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3-Iodo-8β,9α,14α-estra-1,3,5(10)-trien-17-one

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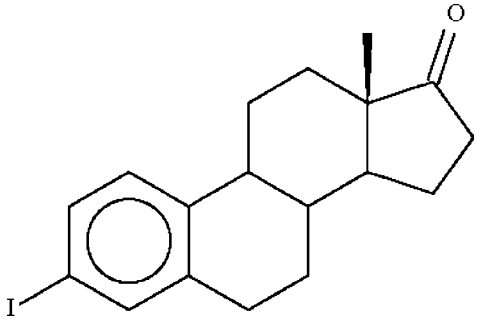
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.018; wR factor = 0.045; data-to-parameter ratio = 19.1.

In the title compound, $C_{18}H_{21}IO$, the cyclohexane ring adopts a chair conformation, whereas the cyclopentane ring and the ten-membered tetraline portions each adopt an envelope conformation. For the five-membered ring, the methine C atom deviates by 0.638 (4) Å (r.m.s. of the four other atoms is 0.005 Å) and for the ten-membered ring, the methine C atom constituting the flap deviates by 0.671 (3) Å (r.m.s. of the other nine atoms is 0.066 Å).

Related literature

There are only a few crystal structure reports of similar compounds; for the methoxyl-substituted derivative, see: Herrmann *et al.* (2006). For the synthesis of the 3-amino-substituted reagent, see: Conrow & Bernstein (1968).



Experimental

Crystal data

$C_{18}H_{21}IO$
M_r = 380.25
Orthorhombic, $P2_12_12_1$
a = 9.9636 (2) Å
b = 10.5246 (2) Å
c = 14.3535 (2) Å
V = 1505.15 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.12 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.636, T_{max} = 0.746
(expected range = 0.690–0.809)
10547 measured reflections
3452 independent reflections
3353 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.045
S = 1.03
3452 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983), 1473 Friedel pairs
Flack parameter: 0.01 (2)

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2435).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Conrow, R. B. & Bernstein, S. (1968). *Steroids*, **11**, 151–164.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Herrmann, P., Kotora, M., Buděšínský, M., Šaman, D. & Císařová, I. (2006). *Org. Lett.* **8**, 1315–1318.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.

supporting information

Acta Cryst. (2009). E**65**, o1195 [doi:10.1107/S1600536809014603]

3-Iodo-8*β*,9*α*,14*α*-estra-1,3,5(10)-trien-17-one

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S1. Experimental

The reactant, 3-amino-estra-1,3,5(1)-trien-17-one, was synthesized by using a literature procedure (Conrow & Bernstein, 1968). The compound (390 mg) was dissolved in hydrobromic acid (54% w/v). Concentrated sulfuric acid (2 ml) and water (4 ml) were added. The solution was cooled in an ice-bath. Sodium nitrite (147 mg) in water (2 ml) was added followed by the addition of excess potassium iodide (1.02 g) dissolved in water (4 ml).

The solution was filtered and the gummy product collected and dissolved in ether–ethyl acetate. The solvent was removed and the crude product (537 mg) chromatographed on a silica-gel (40 g) column. The compound was eluted by chloroform–ethyl acetate (3:1 v/v). The second fraction (465 mg) was a tan glassy material. This was recrystallized from chloroform, methanol and ethyl acetate to give single crystals.

S2. Refinement

Hydrogen atoms were placed at calculated positions (C–H 0.95–1.00 Å) and were treated as riding on their parent carbon atoms, with U(H) set to 1.2–1.5 times U_{eq}(C).

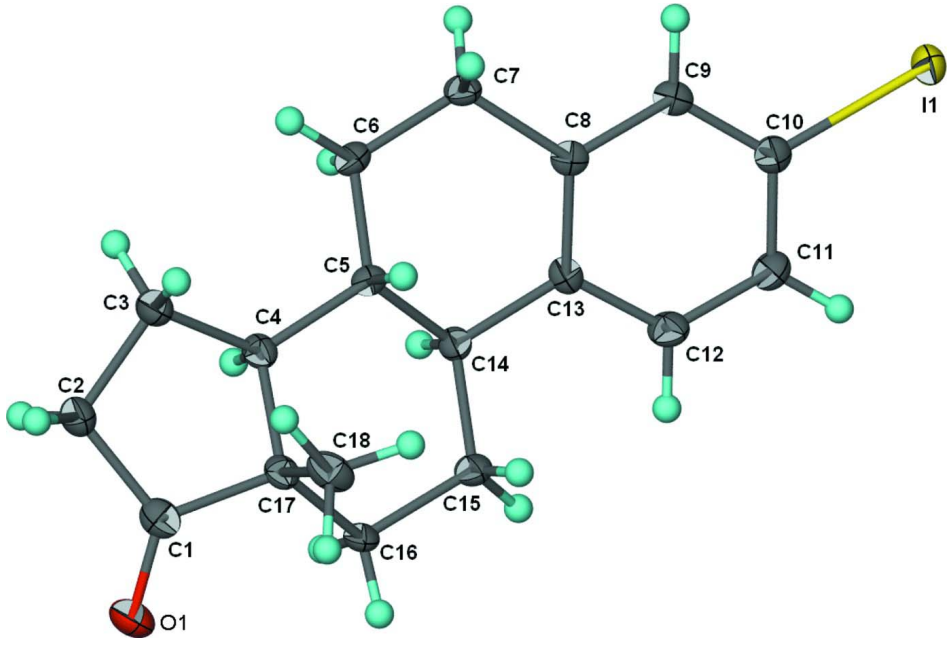

Figure 1

Displacement ellipsoid plot (Barbour, 2001) of $C_{18}H_{21}IO$ at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

3-Iodo-8*β*,9*α*,14*α*-estra-1,3,5(10)-trien-17-one

Crystal data

$C_{18}H_{21}IO$
$M_r = 380.25$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 9.9636$ (2) Å
$b = 10.5246$ (2) Å
$c = 14.3535$ (2) Å
$V = 1505.15$ (5) Å^3
$Z = 4$
$F(000) = 760$
$D_x = 1.678$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6954 reflections
$\theta = 2.4$–$28.3°$
$\mu = 2.12$ mm^{-1}
$T = 100$ K
Irregular block, colorless
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.636$, $T_{max} = 0.746$
10547 measured reflections
3452 independent reflections
3353 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.018$
$wR(F^2) = 0.045$
$S = 1.03$
3452 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Absolute structure: Flack (1983), 1473 Friedel pairs
Absolute structure parameter: 0.01 (2)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.398911 (14)	1.187407 (14)	0.171916 (11)	0.01746 (5)
O1	0.05863 (18)	0.21965 (16)	0.45415 (13)	0.0230 (4)
C1	0.0408 (2)	0.3314 (2)	0.46921 (16)	0.0164 (5)
C2	−0.0646 (3)	0.3860 (2)	0.53518 (19)	0.0192 (6)
H2A	−0.0492	0.3557	0.5996	0.023*
H2B	−0.1558	0.3604	0.5155	0.023*
C3	−0.0483 (3)	0.5320 (2)	0.52963 (18)	0.0185 (5)
H3A	−0.1362	0.5755	0.5333	0.022*
H3B	0.0108	0.5641	0.5798	0.022*
C4	0.0166 (2)	0.5494 (2)	0.43324 (16)	0.0133 (5)
H4	−0.0548	0.5300	0.3864	0.016*
C5	0.0771 (2)	0.6760 (2)	0.40501 (15)	0.0117 (4)

H5	0.1566	0.6930	0.4456	0.014*
C6	−0.0183 (2)	0.7883 (2)	0.41237 (16)	0.0146 (5)
H6A	−0.0606	0.7891	0.4748	0.018*
H6B	−0.0903	0.7799	0.3652	0.018*
C7	0.0573 (2)	0.9119 (2)	0.39693 (17)	0.0143 (5)
H7A	0.1071	0.9338	0.4544	0.017*
H7B	−0.0081	0.9808	0.3848	0.017*
C8	0.1562 (2)	0.9044 (2)	0.31532 (16)	0.0126 (5)
C9	0.2161 (2)	1.0179 (2)	0.28663 (17)	0.0139 (5)
H9	0.1950	1.0952	0.3175	0.017*
C10	0.3063 (2)	1.0176 (2)	0.21308 (17)	0.0144 (5)
C11	0.3359 (2)	0.9057 (2)	0.16548 (19)	0.0169 (5)
H11	0.3958	0.9061	0.1140	0.020*
C12	0.2762 (2)	0.7942 (2)	0.19494 (15)	0.0168 (5)
H12	0.2962	0.7176	0.1628	0.020*
C13	0.1868 (2)	0.7901 (2)	0.27081 (16)	0.0129 (5)
C14	0.1260 (2)	0.6646 (2)	0.30264 (15)	0.0128 (5)
H14	0.0443	0.6503	0.2636	0.015*
C15	0.2184 (3)	0.5497 (2)	0.28692 (18)	0.0176 (5)
H15A	0.2341	0.5399	0.2192	0.021*
H15B	0.3061	0.5670	0.3167	0.021*
C16	0.1628 (2)	0.4241 (2)	0.32552 (19)	0.0162 (5)
H16A	0.0849	0.3967	0.2876	0.019*
H16B	0.2326	0.3574	0.3215	0.019*
C17	0.1194 (2)	0.4408 (2)	0.42701 (15)	0.0133 (5)
C18	0.2423 (2)	0.4595 (2)	0.49110 (18)	0.0191 (5)
H18A	0.2991	0.5274	0.4661	0.029*
H18B	0.2939	0.3804	0.4941	0.029*
H18C	0.2119	0.4827	0.5538	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01720 (7)	0.01525 (7)	0.01993 (8)	−0.00127 (6)	0.00318 (6)	0.00288 (7)
O1	0.0296 (10)	0.0126 (9)	0.0269 (10)	−0.0016 (7)	0.0042 (8)	−0.0013 (7)
C1	0.0185 (11)	0.0152 (12)	0.0155 (11)	−0.0005 (10)	−0.0024 (9)	0.0012 (10)
C2	0.0233 (13)	0.0143 (12)	0.0199 (13)	−0.0021 (10)	0.0064 (10)	0.0013 (10)
C3	0.0237 (12)	0.0134 (12)	0.0185 (12)	−0.0014 (9)	0.0064 (10)	−0.0010 (10)
C4	0.0161 (11)	0.0123 (11)	0.0115 (11)	−0.0015 (9)	0.0001 (9)	−0.0008 (9)
C5	0.0114 (10)	0.0119 (10)	0.0118 (10)	0.0016 (9)	−0.0003 (8)	−0.0013 (9)
C6	0.0148 (11)	0.0152 (12)	0.0138 (11)	0.0014 (9)	0.0031 (9)	−0.0015 (9)
C7	0.0186 (12)	0.0119 (11)	0.0124 (11)	0.0014 (9)	0.0029 (9)	−0.0016 (9)
C8	0.0114 (9)	0.0138 (11)	0.0125 (12)	0.0017 (8)	−0.0034 (9)	0.0005 (10)
C9	0.0148 (12)	0.0131 (11)	0.0137 (11)	0.0008 (9)	−0.0031 (9)	−0.0006 (9)
C10	0.0124 (10)	0.0152 (11)	0.0158 (11)	0.0009 (9)	−0.0013 (9)	0.0014 (9)
C11	0.0182 (11)	0.0187 (11)	0.0137 (11)	0.0004 (9)	0.0030 (10)	−0.0012 (11)
C12	0.0221 (12)	0.0153 (12)	0.0131 (11)	0.0011 (9)	0.0023 (9)	−0.0023 (9)
C13	0.0126 (10)	0.0126 (12)	0.0135 (11)	−0.0009 (9)	−0.0020 (8)	0.0016 (9)

C14	0.0141 (11)	0.0125 (11)	0.0116 (11)	−0.0012 (8)	0.0010 (8)	−0.0008 (8)
C15	0.0232 (13)	0.0132 (12)	0.0165 (12)	0.0008 (10)	0.0080 (10)	−0.0022 (10)
C16	0.0210 (11)	0.0095 (10)	0.0181 (11)	−0.0012 (9)	0.0067 (11)	−0.0038 (12)
C17	0.0161 (12)	0.0115 (10)	0.0124 (11)	−0.0023 (10)	0.0002 (9)	−0.0016 (9)
C18	0.0185 (11)	0.0137 (11)	0.0250 (13)	0.0014 (9)	−0.0075 (10)	0.0028 (11)

Geometric parameters (Å, º)

I1—C10	2.097 (2)	C8—C9	1.397 (3)
O1—C1	1.209 (3)	C8—C13	1.396 (3)
C1—C2	1.526 (3)	C9—C10	1.386 (3)
C1—C17	1.518 (3)	C9—H9	0.9500
C2—C3	1.547 (3)	C10—C11	1.393 (3)
C2—H2A	0.9900	C11—C12	1.382 (3)
C2—H2B	0.9900	C11—H11	0.9500
C3—C4	1.538 (3)	C12—C13	1.407 (3)
C3—H3A	0.9900	C12—H12	0.9500
C3—H3B	0.9900	C13—C14	1.524 (3)
C4—C5	1.517 (3)	C14—C15	1.537 (3)
C4—C17	1.539 (3)	C14—H14	1.0000
C4—H4	1.0000	C15—C16	1.537 (3)
C5—C6	1.520 (3)	C15—H15A	0.9900
C5—C14	1.553 (3)	C15—H15B	0.9900
C5—H5	1.0000	C16—C17	1.529 (3)
C6—C7	1.520 (3)	C16—H16A	0.9900
C6—H6A	0.9900	C16—H16B	0.9900
C6—H6B	0.9900	C17—C18	1.544 (3)
C7—C8	1.533 (3)	C18—H18A	0.9800
C7—H7A	0.9900	C18—H18B	0.9800
C7—H7B	0.9900	C18—H18C	0.9800
O1—C1—C2	125.3 (2)	C8—C9—H9	120.0
O1—C1—C17	126.2 (2)	C9—C10—C11	120.9 (2)
C2—C1—C17	108.5 (2)	C9—C10—I1	119.85 (18)
C1—C2—C3	105.6 (2)	C11—C10—I1	119.26 (18)
C1—C2—H2A	110.6	C12—C11—C10	118.4 (2)
C3—C2—H2A	110.6	C12—C11—H11	120.8
C1—C2—H2B	110.6	C10—C11—H11	120.8
C3—C2—H2B	110.6	C11—C12—C13	122.3 (2)
H2A—C2—H2B	108.7	C11—C12—H12	118.8
C4—C3—C2	102.1 (2)	C13—C12—H12	118.8
C4—C3—H3A	111.4	C8—C13—C12	117.8 (2)
C2—C3—H3A	111.4	C8—C13—C14	121.5 (2)
C4—C3—H3B	111.4	C12—C13—C14	120.7 (2)
C2—C3—H3B	111.4	C13—C14—C15	113.55 (18)
H3A—C3—H3B	109.2	C13—C14—C5	109.98 (19)
C5—C4—C3	120.79 (19)	C15—C14—C5	112.83 (19)
C5—C4—C17	111.87 (18)	C13—C14—H14	106.7

C3—C4—C17	104.08 (19)	C15—C14—H14	106.7
C5—C4—H4	106.4	C5—C14—H14	106.7
C3—C4—H4	106.4	C16—C15—C14	114.06 (19)
C17—C4—H4	106.4	C16—C15—H15A	108.7
C4—C5—C6	114.57 (18)	C14—C15—H15A	108.7
C4—C5—C14	108.02 (18)	C16—C15—H15B	108.7
C6—C5—C14	108.79 (18)	C14—C15—H15B	108.7
C4—C5—H5	108.4	H15A—C15—H15B	107.6
C6—C5—H5	108.4	C17—C16—C15	110.27 (19)
C14—C5—H5	108.4	C17—C16—H16A	109.6
C5—C6—C7	110.23 (18)	C15—C16—H16A	109.6
C5—C6—H6A	109.6	C17—C16—H16B	109.6
C7—C6—H6A	109.6	C15—C16—H16B	109.6
C5—C6—H6B	109.6	H16A—C16—H16B	108.1
C7—C6—H6B	109.6	C1—C17—C16	116.02 (19)
H6A—C6—H6B	108.1	C1—C17—C4	101.32 (18)
C6—C7—C8	112.71 (19)	C16—C17—C4	109.19 (19)
C6—C7—H7A	109.1	C1—C17—C18	105.57 (19)
C8—C7—H7A	109.1	C16—C17—C18	111.01 (19)
C6—C7—H7B	109.1	C4—C17—C18	113.48 (19)
C8—C7—H7B	109.1	C17—C18—H18A	109.5
H7A—C7—H7B	107.8	C17—C18—H18B	109.5
C9—C8—C13	120.6 (2)	H18A—C18—H18B	109.5
C9—C8—C7	117.1 (2)	C17—C18—H18C	109.5
C13—C8—C7	122.3 (2)	H18A—C18—H18C	109.5
C10—C9—C8	119.9 (2)	H18B—C18—H18C	109.5
C10—C9—H9	120.0		

O1—C1—C2—C3	−179.5 (2)	C8—C13—C14—C15	−148.7 (2)
C17—C1—C2—C3	1.1 (3)	C12—C13—C14—C15	31.4 (3)
C1—C2—C3—C4	24.3 (3)	C8—C13—C14—C5	−21.2 (3)
C2—C3—C4—C5	−167.6 (2)	C12—C13—C14—C5	158.9 (2)
C2—C3—C4—C17	−41.0 (2)	C4—C5—C14—C13	179.61 (18)
C3—C4—C5—C6	−55.3 (3)	C6—C5—C14—C13	54.7 (2)
C17—C4—C5—C6	−178.27 (19)	C4—C5—C14—C15	−52.5 (2)
C3—C4—C5—C14	−176.7 (2)	C6—C5—C14—C15	−177.42 (19)
C17—C4—C5—C14	60.3 (2)	C13—C14—C15—C16	175.6 (2)
C4—C5—C6—C7	171.42 (19)	C5—C14—C15—C16	49.6 (3)
C14—C5—C6—C7	−67.6 (2)	C14—C15—C16—C17	−50.9 (3)
C5—C6—C7—C8	43.4 (3)	O1—C1—C17—C16	36.6 (3)
C6—C7—C8—C9	170.8 (2)	C2—C1—C17—C16	−144.0 (2)
C6—C7—C8—C13	−9.4 (3)	O1—C1—C17—C4	154.7 (2)
C13—C8—C9—C10	0.3 (3)	C2—C1—C17—C4	−25.9 (2)
C7—C8—C9—C10	−179.9 (2)	O1—C1—C17—C18	−86.8 (3)
C8—C9—C10—C11	1.6 (4)	C2—C1—C17—C18	92.6 (2)
C8—C9—C10—I1	−178.76 (17)	C15—C16—C17—C1	169.9 (2)
C9—C10—C11—C12	−1.8 (4)	C15—C16—C17—C4	56.3 (2)
I1—C10—C11—C12	178.55 (18)	C15—C16—C17—C18	−69.6 (2)

C10—C11—C12—C13	0.2 (4)	C5—C4—C17—C1	173.37 (18)
C9—C8—C13—C12	−1.9 (3)	C3—C4—C17—C1	41.4 (2)
C7—C8—C13—C12	178.3 (2)	C5—C4—C17—C16	−63.7 (2)
C9—C8—C13—C14	178.2 (2)	C3—C4—C17—C16	164.26 (19)
C7—C8—C13—C14	−1.6 (3)	C5—C4—C17—C18	60.7 (3)
C11—C12—C13—C8	1.7 (4)	C3—C4—C17—C18	−71.3 (2)
C11—C12—C13—C14	−178.4 (2)		