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Ethyl dicyclohexylglycolate

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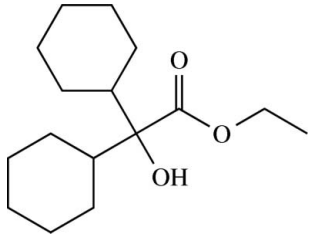
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.063; wR factor = 0.147; data-to-parameter ratio = 20.7.

The title compound, $C_{16}H_{28}O_3$, was prepared as an intermediate in the synthesis of dicyclohexylglycolic acid. The cyclohexyl rings adopt a chair conformation. Intramolecular hydrogen bonds are present in the crystal structure.

Related literature

The title compound was prepared according to a published procedure (Gauerke & Marvel, 1928).



Experimental

Crystal data

$C_{16}H_{28}O_3$
M_r = 268.39
Monoclinic, $P2_1/c$
a = 8.873 (2) Å
b = 23.121 (3) Å
c = 8.4300 (14) Å
β = 116.475 (15)°
V = 1548.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 200 (2) K
0.35 × 0.35 × 0.11 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
9044 measured reflections
3563 independent reflections
2649 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.147
S = 1.09
3563 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 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
O12—H12···O111	0.84	2.12	2.6357 (19)	119

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Sandra Albrecht and Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2457).

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Gauerke, C. G. & Marvel, C. S. (1928). *J. Am. Chem. Soc.* **50**, 1178–1182.
Oxford Diffraction (2005). *CrysAlis CCD* and *CrysAlis RED*. Version 1.171.27p5 beta. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

  doi:10.1107/S1600536807041554

supplementary materials

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Ethyl dicyclohexylglycolate

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Comment

The title compound, $C_{16}H_{28}O_3$, was prepared as an intermediate in the synthesis of dicyclohexylglycolic acid. It was obtained upon reaction of cyclohexyl magnesium bromide with diethyl oxalate.

The cyclohexyl rings invariably adopt chair conformations. Intramolecular hydrogen bonds between the O-bonded H atom and the double-bonded O atom are present in the crystal structure.

The molecular structure (Fig. 1) shows two cyclohexyl rings, a hydroxy group and a carboxy group attached to the central C atom.

The molecular packing is shown in Figure 2.

Experimental

The title compound was prepared according to a published procedure (Gauerke & Marvel, 1928) upon reaction of cyclohexyl magnesium bromide with diethyl oxalate. Crystals suitable for X-ray analysis were directly obtained from the crystallized reaction product.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. H atoms bonded to C atoms were positioned geometrically and allowed to ride on their parent atoms at distances of C—H = 0.98 Å with U_{iso}(H) = 1.5Ueq(C) for methyl H, C—H = 0.99 Å with U_{iso}(H) = 1.2Ueq(C) for methylene H, and C—H = 1.00 Å with U_{iso}(H) = 1.2Ueq(C) for methine H. The H atom bonded to the O atom was positioned geometrically and allowed to ride on its parent atom at a distance of O—H = 0.84 Å with U_{iso}(H) = 1.5Ueq(O).

Figures

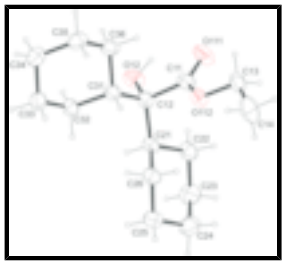

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

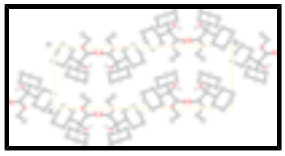

Fig. 2. The packing of (I), viewed along [0 0 $\bar{1}$]. H atoms omitted for clarity except O-bonded H atoms.

Ethyl dicyclohexylglycolate

Crystal data

$C_{16}H_{28}O_3$	$Z = 4$
$M_r = 268.39$	$F_{000} = 592$
Monoclinic, $P2_1/c$	$D_x = 1.152$ Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.873$ (2) Å	$\theta = 3.8$–27.5º
$b = 23.121$ (3) Å	$\mu = 0.08$ mm^{-1}
$c = 8.4300$ (14) Å	$T = 200$ (2) K
$\beta = 116.475$ (15)º	Platelet, colourless
$V = 1548.1$ (5) Å^3	0.35 × 0.35 × 0.11 mm

Data collection

Nonius KappaCCD diffractometer	2649 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.040$
Monochromator: graphite	$\theta_{max} = 27.5$º
$T = 200$(2) K	$\theta_{min} = 3.8$º
ω scans	$h = -6 \rightarrow 11$
Absorption correction: none	$k = -27 \rightarrow 30$
9044 measured reflections	$l = -10 \rightarrow 9$
3563 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.063$	H-atom parameters constrained
$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
3563 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O12	0.35070 (15)	0.17755 (5)	0.07234 (17)	0.0391 (3)
H12	0.3048	0.2097	0.0338	0.059*
O111	0.09969 (18)	0.23581 (6)	0.0822 (2)	0.0499 (4)
O112	0.01746 (14)	0.16183 (5)	0.19875 (17)	0.0349 (3)
C11	0.1170 (2)	0.18694 (7)	0.1387 (2)	0.0332 (4)
C12	0.2577 (2)	0.14687 (7)	0.1467 (2)	0.0295 (4)
C13	−0.1205 (2)	0.19669 (8)	0.1969 (3)	0.0382 (5)
H131	−0.2067	0.2032	0.0735	0.046*
H132	−0.0780	0.2347	0.2530	0.046*
C14	−0.1940 (3)	0.16416 (10)	0.2978 (4)	0.0573 (6)
H141	−0.2931	0.1848	0.2913	0.086*
H142	−0.1102	0.1608	0.4219	0.086*
H143	−0.2273	0.1255	0.2466	0.086*
C21	0.3843 (2)	0.13388 (7)	0.3428 (2)	0.0285 (4)
H21	0.4891	0.1192	0.3401	0.034*
C22	0.4345 (2)	0.18909 (7)	0.4551 (2)	0.0355 (4)
H221	0.4702	0.2188	0.3943	0.043*
H222	0.3357	0.2043	0.4670	0.043*
C23	0.5776 (2)	0.17780 (8)	0.6391 (3)	0.0446 (5)
H231	0.6801	0.1665	0.6278	0.053*
H232	0.6028	0.2138	0.7100	0.053*
C24	0.5322 (3)	0.13022 (9)	0.7343 (3)	0.0475 (5)
H241	0.4406	0.1439	0.7616	0.057*
H242	0.6312	0.1213	0.8477	0.057*
C25	0.4757 (2)	0.07563 (8)	0.6223 (2)	0.0377 (4)
H251	0.4384	0.0467	0.6840	0.045*
H252	0.5723	0.0591	0.6090	0.045*
C26	0.3317 (2)	0.08747 (7)	0.4385 (2)	0.0320 (4)
H261	0.2312	0.1007	0.4504	0.038*
H262	0.3020	0.0514	0.3680	0.038*
C31	0.1795 (2)	0.09223 (7)	0.0338 (2)	0.0286 (4)
H31	0.1194	0.0703	0.0907	0.034*
C32	0.3153 (2)	0.05253 (7)	0.0273 (2)	0.0327 (4)

H321	0.3784	0.0738	−0.0259	0.039*
H322	0.3957	0.0415	0.1495	0.039*
C33	0.2383 (2)	−0.00201 (8)	−0.0810 (2)	0.0375 (4)
H331	0.3285	−0.0255	−0.0879	0.045*
H332	0.1860	−0.0254	−0.0204	0.045*
C34	0.1058 (2)	0.01237 (9)	−0.2680 (2)	0.0415 (5)
H341	0.1609	0.0307	−0.3347	0.050*
H342	0.0514	−0.0238	−0.3301	0.050*
C35	−0.0276 (2)	0.05302 (8)	−0.2645 (2)	0.0391 (5)
H351	−0.0936	0.0326	−0.2131	0.047*
H352	−0.1058	0.0643	−0.3873	0.047*
C36	0.0509 (2)	0.10733 (8)	−0.1559 (2)	0.0365 (4)
H361	−0.0389	0.1318	−0.1520	0.044*
H362	0.1073	0.1298	−0.2141	0.044*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O12	0.0400 (7)	0.0345 (7)	0.0466 (8)	−0.0015 (5)	0.0229 (6)	0.0110 (6)
O111	0.0523 (9)	0.0335 (7)	0.0649 (10)	0.0134 (6)	0.0270 (8)	0.0167 (7)
O112	0.0307 (6)	0.0312 (6)	0.0443 (7)	0.0069 (5)	0.0181 (6)	0.0031 (5)
C11	0.0316 (9)	0.0288 (9)	0.0340 (9)	0.0035 (7)	0.0099 (8)	0.0027 (7)
C12	0.0273 (9)	0.0284 (8)	0.0339 (9)	0.0010 (6)	0.0147 (7)	0.0057 (7)
C13	0.0291 (9)	0.0373 (10)	0.0437 (11)	0.0085 (7)	0.0121 (8)	−0.0043 (8)
C14	0.0485 (13)	0.0487 (13)	0.0897 (18)	0.0056 (10)	0.0443 (13)	0.0014 (12)
C21	0.0239 (8)	0.0253 (8)	0.0336 (9)	0.0019 (6)	0.0105 (7)	0.0026 (7)
C22	0.0363 (10)	0.0265 (9)	0.0409 (10)	−0.0024 (7)	0.0145 (8)	−0.0006 (7)
C23	0.0422 (11)	0.0403 (11)	0.0425 (11)	−0.0084 (8)	0.0110 (9)	−0.0069 (9)
C24	0.0498 (12)	0.0508 (12)	0.0340 (10)	−0.0027 (9)	0.0116 (9)	−0.0019 (9)
C25	0.0346 (10)	0.0385 (10)	0.0365 (10)	0.0039 (8)	0.0128 (8)	0.0080 (8)
C26	0.0299 (9)	0.0290 (9)	0.0345 (9)	−0.0016 (7)	0.0121 (8)	0.0030 (7)
C31	0.0259 (8)	0.0309 (9)	0.0286 (9)	0.0025 (6)	0.0119 (7)	0.0030 (7)
C32	0.0275 (9)	0.0373 (10)	0.0320 (9)	0.0052 (7)	0.0121 (7)	0.0024 (7)
C33	0.0361 (10)	0.0394 (10)	0.0386 (10)	0.0044 (8)	0.0181 (8)	−0.0027 (8)
C34	0.0411 (11)	0.0502 (11)	0.0339 (10)	−0.0021 (8)	0.0173 (9)	−0.0044 (8)
C35	0.0326 (10)	0.0500 (11)	0.0299 (9)	−0.0016 (8)	0.0097 (8)	0.0006 (8)
C36	0.0291 (9)	0.0427 (10)	0.0327 (9)	0.0060 (7)	0.0093 (8)	0.0065 (8)

Geometric parameters (Å, °)

O12—C12	1.428 (2)	C24—H241	0.9900
O12—H12	0.8400	C24—H242	0.9900
O111—C11	1.209 (2)	C25—C26	1.531 (2)
O112—C11	1.332 (2)	C25—H251	0.9900
O112—C13	1.460 (2)	C25—H252	0.9900
C11—C12	1.531 (2)	C26—H261	0.9900
C12—C31	1.548 (2)	C26—H262	0.9900
C12—C21	1.558 (2)	C31—C36	1.534 (2)
C13—C14	1.486 (3)	C31—C32	1.535 (2)

C13—H131	0.9900	C31—H31	1.0000
C13—H132	0.9900	C32—C33	1.528 (2)
C14—H141	0.9800	C32—H321	0.9900
C14—H142	0.9800	C32—H322	0.9900
C14—H143	0.9800	C33—C34	1.525 (3)
C21—C22	1.532 (2)	C33—H331	0.9900
C21—C26	1.535 (2)	C33—H332	0.9900
C21—H21	1.0000	C34—C35	1.522 (3)
C22—C23	1.526 (3)	C34—H341	0.9900
C22—H221	0.9900	C34—H342	0.9900
C22—H222	0.9900	C35—C36	1.527 (3)
C23—C24	1.518 (3)	C35—H351	0.9900
C23—H231	0.9900	C35—H352	0.9900
C23—H232	0.9900	C36—H361	0.9900
C24—C25	1.521 (3)	C36—H362	0.9900
C12—O12—H12	109.5	C24—C25—H251	109.2
C11—O112—C13	116.77 (14)	C26—C25—H251	109.2
O111—C11—O112	124.58 (17)	C24—C25—H252	109.2
O111—C11—C12	122.76 (17)	C26—C25—H252	109.2
O112—C11—C12	112.65 (14)	H251—C25—H252	107.9
O12—C12—C11	107.41 (13)	C25—C26—C21	110.19 (14)
O12—C12—C31	108.86 (14)	C25—C26—H261	109.6
C11—C12—C31	109.50 (13)	C21—C26—H261	109.6
O12—C12—C21	106.08 (13)	C25—C26—H262	109.6
C11—C12—C21	110.59 (14)	C21—C26—H262	109.6
C31—C12—C21	114.13 (13)	H261—C26—H262	108.1
O112—C13—C14	107.11 (15)	C36—C31—C32	109.26 (14)
O112—C13—H131	110.3	C36—C31—C12	112.13 (14)
C14—C13—H131	110.3	C32—C31—C12	111.50 (13)
O112—C13—H132	110.3	C36—C31—H31	107.9
C14—C13—H132	110.3	C32—C31—H31	107.9
H131—C13—H132	108.5	C12—C31—H31	107.9
C13—C14—H141	109.5	C33—C32—C31	111.48 (14)
C13—C14—H142	109.5	C33—C32—H321	109.3
H141—C14—H142	109.5	C31—C32—H321	109.3
C13—C14—H143	109.5	C33—C32—H322	109.3
H141—C14—H143	109.5	C31—C32—H322	109.3
H142—C14—H143	109.5	H321—C32—H322	108.0
C22—C21—C26	109.31 (15)	C34—C33—C32	111.78 (15)
C22—C21—C12	111.60 (13)	C34—C33—H331	109.3
C26—C21—C12	116.61 (13)	C32—C33—H331	109.3
C22—C21—H21	106.2	C34—C33—H332	109.3
C26—C21—H21	106.2	C32—C33—H332	109.3
C12—C21—H21	106.2	H331—C33—H332	107.9
C23—C22—C21	111.49 (14)	C35—C34—C33	111.27 (15)
C23—C22—H221	109.3	C35—C34—H341	109.4
C21—C22—H221	109.3	C33—C34—H341	109.4
C23—C22—H222	109.3	C35—C34—H342	109.4
C21—C22—H222	109.3	C33—C34—H342	109.4

H221—C22—H222	108.0	H341—C34—H342	108.0
C24—C23—C22	111.36 (15)	C34—C35—C36	111.61 (15)
C24—C23—H231	109.4	C34—C35—H351	109.3
C22—C23—H231	109.4	C36—C35—H351	109.3
C24—C23—H232	109.4	C34—C35—H352	109.3
C22—C23—H232	109.4	C36—C35—H352	109.3
H231—C23—H232	108.0	H351—C35—H352	108.0
C23—C24—C25	111.41 (17)	C35—C36—C31	111.50 (15)
C23—C24—H241	109.3	C35—C36—H361	109.3
C25—C24—H241	109.3	C31—C36—H361	109.3
C23—C24—H242	109.3	C35—C36—H362	109.3
C25—C24—H242	109.3	C31—C36—H362	109.3
H241—C24—H242	108.0	H361—C36—H362	108.0
C24—C25—C26	112.03 (15)		
C13—O112—C11—O111	−0.7 (3)	C23—C24—C25—C26	−54.3 (2)
C13—O112—C11—C12	179.84 (14)	C24—C25—C26—C21	56.8 (2)
O111—C11—C12—O12	−1.9 (2)	C22—C21—C26—C25	−57.80 (18)
O112—C11—C12—O12	177.55 (13)	C12—C21—C26—C25	174.47 (14)
O111—C11—C12—C31	−120.01 (19)	O12—C12—C31—C36	−63.89 (17)
O112—C11—C12—C31	59.49 (19)	C11—C12—C31—C36	53.26 (19)
O111—C11—C12—C21	113.39 (19)	C21—C12—C31—C36	177.83 (14)
O112—C11—C12—C21	−67.11 (18)	O12—C12—C31—C32	58.99 (17)
C11—O112—C13—C14	−170.11 (16)	C11—C12—C31—C32	176.15 (14)
O12—C12—C21—C22	69.68 (17)	C21—C12—C31—C32	−59.29 (19)
C11—C12—C21—C22	−46.49 (19)	C36—C31—C32—C33	−56.70 (19)
C31—C12—C21—C22	−170.47 (14)	C12—C31—C32—C33	178.79 (14)
O12—C12—C21—C26	−163.72 (14)	C31—C32—C33—C34	55.8 (2)
C11—C12—C21—C26	80.12 (18)	C32—C33—C34—C35	−53.8 (2)
C31—C12—C21—C26	−43.9 (2)	C33—C34—C35—C36	54.0 (2)
C26—C21—C22—C23	58.06 (19)	C34—C35—C36—C31	−56.4 (2)
C12—C21—C22—C23	−171.45 (15)	C32—C31—C36—C35	57.01 (19)
C21—C22—C23—C24	−56.1 (2)	C12—C31—C36—C35	−178.85 (15)
C22—C23—C24—C25	53.5 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O111	0.84	2.12	2.6357 (19)	119

Fig. 1

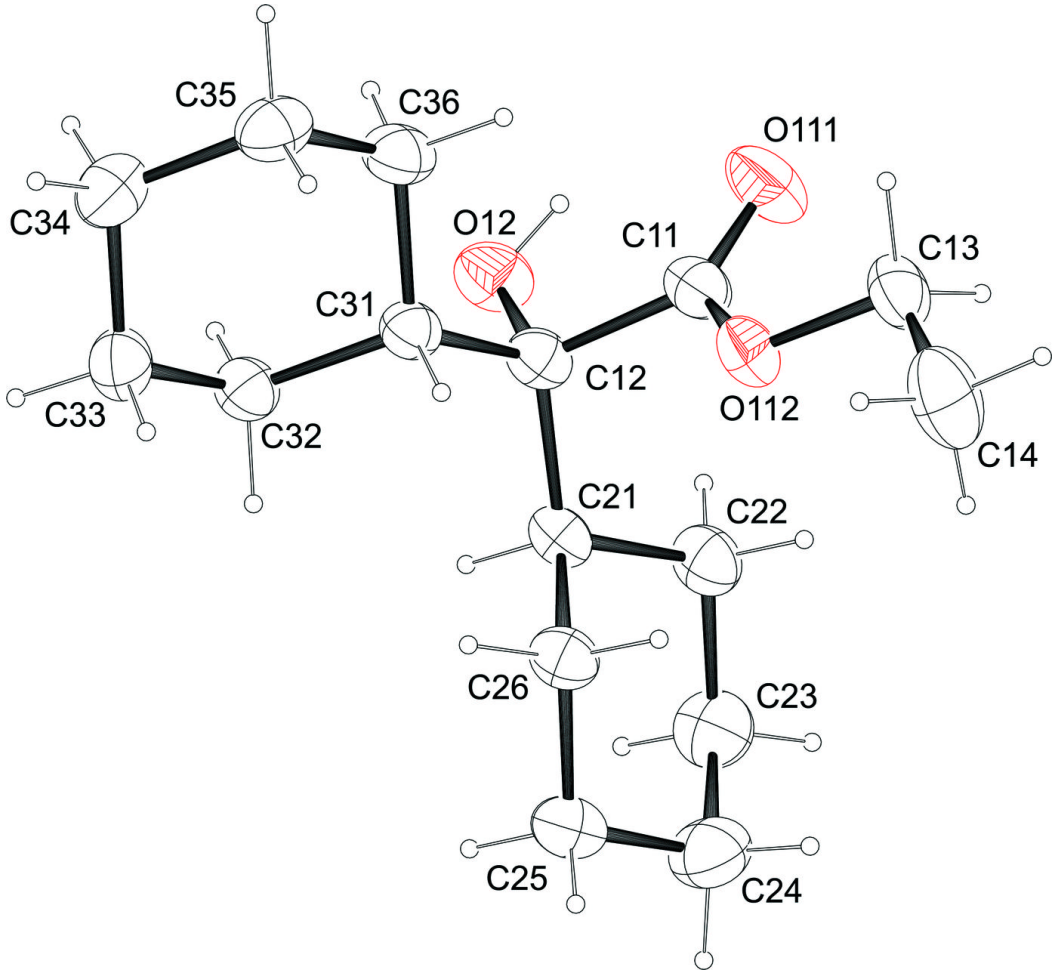
C35
C36
O111
C34
O12
C11
C13
C31
C12
O112
C33
C32
C14
C21
C22
C26
C23
C25
C24

Fig. 2