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3,3′-(2-Oxocyclopentane-1,3-diyl)-dipropanoic acid

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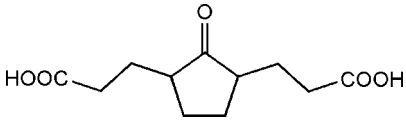
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.126; data-to-parameter ratio = 13.8.

In the title compound, $C_{11}H_{16}O_5$, intermolecular O—H···O hydrogen bonds are observed which help to establish the crystal packing.

Related literature

For the preparation of the title compound, see Westman & Kober (1964); for a related compound, see Lalancette *et al.* (1995).



Experimental

Crystal data

$C_{11}H_{16}O_5$
M_r = 228.24
Triclinic, $P\overline{1}$
a = 6.7644 (17) Å
b = 9.386 (2) Å
c = 9.855 (3) Å
α = 95.510 (4)°
β = 101.915 (4)°
γ = 104.563 (4)°
V = 585.3 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.26 × 0.22 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.974, T_{max} = 0.990
3011 measured reflections
2035 independent reflections
1452 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.126
S = 1.03
2035 reflections
147 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 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
O1—H1···O2^{i}	0.82	1.82	2.639 (2)	173
O5—H5···O4^{ii}	0.82	1.85	2.667 (2)	175

Symmetry codes: (i) $-x+2, -y+2, -z-1$; (ii) $-x, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2292).

References

Bruker (1997). *SMART* (Version 5.611), *SAINT* (Version 6.0), *SADABS* (Version 2.03) and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Lalancette, R. A., Thompson, H. W. & Coté, M. L. (1995). *Acta Cryst.* C**51**, 2617–2619.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Westman, T. L. & Kober, A. E. (1964). *J. Org. Chem.* **29**, 2448–2450.

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supplementary materials

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3,3'-(2-Oxocyclopentane-1,3-diyl)dipropanoic acid

Y. Chen, J. Yang, Y. Deng, G. Li and W. Wang

Comment

The title compound (I) is obtained as a by-product in the synthesis of 6,7-dihydro-5*H*-cyclopenta[*b*]pyridine. We report here the crystal structure of the title compound (I) (Fig. 1). In the crystal, O—H···O hydrogen bonds link the molecules into chains. Table 1. The structure of the related cyclohexyl compound has been reported previously by Lalancette *et al.* (1995).

Experimental

The title compound was prepared according to the method of Westman & Kober (1964). Methyl 3-(2-cyanoethyl)-1-(3-methoxy-3-oxopropyl) −2-oxocyclopentanecarboxylate (14.06 g, 0.05 mol) was refluxed for 3 h, with 27 ml of concentrated hydrochloric acid. At the end of this period, the solution was evaporated to dryness *in vacuo* (steam bath), and the solid residue was triturated with 50 ml e thanol. Removal of ammonium chloride by filtration and evaporation of the ethanol yielded 80% of 3,3'-(2-oxocyclopentane-1,3-diyl)dipropanoic acid, after two recrystallizations from dioxane-hexane. Crystals of (I) were obtained by slow evaporation of a solution of ethyl acetate (m.p. 394–395 K).

Refinement

All H atoms were positioned geometrically (C—H = 0.97–0.98 Å), and refined as riding with U_{iso}(H) = 1.2U_{eq} (carrier) or 1.5$_{eq}$ (methyl groups).

Figures

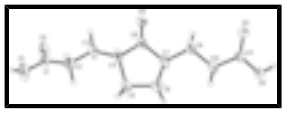

Fig. 1. A view of the molecular of (I). Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

3,3'-(2-Oxocyclopentane-1,3-diyl)dipropanoic acid

Crystal data

$C_{11}H_{16}O_5$	$Z = 2$
$M_r = 228.24$	$F_{000} = 244$
Triclinic, $P\bar{1}$	$D_x = 1.295$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 394-395 K K
$a = 6.7644$ (17) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.386$ (2) Å	Cell parameters from 1257 reflections
$c = 9.855$ (3) Å	$\theta = 2.9$–25.7°

α = 95.510 (4)º	μ = 0.10 mm^{-1}
β = 101.915 (4)º	T = 294 (2) K
γ = 104.563 (4)º	Block, colorless
V = 585.3 (3) Å^3	0.26 × 0.22 × 0.10 mm

Data collection

Bruker SMART CCD area detector diffractometer	2035 independent reflections
Radiation source: fine-focus sealed tube	1452 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.015
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -7 \rightarrow 8$
T_{min} = 0.974, T_{max} = 0.990	$k = -11 \rightarrow 8$
3011 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.1943P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2035 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
147 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
12 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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}

O1	0.9983 (4)	0.8289 (2)	−0.4371 (2)	0.0967 (7)
H1	1.0406	0.8853	−0.4900	0.145*
O2	0.8445 (3)	1.0024 (2)	−0.3900 (2)	0.0815 (6)
O3	0.2993 (2)	0.8393 (2)	−0.05699 (17)	0.0666 (5)
O4	0.0373 (2)	0.55634 (18)	0.34961 (16)	0.0602 (5)
O5	0.2803 (2)	0.5835 (2)	0.54718 (16)	0.0687 (5)
H5	0.1779	0.5422	0.5753	0.103*
C1	0.8831 (4)	0.8854 (3)	−0.3694 (2)	0.0555 (6)
C2	0.8024 (4)	0.7925 (3)	−0.2688 (2)	0.0574 (6)
H2A	0.9215	0.7819	−0.2005	0.069*
H2B	0.7245	0.6939	−0.3195	0.069*
C3	0.6618 (3)	0.8517 (3)	−0.1907 (2)	0.0506 (5)
H3A	0.7327	0.9540	−0.1470	0.061*
H3B	0.5334	0.8510	−0.2567	0.061*
C4	0.6072 (3)	0.7591 (2)	−0.0791 (2)	0.0483 (5)
H4	0.5445	0.6558	−0.1261	0.058*
C5	0.7912 (4)	0.7589 (3)	0.0396 (3)	0.0666 (7)
H5A	0.8891	0.8577	0.0683	0.080*
H5B	0.8657	0.6899	0.0111	0.080*
C6	0.6900 (4)	0.7096 (3)	0.1585 (3)	0.0632 (7)
H6A	0.6353	0.6019	0.1449	0.076*
H6B	0.7910	0.7425	0.2485	0.076*
C7	0.5122 (3)	0.7837 (2)	0.1506 (2)	0.0477 (5)
H7	0.5717	0.8841	0.2050	0.057*
C8	0.4500 (3)	0.7998 (2)	−0.0036 (2)	0.0460 (5)
C9	0.3272 (3)	0.7044 (3)	0.2067 (2)	0.0519 (6)
H9A	0.2219	0.7588	0.1941	0.062*
H9B	0.2638	0.6054	0.1526	0.062*
C10	0.3917 (3)	0.6911 (3)	0.3610 (2)	0.0556 (6)
H10A	0.5054	0.6444	0.3741	0.067*
H10B	0.4453	0.7905	0.4152	0.067*
C11	0.2184 (3)	0.6037 (2)	0.4173 (2)	0.0481 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1374 (19)	0.1006 (16)	0.1023 (16)	0.0674 (14)	0.0866 (15)	0.0393 (12)
O2	0.1068 (15)	0.0789 (13)	0.0915 (14)	0.0421 (12)	0.0680 (12)	0.0294 (11)
O3	0.0482 (9)	0.1018 (14)	0.0617 (10)	0.0314 (9)	0.0175 (8)	0.0319 (10)
O4	0.0482 (9)	0.0817 (12)	0.0521 (9)	0.0130 (8)	0.0148 (7)	0.0254 (8)
O5	0.0500 (9)	0.1064 (14)	0.0519 (10)	0.0147 (9)	0.0160 (8)	0.0334 (9)
C1	0.0558 (14)	0.0672 (17)	0.0470 (13)	0.0191 (12)	0.0204 (11)	0.0027 (12)
C2	0.0601 (14)	0.0632 (15)	0.0541 (14)	0.0203 (12)	0.0212 (11)	0.0091 (12)
C3	0.0497 (12)	0.0577 (14)	0.0473 (12)	0.0143 (10)	0.0182 (10)	0.0098 (11)
C4	0.0425 (11)	0.0489 (13)	0.0541 (13)	0.0099 (9)	0.0152 (10)	0.0105 (10)
C5	0.0489 (13)	0.096 (2)	0.0675 (16)	0.0296 (13)	0.0224 (12)	0.0326 (15)
C6	0.0507 (13)	0.0838 (18)	0.0655 (15)	0.0246 (12)	0.0194 (11)	0.0334 (14)
C7	0.0441 (12)	0.0530 (13)	0.0471 (12)	0.0102 (9)	0.0136 (9)	0.0161 (10)

C8	0.0377 (11)	0.0501 (13)	0.0497 (12)	0.0074 (9)	0.0125 (9)	0.0144 (10)
C9	0.0442 (12)	0.0636 (15)	0.0506 (13)	0.0122 (10)	0.0155 (10)	0.0206 (11)
C10	0.0490 (12)	0.0684 (16)	0.0533 (13)	0.0152 (11)	0.0171 (10)	0.0208 (12)
C11	0.0484 (13)	0.0565 (14)	0.0472 (12)	0.0212 (10)	0.0168 (10)	0.0164 (11)

Geometric parameters (Å, °)

O1—C1	1.299 (3)	C4—H4	0.9800
O1—H1	0.8200	C5—C6	1.527 (3)
O2—C1	1.217 (3)	C5—H5A	0.9700
O3—C8	1.207 (2)	C5—H5B	0.9700
O4—C11	1.215 (3)	C6—C7	1.526 (3)
O5—C11	1.309 (2)	C6—H6A	0.9700
O5—H5	0.8200	C6—H6B	0.9700
C1—C2	1.480 (3)	C7—C8	1.524 (3)
C2—C3	1.514 (3)	C7—C9	1.524 (3)
C2—H2A	0.9700	C7—H7	0.9800
C2—H2B	0.9700	C9—C10	1.519 (3)
C3—C4	1.514 (3)	C9—H9A	0.9700
C3—H3A	0.9700	C9—H9B	0.9700
C3—H3B	0.9700	C10—C11	1.493 (3)
C4—C8	1.519 (3)	C10—H10A	0.9700
C4—C5	1.523 (3)	C10—H10B	0.9700
C1—O1—H1	109.5	C7—C6—H6A	110.8
C11—O5—H5	109.5	C5—C6—H6A	110.8
O2—C1—O1	122.3 (2)	C7—C6—H6B	110.8
O2—C1—C2	124.2 (2)	C5—C6—H6B	110.8
O1—C1—C2	113.5 (2)	H6A—C6—H6B	108.9
C1—C2—C3	115.3 (2)	C8—C7—C9	113.28 (17)
C1—C2—H2A	108.4	C8—C7—C6	104.12 (17)
C3—C2—H2A	108.4	C9—C7—C6	115.81 (18)
C1—C2—H2B	108.4	C8—C7—H7	107.8
C3—C2—H2B	108.4	C9—C7—H7	107.8
H2A—C2—H2B	107.5	C6—C7—H7	107.8
C4—C3—C2	111.47 (19)	O3—C8—C4	125.5 (2)
C4—C3—H3A	109.3	O3—C8—C7	125.13 (19)
C2—C3—H3A	109.3	C4—C8—C7	109.38 (17)
C4—C3—H3B	109.3	C10—C9—C7	112.54 (18)
C2—C3—H3B	109.3	C10—C9—H9A	109.1
H3A—C3—H3B	108.0	C7—C9—H9A	109.1
C3—C4—C8	115.83 (18)	C10—C9—H9B	109.1
C3—C4—C5	115.83 (18)	C7—C9—H9B	109.1
C8—C4—C5	103.80 (18)	H9A—C9—H9B	107.8
C3—C4—H4	106.9	C11—C10—C9	114.30 (19)
C8—C4—H4	106.9	C11—C10—H10A	108.7
C5—C4—H4	106.9	C9—C10—H10A	108.7
C4—C5—C6	104.44 (18)	C11—C10—H10B	108.7
C4—C5—H5A	110.9	C9—C10—H10B	108.7
C6—C5—H5A	110.9	H10A—C10—H10B	107.6

C4—C5—H5B	110.9	O4—C11—O5	123.20 (19)
C6—C5—H5B	110.9	O4—C11—C10	123.33 (19)
H5A—C5—H5B	108.9	O5—C11—C10	113.47 (19)
C7—C6—C5	104.61 (17)		
O2—C1—C2—C3	0.7 (4)	C3—C4—C8—C7	−142.55 (19)
O1—C1—C2—C3	−178.2 (2)	C5—C4—C8—C7	−14.4 (2)
C1—C2—C3—C4	−173.6 (2)	C9—C7—C8—O3	45.1 (3)
C2—C3—C4—C8	−174.42 (19)	C6—C7—C8—O3	171.7 (2)
C2—C3—C4—C5	63.7 (3)	C9—C7—C8—C4	−135.13 (19)
C3—C4—C5—C6	159.9 (2)	C6—C7—C8—C4	−8.5 (2)
C8—C4—C5—C6	31.8 (2)	C8—C7—C9—C10	−179.26 (19)
C4—C5—C6—C7	−37.8 (3)	C6—C7—C9—C10	60.5 (3)
C5—C6—C7—C8	28.1 (2)	C7—C9—C10—C11	−175.48 (19)
C5—C6—C7—C9	153.2 (2)	C9—C10—C11—O4	−5.3 (3)
C3—C4—C8—O3	37.2 (3)	C9—C10—C11—O5	175.0 (2)
C5—C4—C8—O3	165.4 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.82	2.639 (2)	173
O5—H5···O4^{ii}	0.82	1.85	2.667 (2)	175

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*−1; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1