0.0382 (6)	0.0455 (7)	0.0011 (5)	0.0063 (5)	−0.0007 (5)
C12	0.0659 (8)	0.0570 (8)	0.0404 (7)	−0.0069 (6)	0.0043 (5)	0.0037 (6)
C13	0.160 (2)	0.0858 (14)	0.0605 (12)	0.0185 (13)	0.0222 (12)	−0.0092 (10)
C14	0.0870 (11)	0.1104 (16)	0.0641 (11)	0.0197 (11)	0.0047 (8)	0.0177 (10)
C15	0.0692 (10)	0.0719 (11)	0.0881 (13)	0.0225 (8)	0.0040 (8)	−0.0023 (9)

Geometric parameters (Å, °)

O1—C3	1.2201 (18)	C2—H22	0.970
O2—C7	1.3303 (15)	C4—H4	0.930
O2—C8	1.4623 (17)	C6—H61	0.970
O3—C7	1.1918 (16)	C6—H62	0.970
O4—C11	1.3271 (15)	C8—H8	0.980
O4—C12	1.4657 (16)	C9—H91	0.960
O5—C11	1.1963 (17)	C9—H92	0.960
C1—C2	1.5347 (17)	C9—H93	0.960
C1—C6	1.5275 (18)	C10—H101	0.960
C1—C7	1.5291 (18)	C10—H102	0.960
C1—C11	1.5279 (18)	C10—H103	0.960
C2—C3	1.5030 (19)	C12—H12	0.980
C3—C4	1.453 (2)	C13—H131	0.960
C4—C5	1.331 (2)	C13—H132	0.960
C5—C6	1.4992 (19)	C13—H133	0.960
C5—C15	1.498 (2)	C14—H141	0.960
C8—C9	1.489 (2)	C14—H142	0.960
C8—C10	1.487 (2)	C14—H143	0.960
C12—C13	1.498 (2)	C15—H151	0.960
C12—C14	1.478 (2)	C15—H152	0.960
C2—H21	0.970	C15—H153	0.960
C7—O2—C8	118.32 (10)	C5—C6—H62	108.5
C11—O4—C12	117.96 (10)	H61—C6—H62	109.5
C2—C1—C6	109.53 (10)	O2—C8—H8	109.7
C2—C1—C7	110.49 (9)	C9—C8—H8	109.7
C2—C1—C11	109.33 (10)	C10—C8—H8	109.7
C6—C1—C7	109.54 (10)	C8—C9—H91	109.5
C6—C1—C11	109.47 (10)	C8—C9—H92	109.5
C7—C1—C11	108.46 (10)	C8—C9—H93	109.5
C1—C2—C3	113.28 (10)	H91—C9—H92	109.5
O1—C3—C2	120.53 (12)	H91—C9—H93	109.5
O1—C3—C4	122.12 (13)	H92—C9—H93	109.5
C2—C3—C4	117.26 (12)	C8—C10—H101	109.5
C3—C4—C5	123.26 (13)	C8—C10—H102	109.5
C4—C5—C6	121.36 (12)	C8—C10—H103	109.5
C4—C5—C15	123.17 (13)	H101—C10—H102	109.5

C6—C5—C15	115.47 (12)	H101—C10—H103	109.5
C1—C6—C5	113.27 (10)	H102—C10—H103	109.5
O2—C7—O3	124.89 (12)	O4—C12—H12	109.3
O2—C7—C1	110.34 (10)	C13—C12—H12	109.3
O3—C7—C1	124.77 (11)	C14—C12—H12	109.3
O2—C8—C9	105.70 (13)	C12—C13—H131	109.5
O2—C8—C10	108.54 (14)	C12—C13—H132	109.5
C9—C8—C10	113.29 (16)	C12—C13—H133	109.5
O4—C11—O5	124.33 (12)	H131—C13—H132	109.5
O4—C11—C1	111.00 (10)	H131—C13—H133	109.5
O5—C11—C1	124.64 (11)	H132—C13—H133	109.5
O4—C12—C13	106.01 (12)	C12—C14—H141	109.5
O4—C12—C14	108.67 (12)	C12—C14—H142	109.5
C13—C12—C14	114.10 (14)	C12—C14—H143	109.5
C1—C2—H21	108.5	H141—C14—H142	109.5
C1—C2—H22	108.5	H141—C14—H143	109.5
C3—C2—H21	108.5	H142—C14—H143	109.5
C3—C2—H22	108.5	C5—C15—H151	109.5
H21—C2—H22	109.5	C5—C15—H152	109.5
C3—C4—H4	118.4	C5—C15—H153	109.5
C5—C4—H4	118.4	H151—C15—H152	109.5
C1—C6—H61	108.5	H151—C15—H153	109.5
C1—C6—H62	108.5	H152—C15—H153	109.5
C5—C6—H61	108.5		
C7—O2—C8—C9	−145.40 (14)	C6—C1—C7—O3	3.75 (18)
C7—O2—C8—C10	92.77 (16)	C7—C1—C6—C5	170.15 (10)
C8—O2—C7—O3	−2.3 (2)	C6—C1—C11—O4	−73.28 (13)
C8—O2—C7—C1	177.34 (11)	C6—C1—C11—O5	104.93 (14)
C11—O4—C12—C13	142.78 (13)	C11—C1—C6—C5	−71.04 (13)
C11—O4—C12—C14	−94.17 (15)	C7—C1—C11—O4	46.20 (13)
C12—O4—C11—O5	−2.24 (19)	C7—C1—C11—O5	−135.59 (13)
C12—O4—C11—C1	175.98 (10)	C11—C1—C7—O2	64.69 (12)
C2—C1—C6—C5	48.81 (14)	C11—C1—C7—O3	−115.68 (14)
C6—C1—C2—C3	−51.44 (15)	C1—C2—C3—O1	−154.97 (13)
C2—C1—C7—O2	−55.13 (14)	C1—C2—C3—C4	28.45 (18)
C2—C1—C7—O3	124.50 (14)	O1—C3—C4—C5	−177.37 (15)
C7—C1—C2—C3	−172.20 (11)	C2—C3—C4—C5	−0.8 (2)
C2—C1—C11—O4	166.74 (10)	C3—C4—C5—C6	−1.7 (2)
C2—C1—C11—O5	−15.05 (17)	C3—C4—C5—C15	177.76 (15)
C11—C1—C2—C3	68.51 (14)	C4—C5—C6—C1	−23.65 (19)
C6—C1—C7—O2	−175.88 (10)	C15—C5—C6—C1	156.86 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.98	2.47	3.333 (2)	147
C12—H12···O5	0.98	2.35	2.707 (2)	101

Symmetry codes: (i) *x*, *y*−1, *z*.

Fig. 1