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cis-8-Methylbicyclo[4.2.0]octa-1,3,5-triene-7-carboxylic Acid at 293K

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Abstract

The molecules of the title compound are associated as dimers by hydrogen bonds H(1)—O(2)'. Additional characteristic atom distances of the dimer: O(1)—O(2)' 2.650 (3) (intermolecular), H(1)—O(2)' 1.74 (intermolecular), O(1)—H(1) 0.91, C(10)—O(2) 1.225 (4) and C(10)—O(1) 1.293 (3) Å.The C(10)—O(2) bond distance is slightly longer than the standard distance range for such dimers of 1.205 to 1.215 Å whereas the C(10)—O(1) bond distance is slightly shorter than the standard distance of 1.308 Å.

Experimental

Synthesis of the title compound was carried out by irradiation of 2-diazo-3-methyl-1-indanone·The *cis*-isomere was isolated from the yielded a *cis*/*trans* mixture by recrystallization in petrol ether.

Computing details

Data collection: P3 VMS V.4.1 (Siemens 1987); cell refinement: P3 VMS V.4.1 (Siemens 1987); data reduction: *XDISK* V.4.20.2 PC (Siemens 1991); program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1984); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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Crystal data

$C_{10}H_{10}O_2$	$V = 864.7\ (3)$ Å^3
$M_r = 162.18$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$
$a = 8.8787\ (16)$ Å	$\mu = 0.09$ mm^{-1}
$b = 7.9846\ (16)$ Å	$T = 293\ (2)$ K
$c = 12.395\ (3)$ Å	0.24 × 0.21 × 0.12 mm
$\beta = 100.243\ (15)^o$	

Data collection

Siemens P4 four-circle diffractometer	$R_{int} = 0.022$
Absorption correction: none	2 standard reflections
2446 measured reflections	every 100 reflections
2014 independent reflections	intensity decay: <3%

CIF access

1209 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.074$	109 parameters
$wR(F^2) = 0.206$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.07$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
2014 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Acknowledgements

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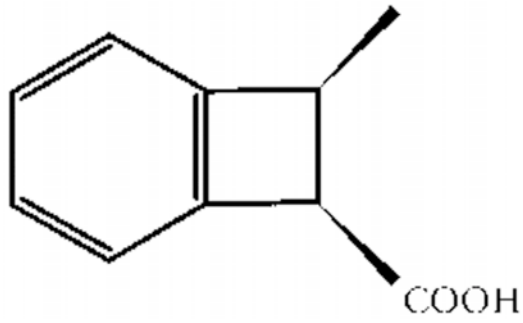
Scheme 1



supplementary materials

cis-8-Methylbicyclo[4.2.0]octa-1,3,5-triene-7-carboxylic acid

Crystal data

$C_{10}H_{10}O_2$	F_{000} = 344
M_r = 162.18	D_x = 1.246 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.8787 (16) Å	Cell parameters from 33 reflections
b = 7.9846 (16) Å	θ = 10–12.5º
c = 12.395 (3) Å	μ = 0.09 mm^{-1}
β = 100.243 (15)º	T = 293 (2) K
V = 864.7 (3) Å^3	Quader, colourless
Z = 4	0.24 × 0.21 × 0.12 mm

Data collection

Siemens P4 four-circle diffractometer	R_{int} = 0.022
Radiation source: fine-focus sealed tube	θ_{max} = 30.0º
Monochromator: graphite	θ_{min} = 2.3º
T = 293(2) K	h = −12→12
Wyckoff scan mode	k = −1→9
Absorption correction: none	l = −1→17
2446 measured reflections	2 standard reflections
2014 independent reflections	every 100 reflections
1209 reflections with $I > 2\sigma(I)$	intensity decay: <3%

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.074	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.206	$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 0.5757P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ = <0.001
2014 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
109 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. Treatment of hydrogen atoms: Riding model on idealized geometrics with the 1.2 fold (1.5 fold for methyl group) isotropic displacement parameters of the equivalent U^{ij} of the corresponding carbon atom. Hydroxy hydrogen atom position taken from a Fourier-map and subsequently refined as riding atom with the 1.5 fold U-value of the corresponding O-atom.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0012 (2)	0.2130 (3)	−0.0418 (2)	0.0856 (8)
H1	−0.0639	0.1246	−0.0421	0.128*
O2	0.1841 (2)	0.0475 (3)	0.04364 (19)	0.0767 (7)
C1	0.4117 (3)	0.4071 (4)	0.1495 (2)	0.0600 (8)
C2	0.5424 (4)	0.4388 (5)	0.2251 (3)	0.0705 (9)
H2	0.5420	0.5076	0.2886	0.085*
C3	0.6734 (4)	0.3629 (5)	0.2039 (3)	0.0734 (9)
H3	0.7679	0.3801	0.2539	0.088*
C4	0.6743 (3)	0.2649 (5)	0.1124 (3)	0.0727 (9)
H4	0.7690	0.2151	0.1019	0.087*
C5	0.5428 (4)	0.2355 (4)	0.0362 (3)	0.0675 (8)
H5	0.5432	0.1674	−0.0276	0.081*
C6	0.4122 (3)	0.3096 (4)	0.0588 (2)	0.0576 (7)
C7	0.2436 (3)	0.3333 (4)	0.0154 (3)	0.0668 (8)
H7	0.2272	0.3990	−0.0505	0.080*
C8	0.2423 (3)	0.4456 (4)	0.1221 (3)	0.0727 (9)
H8	0.2242	0.5614	0.1032	0.087*
C9	0.1442 (4)	0.3892 (6)	0.2022 (3)	0.0995 (14)
H9A	0.1632	0.4596	0.2660	0.149*
H9B	0.1686	0.2753	0.2232	0.149*
H9C	0.0383	0.3969	0.1689	0.149*
C10	0.1403 (3)	0.1848 (4)	0.0067 (2)	0.0617 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0533 (12)	0.0798 (17)	0.113 (2)	−0.0011 (11)	−0.0138 (12)	0.0160 (14)
O2	0.0538 (12)	0.0698 (15)	0.0980 (17)	0.0005 (11)	−0.0093 (11)	0.0097 (13)
C1	0.0551 (15)	0.0602 (18)	0.0657 (17)	−0.0035 (14)	0.0138 (13)	0.0014 (15)
C2	0.0656 (18)	0.078 (2)	0.0679 (19)	−0.0108 (17)	0.0106 (14)	−0.0081 (17)
C3	0.0538 (16)	0.087 (2)	0.077 (2)	−0.0112 (17)	0.0034 (14)	0.0055 (19)
C4	0.0494 (15)	0.080 (2)	0.092 (2)	−0.0040 (16)	0.0226 (15)	0.002 (2)
C5	0.0662 (18)	0.069 (2)	0.0717 (19)	−0.0105 (16)	0.0244 (15)	−0.0080 (16)
C6	0.0540 (15)	0.0608 (18)	0.0576 (16)	−0.0083 (14)	0.0086 (12)	0.0051 (14)

C7	0.0594 (16)	0.072 (2)	0.0648 (18)	−0.0038 (15)	−0.0010 (13)	0.0070 (16)
C8	0.0555 (16)	0.067 (2)	0.095 (2)	0.0022 (15)	0.0132 (16)	−0.0023 (18)
C9	0.074 (2)	0.120 (3)	0.112 (3)	−0.018 (2)	0.037 (2)	−0.030 (3)
C10	0.0471 (14)	0.079 (2)	0.0560 (16)	0.0043 (15)	0.0005 (11)	0.0009 (16)

Geometric parameters (Å, °)

O1—C10	1.293 (3)	C4—C5	1.385 (4)
O2—C10	1.225 (4)	C5—C6	1.374 (4)
C1—C6	1.368 (4)	C6—C7	1.510 (4)
C1—C2	1.379 (4)	C7—C10	1.492 (5)
C1—C8	1.513 (4)	C7—C8	1.599 (5)
C2—C3	1.378 (5)	C8—C9	1.502 (5)
C3—C4	1.379 (5)		
C6—C1—C2	122.5 (3)	C10—C7—C6	119.0 (3)
C6—C1—C8	94.6 (2)	C10—C7—C8	114.3 (3)
C2—C1—C8	142.9 (3)	C6—C7—C8	85.9 (2)
C3—C2—C1	115.3 (3)	C9—C8—C1	117.4 (3)
C2—C3—C4	122.3 (3)	C9—C8—C7	118.0 (3)
C3—C4—C5	122.0 (3)	C1—C8—C7	85.3 (2)
C6—C5—C4	115.3 (3)	O2—C10—O1	122.9 (3)
C1—C6—C5	122.6 (3)	O2—C10—C7	122.3 (3)
C1—C6—C7	94.2 (2)	O1—C10—C7	114.8 (3)
C5—C6—C7	143.2 (3)		