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2,4-Dibromo-2,3-dihydro-1*H*-inden-1-yl acetate

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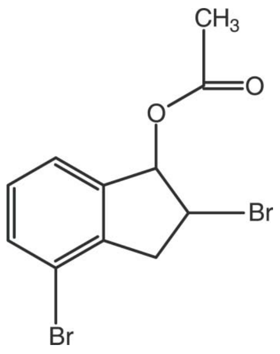
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.009 Å; R factor = 0.067; wR factor = 0.178; data-to-parameter ratio = 19.2.

In the title compound, $C_{11}H_{10}Br_2O_2$, the cyclopentene ring fused to the benzene ring adopts an envelope conformation, with the C atom attached to the Br atom as the flap. The crystal structure does not exhibit any classical hydrogen bonds. The molecular packing is stabilized by van der Waals forces and π–π stacking interactions with a centroid–centroid distance of 3.811 (4) Å.

Related literature

For bromination of hydrocarbons, see: Catto *et al.* (2010); Erenler & Çakmak (2004); Erenler *et al.* (2006); McClure *et al.* (2011); Mitrochkine *et al.* (1995); Snyder & Brill (2011); Wu (2006); Çakmak *et al.* (2006). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{11}H_{10}Br_2O_2$
M_r = 333.99
Triclinic, $P\overline{1}$
a = 8.1423 (7) Å
b = 8.6891 (9) Å
c = 9.0028 (8) Å
α = 76.163 (8)°
β = 68.105 (7)°
γ = 86.397 (8)°
V = 573.60 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 7.04 mm^{-1}
T = 296 K
0.43 × 0.35 × 0.28 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.152, T_{max} = 0.243
6542 measured reflections
2635 independent reflections
1958 reflections with $I > 2\sigma(I)$
R_{int} = 0.110

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.178
S = 1.02
2635 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −1.42 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2559).

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supplementary materials

Acta Cryst. (2012). E**68**, o1884 [doi:10.1107/S1600536812023173]

2,4-Dibromo-2,3-dihydro-1*H*-inden-1-yl acetate

Ísmail Çelik, Mehmet Akkurt, Makbule Yilmaz, Ahmet Tutar, Ramazan Erenler and Canan Kazak

Comment

Brominations of hydrocarbons are important processes in synthetic chemistry (Çakmak *et al.*, 2006; Erenler *et al.*, 2006; Erenler & Çakmak, 2004). Indanes are important class of molecules due to the pharmacological and medicinal properties (Mitrochkine *et al.*, 1995; Catto *et al.*, 2010; Wu, 2006; McClure *et al.*, 2011) as well as natural product chemistry (Snyder & Brill, 2011).

The five-membered C1C6–C9 cyclopentene ring in the title compound, (Fig. 1), exhibits an envelope-shaped conformation, with the C8 atom attached to Br2 atom at the flap [the puckering parameters (Cremer & Pople, 1975) Q(2) = 0.279 (7) Å, $\varphi(2)$ = 290.5 (13) °]. The Br1–C5–C6–C1, Br2–C8–C9–C1, C9–O1–C10–C11 and C9–O1–C10–O2 torsion angles are -178.0 (4), -152.1 (4), -170.2 (6) and 10.3 (9) °, respectively.

In the crystal, there is no classic hydrogen bonds. The crystal structure is stabilized by van der Waals forces and π-π stacking interactions [$Cg2\cdots Cg2(1 - x, 1 - y, 2 - z)$ = 3.811 (4) Å] between the centroids (*Cg*2) of the benzene rings of the adjacent molecules. Fig. 2 shows the molecular packing of the title compound along the *b* axis.

Experimental

To a cooled solution (273 K) of 2,4-dibromo-1-hyodroxyindane (0.2 g, 0.68 mmol) in pyridine (6.0 ml) was added acetic anhydride (1.0 ml) dropwise. After completion of the reaction for 4 h at room temperature, the solvent was removed under reduced pressure to form the solid product which was crystalized from dichloromethane/hexane to yield the 1-acetate-2,4-dibromo-indane (0.21 g, 95%). ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.40–7.60 (m, 3H), 6.0 (d, 1H), 4.90 (dt, 1H), 3.50 (m, 2H), 2.20 (s, 3H).

Refinement

The hydrogen atoms were placed in calculated positions (C—H = 0.93–0.98 Å) and refined as riding atoms with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). Eight poorly fitted reflections (0 1 0), (-2 0 2), (2 - 1 4), (-5 - 8 2), (3 - 1 3), (1 - 1 4), (0 2 1) and (-1 0 2) were omitted from the refinement. The highest residual peak and the deepest hole are located 0.93 and 0.89 Å, respectively, from atom Br2.

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

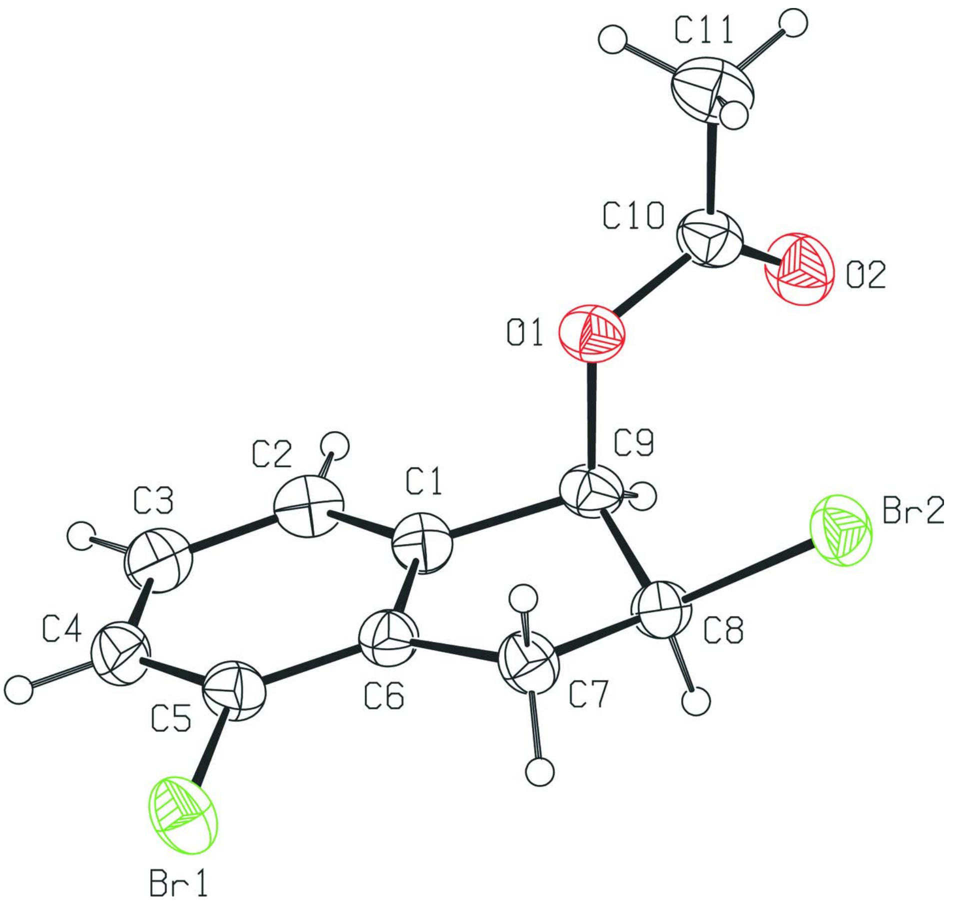

Figure 1

An *ORTEP* drawing of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

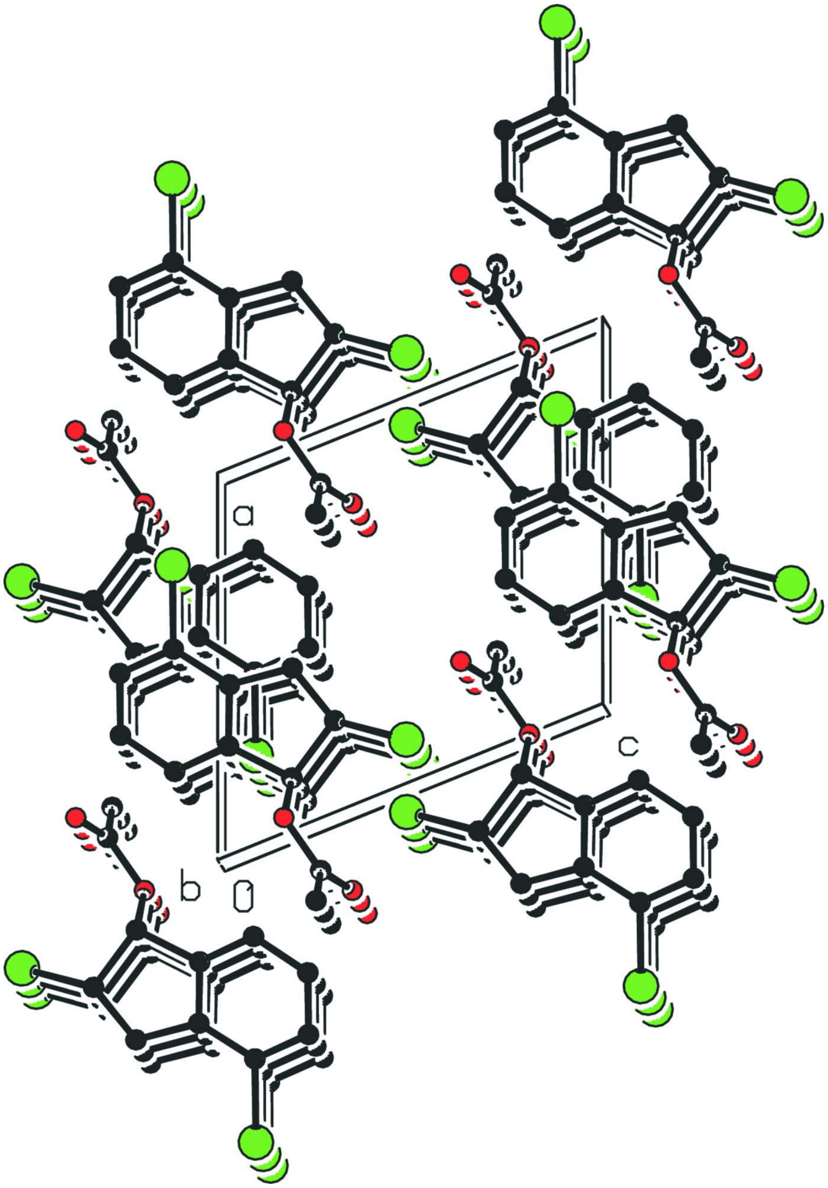

Figure 2

View of the packing of the title compound, along the *b* axis. H atoms are omitted for the sake of clarity.

2,4-Dibromo-2,3-dihydro-1*H*-inden-1-yl acetate

Crystal data

$C_{11}H_{10}Br_2O_2$
M_r = 333.99
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.1423 (7) Å
b = 8.6891 (9) Å
c = 9.0028 (8) Å
α = 76.163 (8)°
β = 68.105 (7)°

γ = 86.397 (8)°
V = 573.60 (10) Å^3
Z = 2
F(000) = 324
D_x = 1.934 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9714 reflections
θ = 2.4–28.1°
μ = 7.04 mm^{-1}

T = 296 K	0.43 × 0.35 × 0.28 mm
Prism, colourless	

Data collection

Stoe IPDS 2 diffractometer	$T_{\min}$ = 0.152, $T_{\max}$ = 0.243
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus	6542 measured reflections
Plane graphite monochromator	2635 independent reflections
Detector resolution: 6.67 pixels mm^{-1}	1958 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.110
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)	$\theta_{\max}$ = 27.5°, $\theta_{\min}$ = 2.5°
	$h = -10 \rightarrow 10$
	$k = -11 \rightarrow 10$
	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.067	H-atom parameters constrained
$wR(F^2)$ = 0.178	$w = 1/[\sigma^2(F_o^2) + (0.1053P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{\max} < 0.001$
2635 reflections	$\Delta\rho_{\max}$ = 1.20 e Å^{-3}
137 parameters	$\Delta\rho_{\min}$ = −1.42 e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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}
Br1	0.22277 (8)	0.18390 (9)	1.10206 (9)	0.0641 (3)
Br2	0.91526 (9)	0.24936 (9)	0.49626 (8)	0.0579 (3)
O1	0.9902 (5)	0.1989 (5)	0.8133 (5)	0.0464 (11)
O2	1.2454 (6)	0.3332 (6)	0.6377 (6)	0.0625 (16)
C1	0.7425 (7)	0.3340 (6)	0.9593 (7)	0.0433 (17)
C2	0.7505 (8)	0.3703 (7)	1.0998 (8)	0.0495 (17)
C3	0.6006 (10)	0.3485 (8)	1.2402 (8)	0.0549 (19)
C4	0.4421 (9)	0.2927 (8)	1.2408 (8)	0.0527 (19)
C5	0.4366 (8)	0.2609 (7)	1.1011 (8)	0.0497 (17)
C6	0.5867 (7)	0.2826 (7)	0.9560 (7)	0.0438 (17)
C7	0.6106 (7)	0.2538 (7)	0.7923 (7)	0.0472 (17)
C8	0.7877 (7)	0.3406 (7)	0.6838 (7)	0.0446 (17)
C9	0.8911 (7)	0.3415 (6)	0.7950 (7)	0.0425 (14)
C10	1.1652 (8)	0.2097 (8)	0.7179 (8)	0.0488 (17)

C11	1.2441 (10)	0.0501 (9)	0.7271 (11)	0.065 (3)
H2	0.85540	0.40870	1.09820	0.0590*
H3	0.60410	0.37060	1.33510	0.0660*
H4	0.34100	0.27740	1.33620	0.0630*
H7A	0.51600	0.29880	0.75610	0.0570*
H7B	0.61690	0.14150	0.79440	0.0570*
H8	0.76310	0.45080	0.64130	0.0530*
H9	0.96600	0.43760	0.75890	0.0510*
H11A	1.18960	−0.01350	0.68340	0.0970*
H11B	1.22470	−0.00010	0.83970	0.0970*
H11C	1.36900	0.06100	0.66390	0.0970*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0461 (4)	0.0769 (5)	0.0653 (5)	−0.0077 (3)	−0.0103 (3)	−0.0237 (4)
Br2	0.0559 (4)	0.0717 (5)	0.0471 (4)	0.0049 (3)	−0.0153 (3)	−0.0228 (3)
O1	0.045 (2)	0.0381 (19)	0.056 (2)	0.0031 (15)	−0.0190 (17)	−0.0109 (17)
O2	0.051 (2)	0.061 (3)	0.067 (3)	−0.006 (2)	−0.014 (2)	−0.010 (2)
C1	0.048 (3)	0.036 (3)	0.046 (3)	0.001 (2)	−0.016 (2)	−0.012 (2)
C2	0.054 (3)	0.042 (3)	0.059 (3)	0.001 (2)	−0.026 (3)	−0.015 (3)
C3	0.070 (4)	0.051 (3)	0.053 (3)	0.013 (3)	−0.029 (3)	−0.022 (3)
C4	0.061 (4)	0.051 (3)	0.043 (3)	0.012 (3)	−0.015 (3)	−0.015 (3)
C5	0.051 (3)	0.043 (3)	0.052 (3)	0.008 (2)	−0.016 (2)	−0.012 (2)
C6	0.046 (3)	0.043 (3)	0.043 (3)	0.006 (2)	−0.018 (2)	−0.010 (2)
C7	0.040 (3)	0.052 (3)	0.049 (3)	−0.002 (2)	−0.014 (2)	−0.014 (3)
C8	0.046 (3)	0.045 (3)	0.045 (3)	0.005 (2)	−0.019 (2)	−0.012 (2)
C9	0.040 (2)	0.033 (2)	0.053 (3)	0.0027 (19)	−0.017 (2)	−0.008 (2)
C10	0.042 (3)	0.056 (3)	0.055 (3)	0.007 (2)	−0.022 (2)	−0.020 (3)
C11	0.062 (4)	0.058 (4)	0.086 (5)	0.012 (3)	−0.031 (4)	−0.034 (4)

Geometric parameters (Å, º)

Br1—C5	1.900 (7)	C7—C8	1.528 (9)
Br2—C8	1.946 (6)	C8—C9	1.531 (8)
O1—C9	1.446 (7)	C10—C11	1.489 (11)
O1—C10	1.357 (8)	C2—H2	0.9300
O2—C10	1.210 (9)	C3—H3	0.9300
C1—C2	1.400 (9)	C4—H4	0.9300
C1—C6	1.384 (9)	C7—H7A	0.9700
C1—C9	1.512 (8)	C7—H7B	0.9700
C2—C3	1.374 (10)	C8—H8	0.9800
C3—C4	1.405 (11)	C9—H9	0.9800
C4—C5	1.368 (9)	C11—H11A	0.9600
C5—C6	1.399 (9)	C11—H11B	0.9600
C6—C7	1.493 (8)	C11—H11C	0.9600
C9—O1—C10	116.4 (5)	C1—C2—H2	121.00
C2—C1—C6	121.9 (6)	C3—C2—H2	121.00
C2—C1—C9	128.1 (6)	C2—C3—H3	120.00

C6—C1—C9	110.0 (5)	C4—C3—H3	120.00
C1—C2—C3	118.9 (6)	C3—C4—H4	120.00
C2—C3—C4	120.1 (6)	C5—C4—H4	120.00
C3—C4—C5	120.0 (6)	C6—C7—H7A	112.00
Br1—C5—C4	120.1 (5)	C6—C7—H7B	111.00
Br1—C5—C6	118.7 (5)	C8—C7—H7A	112.00
C4—C5—C6	121.3 (6)	C8—C7—H7B	112.00
C1—C6—C5	117.8 (6)	H7A—C7—H7B	109.00
C1—C6—C7	112.1 (5)	Br2—C8—H8	108.00
C5—C6—C7	130.1 (6)	C7—C8—H8	108.00
C6—C7—C8	101.3 (5)	C9—C8—H8	108.00
Br2—C8—C7	112.1 (4)	O1—C9—H9	112.00
Br2—C8—C9	113.7 (4)	C1—C9—H9	112.00
C7—C8—C9	107.2 (5)	C8—C9—H9	112.00
O1—C9—C1	106.8 (4)	C10—C11—H11A	109.00
O1—C9—C8	111.9 (5)	C10—C11—H11B	109.00
C1—C9—C8	101.4 (5)	C10—C11—H11C	109.00
O1—C10—O2	124.1 (6)	H11A—C11—H11B	109.00
O1—C10—C11	110.8 (6)	H11A—C11—H11C	109.00
O2—C10—C11	125.0 (7)	H11B—C11—H11C	109.00

C9—O1—C10—C11	−170.2 (6)	C2—C3—C4—C5	−0.4 (11)
C10—O1—C9—C1	−150.4 (5)	C3—C4—C5—C6	0.1 (10)
C10—O1—C9—C8	99.5 (6)	C3—C4—C5—Br1	179.5 (5)
C9—O1—C10—O2	10.3 (9)	Br1—C5—C6—C7	−0.3 (9)
C9—C1—C2—C3	−177.0 (6)	C4—C5—C6—C1	1.4 (9)
C2—C1—C6—C5	−2.7 (9)	C4—C5—C6—C7	179.2 (6)
C2—C1—C6—C7	179.2 (5)	Br1—C5—C6—C1	−178.0 (4)
C6—C1—C2—C3	2.5 (9)	C1—C6—C7—C8	−15.9 (7)
C6—C1—C9—O1	−99.5 (6)	C5—C6—C7—C8	166.3 (6)
C6—C1—C9—C8	17.8 (6)	C6—C7—C8—Br2	152.3 (4)
C2—C1—C9—O1	80.1 (7)	C6—C7—C8—C9	26.7 (6)
C9—C1—C6—C5	176.8 (5)	Br2—C8—C9—C1	−152.1 (4)
C9—C1—C6—C7	−1.3 (7)	C7—C8—C9—O1	86.0 (6)
C2—C1—C9—C8	−162.7 (6)	C7—C8—C9—C1	−27.5 (6)
C1—C2—C3—C4	−0.8 (10)	Br2—C8—C9—O1	−38.6 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O2	0.98	2.36	2.704 (8)	100