Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

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

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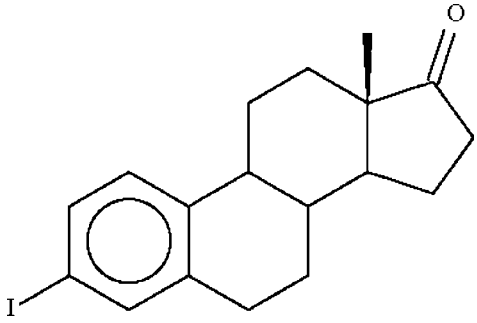
Received 18 April 2009; accepted 20 April 2009

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(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
$\mu$ = 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).

We thank the University of Malaya for supporting this study.

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.

# supplementary materials

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

# 3-Iodo-8$\beta$,9$\alpha$,14$\alpha$-estra-1,3,5(10)-trien-17-one

**K. A. Ketuly, A. H. A. Hadi and S. W. Ng**

## 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.

## 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 $U_{eq}(C)$.

## Figures

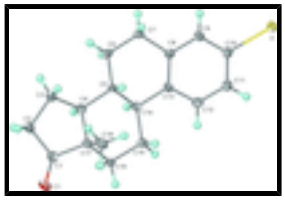


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) of $C_{18}H_{21}IO$ at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

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

*Crystal data*

| | |
|---|---|
| $C_{18}H_{21}IO$ | $F_{000}$ = 760 |
| $M_r$ = 380.25 | $D_x$ = 1.678 Mg m$^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2ac 2ab | Cell parameters from 6954 reflections |
| $a$ = 9.9636 (2) Å | θ = 2.4–28.3° |
| $b$ = 10.5246 (2) Å | μ = 2.12 mm$^{-1}$ |
| $c$ = 14.3535 (2) Å | $T$ = 100 K |
| $V$ = 1505.15 (5) Å$^3$ | Irregular block, colorless |

| | |
|---|---|
| $Z = 4$ | 0.20 × 0.15 × 0.10 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 3452 independent reflections |
| Radiation source: fine-focus sealed tube | 3353 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.021$ |
| $T = 100$ K | $\theta_{max} = 27.5^{o}$ |
| ω scans | $\theta_{min} = 2.4^{o}$ |
| Absorption correction: Multi-scan (SADABS; Sheldrick, 1996) | $h = -12 \rightarrow 12$ |
| $T_{min} = 0.636$, $T_{max} = 0.746$ | $k = -13 \rightarrow 13$ |
| 10547 measured reflections | $l = -18 \rightarrow 18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.018$ | $w = 1/[\sigma^2(F_o^2) + (0.0262P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.045$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $S = 1.03$ | $\Delta\rho_{max} = 0.47$ e Å$^{-3}$ |
| 3452 reflections | $\Delta\rho_{min} = -0.39$ e Å$^{-3}$ |
| 181 parameters | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 1473 Friedel pairs |
| Secondary atom site location: difference Fourier map | Flack parameter: 0.01 (2) |

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

| | *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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| | | | |
|---|---|---|---|
| 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) | | |

**Fig. 1**