IUCrData

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(3*Z*)-Tricyclo[6.3.3.0^{1,8}]tetradec-3-ene-10,13-dione

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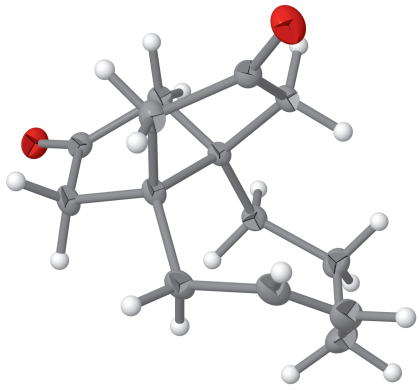
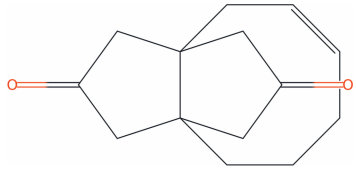
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{14}H_{18}O_2$, features a diquinane being annulated to a cyclooctene in twist-boat-chair conformation. In the crystal, C—H···O hydrogen bonds connect the molecules into centrosymmetric dimers, which are further connected into chains.

3D view



Chemical scheme



Structure description

In a project on cyclooctatetraenophanes (Paquette & Kesselmayer, 1990; Paquette *et al.*, 1992; Detert *et al.*, 1995), the Weiss–Cook condensation (Mitschka *et al.*, 1981) of cyclooct-1-ene-5,6-dione (Yates *et al.*, 1972) gave, after acidic hydrolysis, the 4-ene isomer of the title compound with m.p. = 346 K. The title compound, $C_{14}H_{18}O_2$, m.p. = 366–369 K (Fig. 1), appeared as the main product in a single experiment, probably due to a prolonged acidic treatment. The propellane is composed of a diquinane unit (C1, C8–C14) with a six-membered carbon chain connecting the bridgehead carbon atoms. Both cyclopentanone groups adopt an envelope conformation, the diquinane subunit is C_2 symmetrical and the cyclooctene ring adopts a nearly twist-boat-chair conformation. Four molecules comprise the monoclinic unit cell.

In the crystal, C12—H12A···O1 hydrogen bonds (Table 1, Fig. 2) connect the molecules into centrosymmetric dimers, while C12—H12*B*···O1 hydrogen bonds connect molecules with translation symmetry along the *a*-axis direction to form chains. These chains are connected *via* centers of inversion. A further hydrogen bond, C7—H7*B*···O2, connects these chains.

Synthesis and crystallization

The title compound appeared in a single experiment and was purified by recrystallization from the mixed solvents of chloroform/propan-2-ol to yield colorless crystals with m.p. = 366–369 K. The annotation of NMR signals follows IUPAC nomenclature (cp: cyclopentane, co: cyclooctene), assignment is based on two-dimensional NMR. ^{1}H-NMR (300 MHz, $CDCl_3$): δ = 5.83 (*dt*, 1 H, J_d = 190 Hz, J_t = 7.7 Hz), 5.70 (*dt*, 1 H, J_d = 190 Hz, J_t

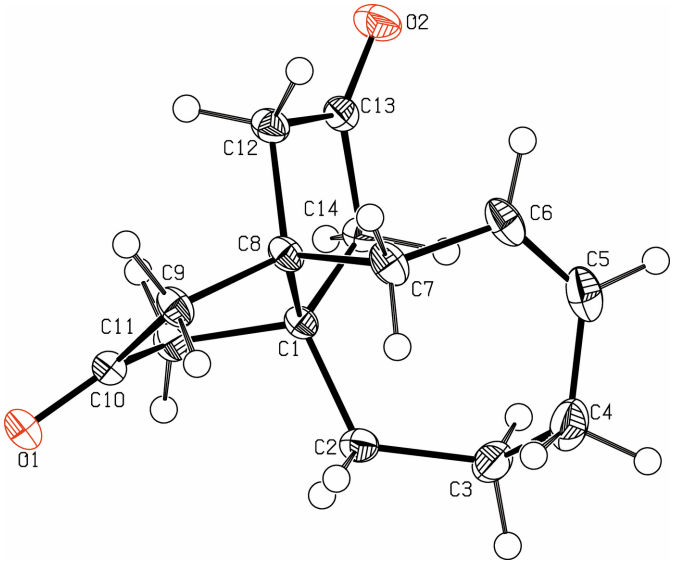


Figure 1
View of the title compound with displacement ellipsoids drawn at the 50% probability level.

= 7.7 Hz), 2.62 (*d*, 2 H, *J* = 29 Hz, cp), 2.53 (*m*, 2 H, J = 29 Hz, cp), 2.42 (*d*, 2 H, J = 13 Hz, co), 2.37 (*d*, 2 H, *J* = 29 Hz, cp),

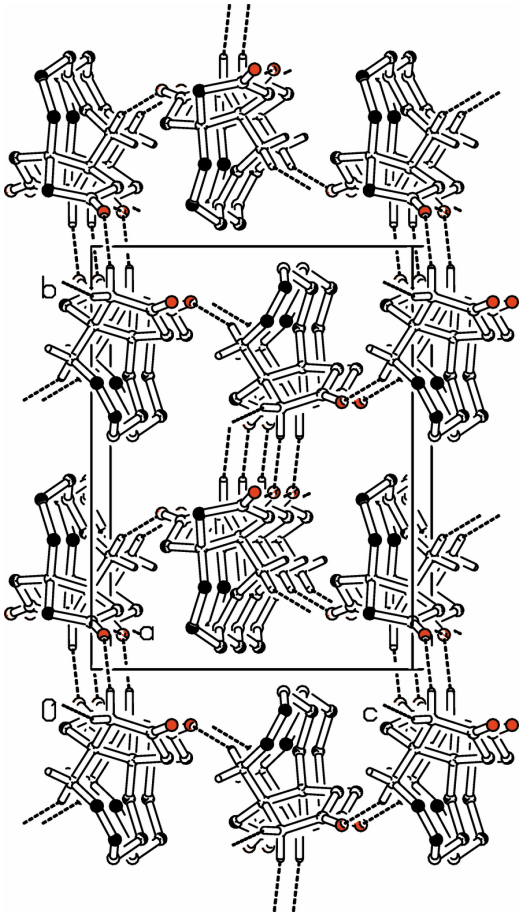


Figure 2
Partial packing diagram. View nearly along the *a*-axis direction. Hydrogen bonds are drawn with dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O2^{i}	0.99	2.48	3.4277 (15)	159
C12—H12*A*···O1^{ii}	0.99	2.49	3.4363 (15)	161
C12—H12*B*···O1^{iii}	0.99	2.56	3.5216 (14)	165

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $x+1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{18}O_2$
M_r	218.28
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	120
a, *b*, *c* (Å)	7.3752 (8), 14.2518 (12), 10.8516 (13)
β (°)	90.735 (10)
V (Å^3)	1140.5 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.72 × 0.60 × 0.24
Data collection	
Diffractometer	Stoe IPDS 2T
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5539, 2712, 2530
R_{int}	0.022
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.045, 0.117, 1.08
No. of reflections	2712
No. of parameters	145
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.21

Computer programs: *X-AREA WinXpose*, *Recipe* and *Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2009).

2.26 (*d*, 2 H, *J* = 29 Hz, cp), 2.23 (*m*, 2 H, co), 1.93 (*m*, 2 H,co), 1.77 (*m*, 2 H, co). ^{13}C-NMR (75 MHz, $CDCl_3$): δ = 216.64 (C═O), 134.17, 127.81 (CH, alkene), 52.31 (*bs*), 52.11, 50.15 (2×CH_q, cp), 49.87 (2×2 CH_2, cp), 35.71, 35.24, 26.65, 26.23 (4×CH_2, co).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2025). **10**, x251132 [https://doi.org/10.1107/S2414314625011320]

(3*Z*)-Tricyclo[6.3.3.0^{1,8}]tetradec-3-ene-10,13-dione

Dieter Schollmeyer and Heiner Detert

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Crystal data

$C_{14}H_{18}O_2$
M_r = 218.28
Monoclinic, $P2_1/n$
a = 7.3752 (8) Å
b = 14.2518 (12) Å
c = 10.8516 (13) Å
β = 90.735 (10)°
V = 1140.5 (2) Å^3
Z = 4
$F(000)$ = 472
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10030 reflections
θ = 3.1–28.4°
μ = 0.08 mm^{-1}
T = 120 K
Block, colorless
0.72 × 0.60 × 0.24 mm

Data collection

Stoe IPDS 2T diffractometer
Detector resolution: 6.67 pixels mm^{-1}
rotation method, ω scans
5539 measured reflections
2712 independent reflections
2530 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.9°, θ_{min} = 3.1°
h = −9→9
k = −18→17
l = −14→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.117
S = 1.08
2712 reflections
145 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.5072P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

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

Refinement. Hydrogen atoms were placed at calculated positions and were refined in the riding-model approximation with $C_{methylene}$—H = 0.99 Å or with vinyl C—H = 0.95 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.34531 (11)	0.41848 (6)	0.54746 (8)	0.0239 (2)
O2	1.05457 (12)	0.36839 (7)	0.21695 (8)	0.0257 (2)
C1	0.64732 (14)	0.28588 (8)	0.36384 (9)	0.0145 (2)
C2	0.53772 (15)	0.19405 (8)	0.38070 (10)	0.0186 (2)
H2A	0.512818	0.188121	0.469794	0.022*
H2B	0.419153	0.203398	0.338688	0.022*
C3	0.61074 (18)	0.09886 (9)	0.33785 (12)	0.0251 (3)
H3A	0.656113	0.106588	0.252984	0.030*
H3B	0.507690	0.054416	0.333558	0.030*
C4	0.7620 (2)	0.05340 (9)	0.41584 (13)	0.0310 (3)
H4A	0.728782	0.057295	0.503799	0.037*
H4B	0.769947	−0.013881	0.393859	0.037*
C5	0.94540 (19)	0.09744 (10)	0.40004 (12)	0.0287 (3)
H5	1.029040	0.066217	0.348308	0.034*
C6	0.99890 (16)	0.17679 (10)	0.45313 (11)	0.0243 (3)
H6	1.117424	0.199361	0.436903	0.029*
C7	0.88049 (15)	0.23258 (9)	0.53777 (10)	0.0208 (2)
H7A	0.957078	0.258313	0.605316	0.025*
H7B	0.791270	0.189606	0.575211	0.025*
C8	0.77751 (14)	0.31382 (8)	0.47614 (10)	0.0159 (2)
C9	0.65282 (15)	0.36043 (9)	0.57096 (10)	0.0190 (2)
H9A	0.624344	0.316026	0.638145	0.023*
H9B	0.711910	0.416559	0.607204	0.023*
C10	0.48199 (15)	0.38777 (8)	0.50110 (11)	0.0177 (2)
C11	0.50950 (15)	0.36783 (8)	0.36606 (10)	0.0178 (2)
H11A	0.558481	0.423645	0.323579	0.021*
H11B	0.393841	0.349429	0.325550	0.021*
C12	0.91158 (15)	0.38249 (8)	0.41639 (10)	0.0184 (2)
H12A	0.864300	0.447451	0.419465	0.022*
H12B	1.030510	0.380495	0.459657	0.022*
C13	0.92976 (14)	0.35030 (8)	0.28456 (10)	0.0171 (2)
C14	0.76690 (15)	0.29068 (8)	0.24869 (10)	0.0165 (2)
H14A	0.805928	0.227018	0.223961	0.020*
H14B	0.699427	0.319666	0.179023	0.020*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0172 (4)	0.0259 (5)	0.0288 (5)	0.0036 (3)	0.0058 (3)	−0.0024 (3)
O2	0.0205 (4)	0.0328 (5)	0.0239 (4)	−0.0049 (3)	0.0070 (3)	−0.0015 (4)
C1	0.0125 (5)	0.0175 (5)	0.0135 (5)	0.0006 (4)	−0.0002 (4)	−0.0001 (4)
C2	0.0170 (5)	0.0202 (5)	0.0185 (5)	−0.0025 (4)	0.0021 (4)	0.0001 (4)
C3	0.0297 (6)	0.0185 (6)	0.0271 (6)	−0.0030 (5)	0.0044 (5)	−0.0019 (4)
C4	0.0403 (8)	0.0193 (6)	0.0337 (7)	0.0060 (5)	0.0052 (6)	0.0029 (5)
C5	0.0306 (7)	0.0298 (7)	0.0259 (6)	0.0143 (5)	0.0041 (5)	0.0027 (5)

C6	0.0190 (5)	0.0333 (7)	0.0205 (5)	0.0105 (5)	0.0015 (4)	0.0040 (5)
C7	0.0178 (5)	0.0287 (6)	0.0160 (5)	0.0062 (4)	−0.0007 (4)	0.0016 (4)
C8	0.0129 (5)	0.0215 (5)	0.0134 (5)	0.0015 (4)	−0.0005 (4)	−0.0017 (4)
C9	0.0163 (5)	0.0258 (6)	0.0150 (5)	0.0030 (4)	0.0013 (4)	−0.0034 (4)
C10	0.0157 (5)	0.0168 (5)	0.0206 (5)	−0.0001 (4)	0.0024 (4)	−0.0008 (4)
C11	0.0155 (5)	0.0201 (5)	0.0176 (5)	0.0027 (4)	−0.0008 (4)	0.0001 (4)
C12	0.0148 (5)	0.0226 (5)	0.0178 (5)	−0.0018 (4)	0.0006 (4)	−0.0041 (4)
C13	0.0154 (5)	0.0181 (5)	0.0179 (5)	0.0008 (4)	0.0008 (4)	0.0002 (4)
C14	0.0165 (5)	0.0196 (5)	0.0135 (5)	−0.0013 (4)	0.0009 (4)	−0.0012 (4)

Geometric parameters (Å, º)

O1—C10	1.2140 (14)	C6—H6	0.9500
O2—C13	1.2122 (14)	C7—C8	1.5334 (15)
C1—C14	1.5402 (14)	C7—H7A	0.9900
C1—C11	1.5487 (15)	C7—H7B	0.9900
C1—C2	1.5503 (15)	C8—C9	1.5395 (14)
C1—C8	1.5923 (14)	C8—C12	1.5403 (15)
C2—C3	1.5339 (17)	C9—C10	1.5130 (16)
C2—H2A	0.9900	C9—H9A	0.9900
C2—H2B	0.9900	C9—H9B	0.9900
C3—C4	1.5351 (19)	C10—C11	1.5089 (15)
C3—H3A	0.9900	C11—H11A	0.9900
C3—H3B	0.9900	C11—H11B	0.9900
C4—C5	1.503 (2)	C12—C13	1.5100 (15)
C4—H4A	0.9900	C12—H12A	0.9900
C4—H4B	0.9900	C12—H12B	0.9900
C5—C6	1.327 (2)	C13—C14	1.5179 (15)
C5—H5	0.9500	C14—H14A	0.9900
C6—C7	1.5030 (16)	C14—H14B	0.9900
C14—C1—C11	111.21 (9)	C7—C8—C9	109.34 (9)
C14—C1—C2	115.99 (9)	C7—C8—C12	110.26 (9)
C11—C1—C2	106.95 (8)	C9—C8—C12	113.54 (9)
C14—C1—C8	105.30 (8)	C7—C8—C1	115.81 (9)
C11—C1—C8	100.89 (8)	C9—C8—C1	105.06 (8)
C2—C1—C8	115.53 (9)	C12—C8—C1	102.75 (8)
C3—C2—C1	121.68 (9)	C10—C9—C8	106.08 (9)
C3—C2—H2A	106.9	C10—C9—H9A	110.5
C1—C2—H2A	106.9	C8—C9—H9A	110.5
C3—C2—H2B	106.9	C10—C9—H9B	110.5
C1—C2—H2B	106.9	C8—C9—H9B	110.5
H2A—C2—H2B	106.7	H9A—C9—H9B	108.7
C2—C3—C4	117.49 (11)	O1—C10—C11	126.38 (11)
C2—C3—H3A	107.9	O1—C10—C9	125.15 (11)
C4—C3—H3A	107.9	C11—C10—C9	108.46 (9)
C2—C3—H3B	107.9	C10—C11—C1	104.69 (9)
C4—C3—H3B	107.9	C10—C11—H11A	110.8

H3A—C3—H3B	107.2	C1—C11—H11A	110.8
C5—C4—C3	114.18 (11)	C10—C11—H11B	110.8
C5—C4—H4A	108.7	C1—C11—H11B	110.8
C3—C4—H4A	108.7	H11A—C11—H11B	108.9
C5—C4—H4B	108.7	C13—C12—C8	105.73 (9)
C3—C4—H4B	108.7	C13—C12—H12A	110.6
H4A—C4—H4B	107.6	C8—C12—H12A	110.6
C6—C5—C4	124.71 (12)	C13—C12—H12B	110.6
C6—C5—H5	117.6	C8—C12—H12B	110.6
C4—C5—H5	117.6	H12A—C12—H12B	108.7
C5—C6—C7	122.98 (12)	O2—C13—C12	125.88 (10)
C5—C6—H6	118.5	O2—C13—C14	124.64 (10)
C7—C6—H6	118.5	C12—C13—C14	109.48 (9)
C6—C7—C8	114.92 (9)	C13—C14—C1	106.04 (9)
C6—C7—H7A	108.5	C13—C14—H14A	110.5
C8—C7—H7A	108.5	C1—C14—H14A	110.5
C6—C7—H7B	108.5	C13—C14—H14B	110.5
C8—C7—H7B	108.5	C1—C14—H14B	110.5
H7A—C7—H7B	107.5	H14A—C14—H14B	108.7
C14—C1—C2—C3	32.71 (14)	C7—C8—C9—C10	−143.18 (10)
C11—C1—C2—C3	157.42 (10)	C12—C8—C9—C10	93.24 (11)
C8—C1—C2—C3	−91.21 (12)	C1—C8—C9—C10	−18.27 (12)
C1—C2—C3—C4	74.85 (14)	C8—C9—C10—O1	172.76 (11)
C2—C3—C4—C5	−75.41 (15)	C8—C9—C10—C11	−5.70 (12)
C3—C4—C5—C6	78.96 (17)	O1—C10—C11—C1	−150.31 (11)
C4—C5—C6—C7	0.5 (2)	C9—C10—C11—C1	28.13 (12)
C5—C6—C7—C8	−96.18 (15)	C14—C1—C11—C10	−149.04 (9)
C6—C7—C8—C9	176.29 (10)	C2—C1—C11—C10	83.40 (10)
C6—C7—C8—C12	−58.22 (13)	C8—C1—C11—C10	−37.76 (10)
C6—C7—C8—C1	57.88 (13)	C7—C8—C12—C13	92.57 (10)
C14—C1—C8—C7	−89.14 (11)	C9—C8—C12—C13	−144.36 (9)
C11—C1—C8—C7	155.10 (9)	C1—C8—C12—C13	−31.45 (10)
C2—C1—C8—C7	40.21 (12)	C8—C12—C13—O2	−158.91 (11)
C14—C1—C8—C9	150.13 (9)	C8—C12—C13—C14	20.82 (12)
C11—C1—C8—C9	34.36 (10)	O2—C13—C14—C1	179.06 (11)
C2—C1—C8—C9	−80.53 (11)	C12—C13—C14—C1	−0.68 (12)
C14—C1—C8—C12	31.12 (10)	C11—C1—C14—C13	89.44 (10)
C11—C1—C8—C12	−84.65 (9)	C2—C1—C14—C13	−148.08 (9)
C2—C1—C8—C12	160.46 (9)	C8—C1—C14—C13	−19.00 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*B*···O2^{i}	0.99	2.48	3.4277 (15)	159

C12—H12*A*···O1^{ii}	0.99	2.49	3.4363 (15)	161
C12—H12*B*···O1^{iii}	0.99	2.56	3.5216 (14)	165

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