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(4*RS*)-Methyl 4-cyano-4-cyclohexyl-4-phenylbutanoate

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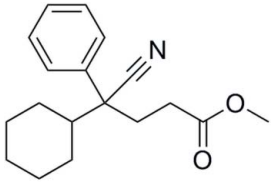
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.153; data-to-parameter ratio = 19.9.

In the crystal structure of the title compound, $C_{18}H_{23}NO_2$, there are only van der Waals interactions present. The cyclohexyl ring has a chair conformation. The longer axes of the displacement parameters of the non-H atoms forming the ethylmethylcarboxylate skeleton are perpendicular to the plane through the non-H atoms of this skeleton.

Related literature

For general background to pharmaceutical applications of methyl 4-cyano-4-cyclohexyl-4-phenylbutanoates, see: Hartmann & Batzl (1986), Hartmann *et al.* (1992); Fadel & Garcia-Argote (1996).



Experimental

Crystal data

$C_{18}H_{23}NO_2$
M_r = 285.37
Monoclinic, $P2_1/c$
a = 8.877 (6) Å
b = 9.120 (6) Å
c = 20.690 (14) Å
β = 95.769 (6)°
V = 1666.5 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 296 K
0.23 × 0.21 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.983, T_{max} = 0.986
13440 measured reflections
3816 independent reflections
2026 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.153
S = 1.06
3816 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*b*); molecular graphics: *SHELXTL* (Sheldrick, 2008*b*); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Key Laboratory of Polymer Materials of Gansu Province (Northwest Normal University).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2251).

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supporting information

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(4*RS*)-Methyl 4-cyano-4-cyclohexyl-4-phenylbutanoate

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S1. Comment

Methyl 4-cyano-4-cyclohexyl-4-phenylbutanoates have attracted widespread attention as a pharmaceutical material to synthesis of 3-(4-aminophenyl)-3-cyclohexylpiperidine-2,6-dione (Fadel & Garcia-Argote, 1996). A variety of 3-(4-aminophenyl)-3-cyclohexylpiperidine-2,6-dione derivatives have been screened as inhibitors of human placental aromatase and treatment of estrogen-dependent diseases (Hartmann & Batzl, 1986, and Hartmann *et al.*, 1992).

The title molecule is shown in Fig. 1. The cyclohexane ring adopts a chair conformation. There are no weak hydrogen bonds and no significant intermolecular π–π electron interactions in the structure. The longer axes of the displacement parameters of the non-hydrogen atoms forming the ethylmethylcarboxylate skeleton are perpendicular to the plane through the non-hydrogen atoms through this skeleton. The displacement parameters of these atoms are rather elongated, possibly due to weak intermolecular interactions between the molecules which may enable more intense thermal agitation of the molecules in the structure.

Fig. 2 shows the packing of the molecules in the title structure.

S2. Experimental

Powdered potassium carbonate (6.9 g, 0.05 mol) and tetrabutylammonium bromide (0.8 g, 0.0025 mol) were added to a solution of 2-cyclohexyl-2-phenylacetonitrile (9.95 g, 0.05 mol) in toluene (35 ml). Methyl acrylate (5.16 g, 0.06 mol) was slowly added in the mixture at 110 °C. After refluxing for 7h, the mixture was cooled and water was added, extracted with ethyl acetate (3 × 50 ml), combined organic solutions, washed with water (3 × 50 ml), and dried over $MgSO_4$. The volatiles were removed in vacuo and the crude product was purified by column chromatography over silica gel with ethyl acetate/petroleum ether (1:20 *v*/*v*). The title compound was isolated in 96% yield as a white solid. Colourless transparent block-like crystals with approx. dimensions 0.2 × 0.4 × 0.5 mm were obtained by slow evaporation of ethyl acetate/petroleum ether (1:20 *v*/*v*).

S3. Refinement

All the H atoms were located in the difference electron density map. Nevertheless, the H atoms were constrained and refined in the riding motion approximation: C_{aryl}—H = 0.93, $C_{methine}$—H = 0.98, $C_{methylene}$—H = 0.97, C_{methyl}—H = 0.96 Å. $U_{iso}(H_{aryl/methane/methylene}) = 1.2 \times U_{eq}(C_{carrier})$ and $U_{iso}(H_{methyl}) = 1.5 \times U_{eq}(C_{carrier})$.

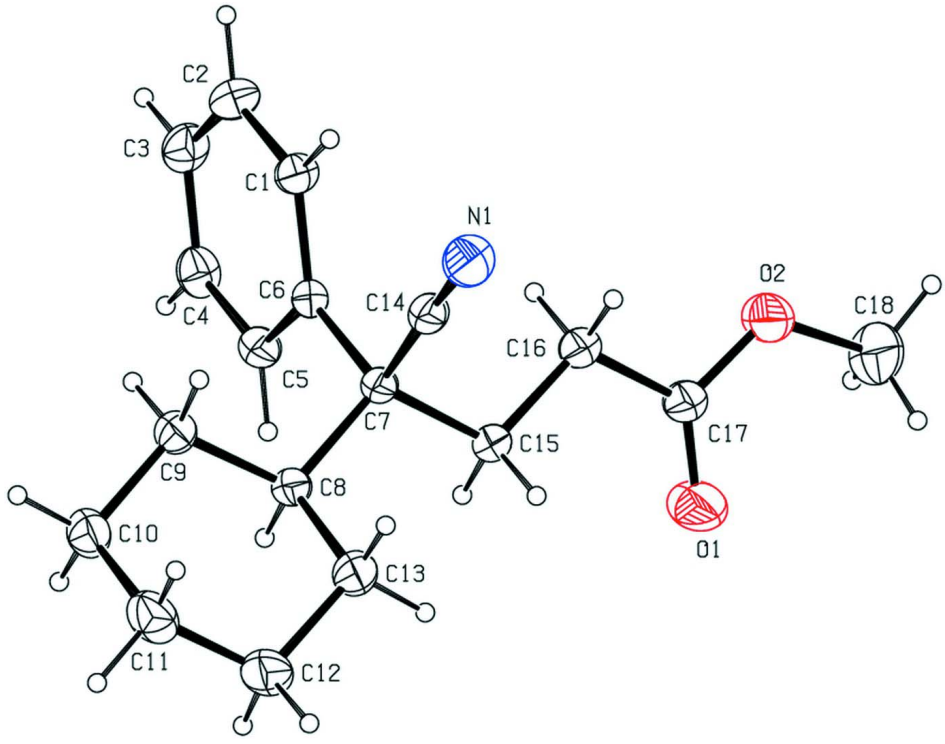

Figure 1

The title molecule with the atom labels and the displacement ellipsoids shown at the 30% probability level.

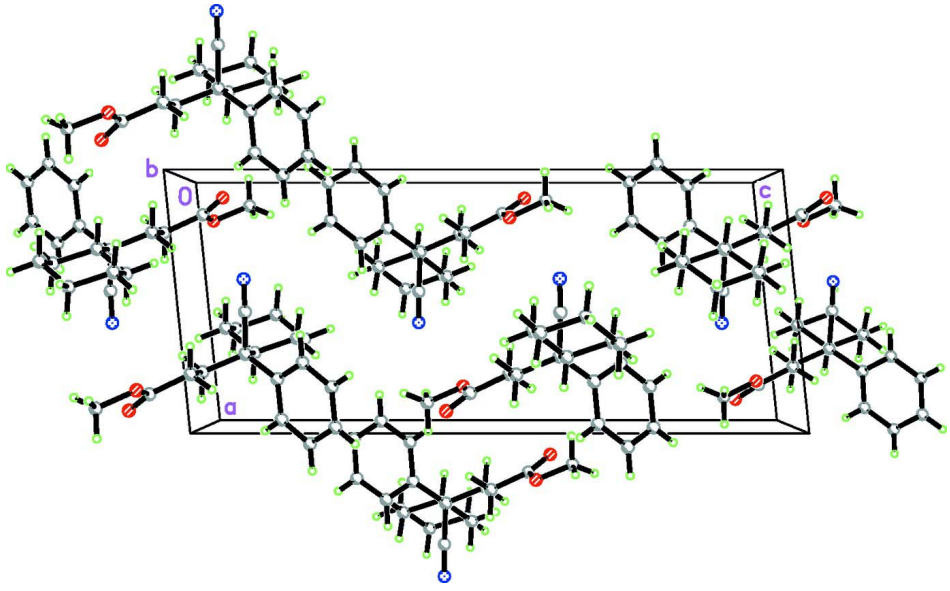

Figure 2

A packing diagram, viewed approximately along the *b* axis. Applied colourse for the atomic species: C - grey, H - green, N - blue; O - red.

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$c = 20.690$ (14) Å
$\beta = 95.769$ (6)°
$V = 1666.5$ (19) Å^3
$Z = 4$
$F(000) = 616$
$D_x = 1.137$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2935 reflections
$\theta = 2.4$–24.9°
$\mu = 0.07$ mm^{-1}
$T = 296$ K
Block, colourless
0.23 × 0.21 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
$T_{min} = 0.983$, $T_{max} = 0.986$
13440 measured reflections
3816 independent reflections
2026 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.4°$
$h = -11 \rightarrow 11$
$k = -10 \rightarrow 11$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.153$
$S = 1.06$
3816 reflections
192 parameters
0 restraints
91 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 0.7938P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0181 (18)

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 $F^2 > \sigma(F^2)$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2736 (3)	0.2805 (2)	0.70020 (10)	0.0531 (6)
H1	0.3761	0.2581	0.7018	0.064*
C2	0.1862 (3)	0.2158 (3)	0.74406 (11)	0.0652 (7)

H2	0.2305	0.1505	0.7748	0.078*
C3	0.0358 (3)	0.2470 (3)	0.74258 (12)	0.0666 (7)
H3	−0.0221	0.2035	0.7723	0.080*
C4	−0.0298 (3)	0.3430 (3)	0.69695 (12)	0.0613 (6)
H4	−0.1326	0.3639	0.6956	0.074*
C5	0.0566 (2)	0.4086 (3)	0.65311 (11)	0.0537 (6)
H5	0.0116	0.4740	0.6225	0.064*
C6	0.2101 (2)	0.3778 (2)	0.65421 (9)	0.0431 (5)
C7	0.3020 (2)	0.4512 (2)	0.60391 (9)	0.0438 (5)
C8	0.3038 (2)	0.6215 (2)	0.61212 (10)	0.0478 (5)
H8	0.1991	0.6555	0.6031	0.057*
C9	0.3583 (3)	0.6683 (2)	0.68142 (10)	0.0539 (6)
H9A	0.2947	0.6233	0.7113	0.065*
H9B	0.4611	0.6339	0.6924	0.065*
C10	0.3539 (3)	0.8345 (3)	0.68922 (12)	0.0664 (7)
H10A	0.2500	0.8681	0.6819	0.080*
H10B	0.3924	0.8605	0.7333	0.080*
C11	0.4475 (3)	0.9097 (3)	0.64200 (15)	0.0814 (8)
H11A	0.4389	1.0152	0.6465	0.098*
H11B	0.5532	0.8832	0.6517	0.098*
C12	0.3937 (4)	0.8653 (3)	0.57336 (14)	0.0828 (9)
H12A	0.4576	0.9109	0.5437	0.099*
H12B	0.2911	0.9004	0.5625	0.099*
C13	0.3972 (3)	0.6996 (3)	0.56456 (12)	0.0712 (8)
H13A	0.5011	0.6656	0.5712	0.085*
H13B	0.3576	0.6751	0.5205	0.085*
C14	0.4603 (3)	0.3992 (3)	0.61248 (10)	0.0525 (6)
C15	0.2354 (3)	0.4061 (2)	0.53446 (10)	0.0519 (6)
H15A	0.1331	0.4440	0.5265	0.062*
H15B	0.2954	0.4507	0.5031	0.062*
C16	0.2321 (3)	0.2420 (3)	0.52422 (11)	0.0638 (7)
H16A	0.3351	0.2051	0.5299	0.077*
H16B	0.1773	0.1972	0.5574	0.077*
C17	0.1604 (3)	0.1957 (3)	0.45895 (11)	0.0600 (6)
C18	0.1176 (4)	−0.0077 (4)	0.38785 (14)	0.1048 (11)
H18A	0.1542	0.0453	0.3525	0.157*
H18B	0.0091	−0.0021	0.3846	0.157*
H18C	0.1481	−0.1085	0.3860	0.157*
N1	0.5830 (3)	0.3602 (3)	0.61735 (10)	0.0782 (7)
O1	0.0970 (3)	0.2726 (2)	0.41959 (11)	0.1196 (9)
O2	0.1799 (3)	0.0558 (2)	0.44876 (9)	0.0983 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0528 (13)	0.0558 (14)	0.0509 (12)	0.0029 (11)	0.0056 (10)	0.0068 (11)
C2	0.0753 (18)	0.0634 (17)	0.0575 (14)	−0.0026 (14)	0.0097 (12)	0.0161 (12)
C3	0.0739 (18)	0.0654 (17)	0.0638 (15)	−0.0129 (14)	0.0233 (13)	0.0035 (13)

C4	0.0519 (14)	0.0630 (16)	0.0714 (16)	−0.0043 (12)	0.0185 (12)	−0.0075 (13)
C5	0.0542 (14)	0.0514 (14)	0.0556 (13)	0.0078 (11)	0.0065 (10)	0.0024 (11)
C6	0.0487 (12)	0.0408 (12)	0.0401 (10)	0.0021 (9)	0.0063 (9)	−0.0015 (9)
C7	0.0475 (12)	0.0452 (13)	0.0393 (10)	0.0096 (10)	0.0066 (9)	0.0034 (9)
C8	0.0526 (13)	0.0461 (13)	0.0456 (11)	0.0047 (10)	0.0086 (9)	0.0050 (10)
C9	0.0567 (14)	0.0526 (14)	0.0534 (13)	−0.0002 (11)	0.0096 (10)	0.0007 (11)
C10	0.0753 (17)	0.0576 (16)	0.0672 (16)	0.0004 (13)	0.0113 (13)	−0.0077 (13)
C11	0.095 (2)	0.0509 (16)	0.101 (2)	−0.0073 (15)	0.0258 (17)	−0.0033 (15)
C12	0.115 (2)	0.0532 (17)	0.085 (2)	−0.0024 (16)	0.0358 (17)	0.0148 (15)
C13	0.101 (2)	0.0556 (16)	0.0617 (15)	−0.0003 (14)	0.0320 (14)	0.0084 (12)
C14	0.0559 (14)	0.0576 (15)	0.0452 (12)	0.0075 (12)	0.0116 (10)	0.0051 (10)
C15	0.0653 (14)	0.0503 (14)	0.0398 (11)	0.0109 (11)	0.0045 (10)	0.0034 (10)
C16	0.0911 (19)	0.0530 (15)	0.0457 (12)	0.0089 (13)	−0.0007 (12)	0.0013 (11)
C17	0.0696 (16)	0.0571 (16)	0.0514 (13)	0.0059 (13)	−0.0031 (11)	0.0007 (12)
C18	0.143 (3)	0.085 (2)	0.081 (2)	−0.026 (2)	−0.0188 (19)	−0.0271 (18)
N1	0.0605 (14)	0.0992 (19)	0.0761 (15)	0.0187 (13)	0.0131 (11)	0.0069 (13)
O1	0.175 (2)	0.0836 (16)	0.0866 (15)	0.0319 (15)	−0.0539 (15)	−0.0107 (12)
O2	0.158 (2)	0.0592 (13)	0.0704 (12)	0.0056 (12)	−0.0256 (12)	−0.0117 (10)

Geometric parameters (Å, º)

C1—C6	1.379 (3)	C10—H10B	0.9700
C1—C2	1.384 (3)	C11—C12	1.508 (4)
C1—H1	0.9300	C11—H11A	0.9700
C2—C3	1.362 (3)	C11—H11B	0.9700
C2—H2	0.9300	C12—C13	1.523 (4)
C3—C4	1.374 (4)	C12—H12A	0.9700
C3—H3	0.9300	C12—H12B	0.9700
C4—C5	1.382 (3)	C13—H13A	0.9700
C4—H4	0.9300	C13—H13B	0.9700
C5—C6	1.389 (3)	C14—N1	1.140 (3)
C5—H5	0.9300	C15—C16	1.512 (3)
C6—C7	1.538 (3)	C15—H15A	0.9700
C7—C14	1.477 (3)	C15—H15B	0.9700
C7—C15	1.553 (3)	C16—C17	1.495 (3)
C7—C8	1.562 (3)	C16—H16A	0.9700
C8—C13	1.525 (3)	C16—H16B	0.9700
C8—C9	1.528 (3)	C17—O1	1.174 (3)
C8—H8	0.9800	C17—O2	1.308 (3)
C9—C10	1.525 (3)	C18—O2	1.445 (3)
C9—H9A	0.9700	C18—H18A	0.9600
C9—H9B	0.9700	C18—H18B	0.9600
C10—C11	1.510 (4)	C18—H18C	0.9600
C10—H10A	0.9700		
C6—C1—C2	120.6 (2)	C12—C11—C10	110.1 (2)
C6—C1—H1	119.7	C12—C11—H11A	109.6
C2—C1—H1	119.7	C10—C11—H11A	109.6

C3—C2—C1	120.6 (2)	C12—C11—H11B	109.6
C3—C2—H2	119.7	C10—C11—H11B	109.6
C1—C2—H2	119.7	H11A—C11—H11B	108.1
C2—C3—C4	119.7 (2)	C11—C12—C13	111.8 (2)
C2—C3—H3	120.1	C11—C12—H12A	109.3
C4—C3—H3	120.1	C13—C12—H12A	109.3
C3—C4—C5	120.1 (2)	C11—C12—H12B	109.3
C3—C4—H4	119.9	C13—C12—H12B	109.3
C5—C4—H4	119.9	H12A—C12—H12B	107.9
C4—C5—C6	120.7 (2)	C12—C13—C8	111.6 (2)
C4—C5—H5	119.7	C12—C13—H13A	109.3
C6—C5—H5	119.7	C8—C13—H13A	109.3
C1—C6—C5	118.2 (2)	C12—C13—H13B	109.3
C1—C6—C7	122.61 (19)	C8—C13—H13B	109.3
C5—C6—C7	119.14 (18)	H13A—C13—H13B	108.0
C14—C7—C6	110.00 (17)	N1—C14—C7	178.1 (2)
C14—C7—C15	107.19 (17)	C16—C15—C7	113.07 (17)
C6—C7—C15	109.40 (18)	C16—C15—H15A	109.0
C14—C7—C8	107.87 (18)	C7—C15—H15A	109.0
C6—C7—C8	111.05 (16)	C16—C15—H15B	109.0
C15—C7—C8	111.26 (16)	C7—C15—H15B	109.0
C13—C8—C9	109.49 (19)	H15A—C15—H15B	107.8
C13—C8—C7	113.23 (18)	C17—C16—C15	113.97 (19)
C9—C8—C7	112.28 (17)	C17—C16—H16A	108.8
C13—C8—H8	107.2	C15—C16—H16A	108.8
C9—C8—H8	107.2	C17—C16—H16B	108.8
C7—C8—H8	107.2	C15—C16—H16B	108.8
C10—C9—C8	111.61 (19)	H16A—C16—H16B	107.7
C10—C9—H9A	109.3	O1—C17—O2	122.2 (2)
C8—C9—H9A	109.3	O1—C17—C16	126.1 (2)
C10—C9—H9B	109.3	O2—C17—C16	111.7 (2)
C8—C9—H9B	109.3	O2—C18—H18A	109.5
H9A—C9—H9B	108.0	O2—C18—H18B	109.5
C11—C10—C9	111.2 (2)	H18A—C18—H18B	109.5
C11—C10—H10A	109.4	O2—C18—H18C	109.5
C9—C10—H10A	109.4	H18A—C18—H18C	109.5
C11—C10—H10B	109.4	H18B—C18—H18C	109.5
C9—C10—H10B	109.4	C17—O2—C18	119.1 (2)
H10A—C10—H10B	108.0		

C6—C1—C2—C3	0.0 (4)	C6—C7—C8—C9	53.8 (2)
C1—C2—C3—C4	0.3 (4)	C15—C7—C8—C9	175.90 (17)
C2—C3—C4—C5	−0.5 (4)	C13—C8—C9—C10	55.4 (3)
C3—C4—C5—C6	0.4 (4)	C7—C8—C9—C10	−177.91 (19)
C2—C1—C6—C5	−0.1 (3)	C8—C9—C10—C11	−57.1 (3)
C2—C1—C6—C7	−179.3 (2)	C9—C10—C11—C12	56.5 (3)
C4—C5—C6—C1	−0.1 (3)	C10—C11—C12—C13	−56.4 (3)
C4—C5—C6—C7	179.1 (2)	C11—C12—C13—C8	56.4 (3)

C1—C6—C7—C14	1.1 (3)	C9—C8—C13—C12	−54.9 (3)
C5—C6—C7—C14	−178.02 (19)	C7—C8—C13—C12	179.0 (2)
C1—C6—C7—C15	118.6 (2)	C14—C7—C15—C16	62.0 (2)
C5—C6—C7—C15	−60.5 (2)	C6—C7—C15—C16	−57.2 (2)
C1—C6—C7—C8	−118.2 (2)	C8—C7—C15—C16	179.7 (2)
C5—C6—C7—C8	62.6 (2)	C7—C15—C16—C17	176.7 (2)
C14—C7—C8—C13	57.8 (2)	C15—C16—C17—O1	−7.4 (4)
C6—C7—C8—C13	178.43 (18)	C15—C16—C17—O2	170.7 (2)
C15—C7—C8—C13	−59.5 (3)	O1—C17—O2—C18	−1.8 (5)
C14—C7—C8—C9	−66.8 (2)	C16—C17—O2—C18	180.0 (2)