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11-[[2-(3-Fluorophenyl)ethyl](methyl)-amino]pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]-undecan-8-one

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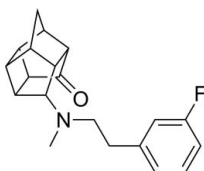
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.087; wR factor = 0.271; data-to-parameter ratio = 13.3.

In the title compound, $\text{C}_{20}\text{H}_{22}\text{FNO}$, the distances close to the carbonyl and amine are: $\text{N}-\text{O} = 3.232$ (4) Å and $\text{N}-\text{C} = 2.666$ (5) Å. The crystal packing is unremarkable.

Related literature

For *in vitro* σ -receptor affinity of trishomocubane derivatives related to the title compound, see: Nguyen *et al.* (1996); Liu *et al.* (1999). For *in vivo* pharmacology of related trishomocubanes, see: Liu *et al.* (2001, 2007). For rationalization of observed structure–affinity relationships of trishomocubanes at σ -receptors using molecular modeling, see: Banister *et al.* (2010). For X-ray crystallographic studies of biologically active trishomocubanes related to the title compound, see: Hambley *et al.* (2000).



Experimental

Crystal data

$\text{C}_{20}\text{H}_{22}\text{FNO}$
 $M_r = 311.39$

Monoclinic, $P2_1/n$
 $a = 10.5450$ (18) Å

$b = 10.980$ (2) Å
 $c = 13.822$ (3) Å
 $\beta = 95.214$ (8)°
 $V = 1593.8$ (5) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 150$ K
 $0.25 \times 0.20 \times 0.15$ mm

Data collection

Bruker–Nonius APEXII FR591 diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)
 $T_{\min} = 0.684$, $T_{\max} = 0.746$

17270 measured reflections
2773 independent reflections
1566 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.087$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.087$
 $wR(F^2) = 0.271$
 $S = 1.14$
2773 reflections

209 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.69$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Data collection: APEX2 (Bruker–Nonius, 2003); cell refinement: SAINT (Bruker–Nonius, 2003); data reduction: SAINT and XPREP (Bruker–Nonius, 2003); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997), WinGX (Farrugia, 1999) and POV-RAY (Cason, 2002); software used to prepare material for publication: enCIFer (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2233).

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supporting information

Acta Cryst. (2010). E66, o2693 [doi:10.1107/S1600536810038523]

11-[[2-(3-Fluorophenyl)ethyl](methyl)amino]pentacyclo- [5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-8-one

Samuel D. Banister, Jack K. Clegg, Raphy Hanani and Michael Kassiou

S1. Comment

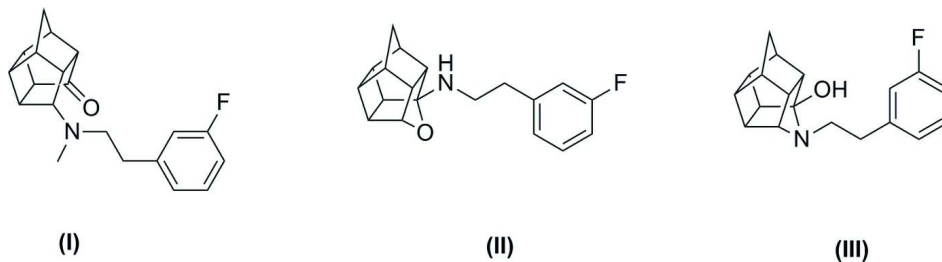
Trishomocubanes have been shown to have *in vitro* σ -receptor affinity and selectivity [Nguyen *et al.*, (1996); Liu *et al.*, (1999)] and a number of their crystal structures have been reported [Hambley *et al.*, (2000)]. Several trishomocubane derivatives synthesized in our laboratory were reported to possess anti-cocaine activity *in vivo* [Liu *et al.*, (2001), (Liu *et al.*, (2007)]. The importance of the nature of the hemiaminal bridge of *N*-(2-(3-fluorophenyl)ethyl)-4-azahexacyclo [5.4.1.0^{2,6}.0^{3,10}.0^{5,9}.0^{8,11}]dodecan-3-ol **I** (Fig. 1) to σ -receptor binding was demonstrated by the reduced affinity, and off-target activity, of the corresponding hemiaminal ether, *N*-(2-(3-fluorophenyl)ethyl)-3-amino-4-oxapentacyclo [5.4.1.0^{2,6}.0^{3,10}.0^{5,9}.0^{8,11}]dodecane **II** [Banister *et al.*, (2010)] (Fig. 1). In our ongoing efforts to elucidate the nature of σ -receptor binding we have synthesized the title compound **III** (Fig. 1) as a methyl homologue of **I**, representing a "locked" form of the non-transannular, aminoketone tautomer of the latter. A molecular and crystal structures were obtained to unambiguously confirm the structure of **III** (Fig. 2), and to identify key interatomic distances, for use in modeling studies. Important distances are those close to the carbonyl and amine, including: N1–O1 = 3.232 (4) Å; N1–C18 = 2.666 (5) Å; O1–C9 = 3.943 (5) Å; C9–C18 = 3.574 (6) Å.

S2. Experimental

A solution of *N*-(2-(3-fluorophenyl)ethyl)-4-azahexacyclo [5.4.1.0^{2,6}.0^{3,10}.0^{5,9}.0^{8,11}]dodecan-3-ol (942 mg, 3.17 mmol) and 37% aqueous formaldehyde (285 μ L, 3.80 mmol, 1.2 equiv.) in ClCH₂CH₂Cl (30 ml) was treated with NaBH(OAc)₃ (3.359 g, 15.85 mmol, 5 equiv.) and the mixture stirred for 18 h. The reaction was quenched with 1 M aqueous NaOH (30 ml), and the layers separated. The aqueous layer was extracted with CH₂Cl₂ (3 \times 15 ml) and the combined organic layers were washed with brine (25 ml), dried (Na₂SO₄) and the solvent evaporated. Purification was achieved using column chromatography on silica eluting with CHCl₃-MeOH-conc. aq. NH₄OH (90:9:1) to give *N*-(2-(3-fluorophenyl)ethyl)-*N*-methyl-11-aminopentacyclo [5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-8-one **III** as colourless crystals (902 mg, 91%); m. pt. 363–364.5 K; *R*_f 0.43 (90:9:1 v/v/v CHCl₃:MeOH: conc. aq. NH₄OH); IR (thin film) cm⁻¹; 2970, 2861, 1721 (C=O), 1582, 1484, 1426, 1343, 1229, 1141, 1059, 981, 939, 906, 791; ¹H NMR (400 MHz, CDCl₃); δ 7.25–7.19 (1H, m, *ArH*), 6.93 (1H, d, *J* = 7.9 Hz, *ArH*), 6.89–6.85 (2H, m, *ArH*), 3.02–2.97 (1H, m, CH), 2.87–2.67 (7H, m, CH), 2.66–2.62 (2H, m, CH), 2.50 (1H, t, *J* = 4.2 Hz, CH), 2.47–2.43 (1H, m, CH), 2.35–2.31 (1H, m, CH), 2.30 (3H, s, CH₃), 1.86 (1H, d, *J* = 10.8 Hz, CHCH₂CH), 1.48 (1H, d, *J* = 10.8 Hz, CHCH₂CH); ¹³C NMR (100.6 MHz, CDCl₃); δ 213.1 (C=O), 163.0 (3'-C, ¹J_{C-F} = 245.3 Hz), 143.4 (1'-C, ³J_{C-F} = 7.4 Hz), 129.9 (5'-C, ³J_{C-F} = 8.3 Hz), 124.5 (6'-C, ⁴J_{C-F} = 2.6 Hz), 115.6 (2'-C, ²J_{C-F} = 20.8 Hz), 112.9 (4'-C, ²J_{C-F} = 21.1 Hz), 64.6 (CH), 57.1 (CH₂), 51.6 (CH), 50.1 (CH), 46.4 (CH), 42.1 (CH), 41.6 (CH), 41.4 (CH), 40.8 (CH), 40.2 (CH), 38.5 (CH₂), 37.2 (CH), 31.7 (CH₂); *m/z* (+ESI) 312.13 ([*M* + H]⁺, 100); Anal. (C₂₀H₂₂NOF): calc, C 77.14, H 7.12, N 4.50; found, C 76.90, H 7.19, N 4.55. Crystals suitable for X-ray diffraction were grown by the slow evaporation of a hexane solution.

S3. Refinement

C bound H atoms were included in idealized positions and refined using a riding-model approximation with aromatic C–H bond lengths fixed at 0.95 Å and aliphatic bond lengths at 1.00 Å, 0.99 Å and 0.98 Å for methine, methylene and methyl H atoms respectively. $U_{\text{iso}}(\text{H})$ values were fixed at $1.2U_{\text{eq}}$ of the parent C atoms, except for the methyl protons, which were fixed at $1.5U_{\text{eq}}(\text{C})$. The highest residual peak is 0.69 eÅ⁻³ and is located 1.17 Å from C12 with the deepest hole -0.47 eÅ⁻³ 1.07 Å from F1.

**Figure 1**

Chemical structures of **I**, **II** and **III**.

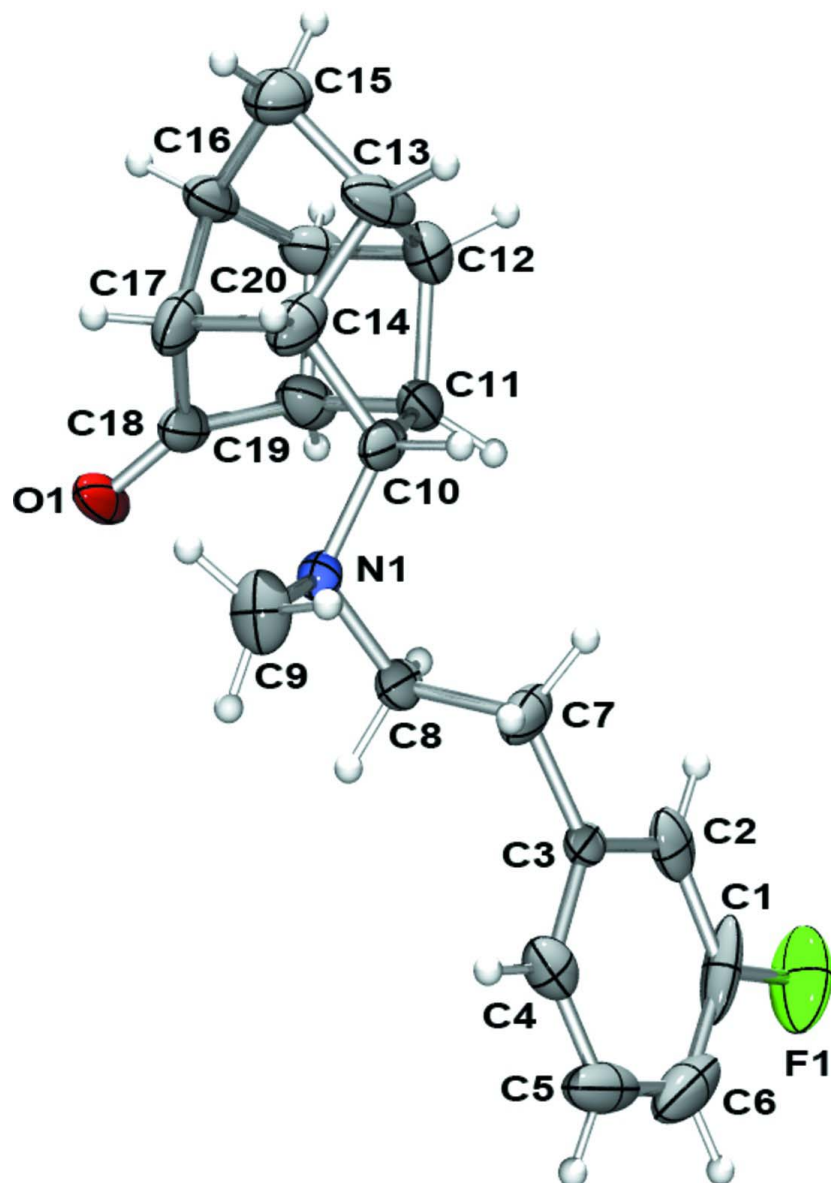


Figure 2

221ORTEP representation of **III** with atom numbering scheme. Displacement ellipsoids are shown at 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

11-[[2-(3-Fluorophenyl)ethyl](methyl)amino]pentacyclo [5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-8-one

Crystal data

C₂₀H₂₂FNO

M_r = 311.39

Monoclinic, *P*2₁/*n*

Hall symbol: -*P* 2₁/*n*

a = 10.5450 (18) Å

b = 10.980 (2) Å

c = 13.822 (3) Å

β = 95.214 (8)°

V = 1593.8 (5) Å³

Z = 4

F(000) = 664

D_x = 1.298 Mg m⁻³

Melting point: 363.5 K

Mo *Kα* radiation, *λ* = 0.71073 Å

Cell parameters from 2297 reflections

θ = 2.7–23.1°

$\mu = 0.09 \text{ mm}^{-1}$
 $T = 150 \text{ K}$

Block, colourless
 $0.25 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Bruker–Nonius APEXII FR591
 diffractometer
 Radiation source: rotating anode
 Graphite monochromator
 ω and φ scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1999)
 $T_{\min} = 0.684$, $T_{\max} = 0.746$

17270 measured reflections
 2773 independent reflections
 1566 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.087$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -12 \rightarrow 12$
 $k = -13 \rightarrow 12$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.087$
 $wR(F^2) = 0.271$
 $S = 1.14$
 2773 reflections
 209 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
 $w = 1/[\sigma^2(F_o^2) + (0.1415P)^2 + 0.2766P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.69 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.48 \text{ e } \text{Å}^{-3}$

Special details

Experimental. The crystal was coated in Exxon Paratone N hydrocarbon oil and mounted on a thin mohair fibre attached to a copper pin. Upon mounting on the diffractometer, the crystal was quenched to 150 K under a cold nitrogen gas stream supplied by an Oxford Cryosystems Cryostream and data were collected at this temperature.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5938 (5)	0.3609 (4)	0.6172 (5)	0.0588 (17)
C2	0.5803 (4)	0.2626 (4)	0.6825 (3)	0.0399 (11)
H2	0.5450	0.2744	0.7426	0.048*
C3	0.6209 (3)	0.1482 (3)	0.6545 (3)	0.0250 (9)
C4	0.6703 (4)	0.1374 (4)	0.5656 (3)	0.0382 (11)
H4	0.6988	0.0598	0.5462	0.046*
C5	0.6797 (5)	0.2343 (6)	0.5047 (4)	0.0609 (16)
H5	0.7123	0.2220	0.4436	0.073*
C6	0.6442 (6)	0.3449 (6)	0.5291 (5)	0.0680 (18)
H6	0.6532	0.4120	0.4869	0.082*
C7	0.6123 (5)	0.0379 (4)	0.7189 (3)	0.0503 (13)

H7A	0.6156	0.0645	0.7875	0.060*
H7B	0.6865	-0.0156	0.7121	0.060*
C8	0.4885 (4)	-0.0351 (4)	0.6931 (3)	0.0304 (10)
H8A	0.4162	0.0131	0.7137	0.036*
H8B	0.4758	-0.0436	0.6216	0.036*
C9	0.5715 (4)	-0.2420 (4)	0.6958 (4)	0.0490 (13)
H9A	0.6587	-0.2249	0.7233	0.074*
H9B	0.5661	-0.2326	0.6250	0.074*
H9C	0.5486	-0.3256	0.7119	0.074*
C10	0.4948 (4)	-0.1550 (4)	0.8423 (3)	0.0348 (11)
H10	0.5795	-0.1212	0.8675	0.042*
C11	0.3871 (4)	-0.0811 (4)	0.8822 (3)	0.0408 (12)
H11	0.3956	0.0094	0.8774	0.049*
C12	0.3700 (5)	-0.1303 (4)	0.9854 (4)	0.0506 (13)
H12	0.3736	-0.0702	1.0400	0.061*
C13	0.4537 (4)	-0.2406 (5)	0.9980 (3)	0.0502 (14)
H13	0.5343	-0.2286	1.0409	0.060*
C14	0.4724 (4)	-0.2781 (4)	0.8898 (3)	0.0479 (13)
H14	0.5445	-0.3364	0.8851	0.057*
C15	0.3636 (5)	-0.3402 (5)	1.0322 (4)	0.0542 (14)
H15A	0.4011	-0.4229	1.0328	0.065*
H15B	0.3314	-0.3216	1.0957	0.065*
C16	0.2635 (4)	-0.3182 (5)	0.9450 (3)	0.0470 (13)
H16	0.1859	-0.3705	0.9451	0.056*
C17	0.3392 (4)	-0.3332 (4)	0.8509 (4)	0.0469 (13)
H17	0.3429	-0.4186	0.8262	0.056*
C18	0.2639 (4)	-0.2484 (4)	0.7847 (3)	0.0379 (11)
C19	0.2540 (4)	-0.1331 (5)	0.8456 (3)	0.0429 (12)
H19	0.1893	-0.0720	0.8196	0.052*
C20	0.2372 (4)	-0.1834 (4)	0.9485 (3)	0.0435 (12)
H20	0.1614	-0.1557	0.9812	0.052*
N1	0.4837 (3)	-0.1570 (3)	0.7364 (2)	0.0259 (8)
O1	0.2010 (3)	-0.2730 (3)	0.70829 (19)	0.0431 (9)
F1	0.5547 (4)	0.4710 (3)	0.6455 (4)	0.128 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.035 (3)	0.016 (2)	0.119 (5)	0.004 (2)	-0.030 (3)	-0.007 (3)
C2	0.023 (2)	0.053 (3)	0.043 (3)	-0.007 (2)	-0.0052 (18)	-0.016 (2)
C3	0.028 (2)	0.026 (2)	0.020 (2)	-0.0055 (18)	-0.0039 (16)	0.0031 (16)
C4	0.036 (2)	0.045 (3)	0.033 (3)	0.001 (2)	0.0019 (19)	-0.007 (2)
C5	0.044 (3)	0.104 (5)	0.034 (3)	-0.023 (3)	-0.001 (2)	0.017 (3)
C6	0.056 (4)	0.070 (4)	0.074 (4)	-0.032 (3)	-0.019 (3)	0.038 (4)
C7	0.053 (3)	0.047 (3)	0.046 (3)	-0.028 (2)	-0.021 (2)	0.021 (2)
C8	0.028 (2)	0.036 (2)	0.026 (2)	-0.0048 (19)	-0.0042 (17)	0.0024 (18)
C9	0.039 (3)	0.044 (3)	0.063 (3)	0.007 (2)	0.001 (2)	-0.011 (2)
C10	0.031 (2)	0.041 (3)	0.030 (2)	-0.017 (2)	-0.0098 (18)	0.0099 (19)

C11	0.046 (3)	0.050 (3)	0.027 (2)	-0.023 (2)	0.009 (2)	-0.006 (2)
C12	0.066 (3)	0.043 (3)	0.040 (3)	-0.015 (3)	-0.009 (2)	-0.005 (2)
C13	0.031 (2)	0.090 (4)	0.028 (2)	-0.011 (3)	-0.0097 (19)	0.012 (2)
C14	0.040 (3)	0.044 (3)	0.056 (3)	-0.014 (2)	-0.015 (2)	0.020 (2)
C15	0.057 (3)	0.059 (3)	0.045 (3)	-0.008 (3)	-0.006 (2)	0.018 (2)
C16	0.046 (3)	0.070 (4)	0.023 (2)	-0.028 (3)	-0.003 (2)	0.006 (2)
C17	0.042 (3)	0.037 (3)	0.058 (3)	-0.016 (2)	-0.013 (2)	0.010 (2)
C18	0.033 (2)	0.051 (3)	0.028 (2)	-0.019 (2)	-0.0031 (19)	0.008 (2)
C19	0.039 (3)	0.058 (3)	0.033 (3)	-0.006 (2)	0.005 (2)	0.001 (2)
C20	0.039 (3)	0.060 (3)	0.032 (3)	-0.002 (2)	0.006 (2)	0.008 (2)
N1	0.0248 (17)	0.0248 (18)	0.0278 (19)	-0.0006 (15)	0.0002 (13)	0.0012 (14)
O1	0.0350 (16)	0.070 (2)	0.0232 (16)	-0.0208 (16)	-0.0058 (13)	0.0024 (14)
F1	0.088 (3)	0.038 (2)	0.246 (6)	0.0133 (19)	-0.048 (3)	-0.037 (2)

Geometric parameters (Å, °)

C1—F1	1.347 (6)	C10—H10	1.0000
C1—C6	1.384 (9)	C11—C12	1.551 (7)
C1—C2	1.422 (7)	C11—C19	1.557 (6)
C2—C3	1.393 (6)	C11—H11	1.0000
C2—H2	0.9500	C12—C13	1.499 (7)
C3—C4	1.382 (6)	C12—C20	1.560 (7)
C3—C7	1.511 (6)	C12—H12	1.0000
C4—C5	1.366 (7)	C13—C15	1.551 (7)
C4—H4	0.9500	C13—C14	1.581 (7)
C5—C6	1.323 (8)	C13—H13	1.0000
C5—H5	0.9500	C14—C17	1.579 (6)
C6—H6	0.9500	C14—H14	1.0000
C7—C8	1.546 (6)	C15—C16	1.546 (6)
C7—H7A	0.9900	C15—H15A	0.9900
C7—H7B	0.9900	C15—H15B	0.9900
C8—N1	1.469 (5)	C16—C20	1.508 (7)
C8—H8A	0.9900	C16—C17	1.595 (7)
C8—H8B	0.9900	C16—H16	1.0000
C9—N1	1.463 (5)	C17—C18	1.484 (6)
C9—H9A	0.9800	C17—H17	1.0000
C9—H9B	0.9800	C18—O1	1.225 (5)
C9—H9C	0.9800	C18—C19	1.529 (7)
C10—N1	1.458 (5)	C19—C20	1.550 (6)
C10—C14	1.531 (6)	C19—H19	1.0000
C10—C11	1.538 (6)	C20—H20	1.0000
F1—C1—C6	121.4 (6)	C13—C12—H12	117.7
F1—C1—C2	116.5 (6)	C11—C12—H12	117.7
C6—C1—C2	122.0 (5)	C20—C12—H12	117.7
C3—C2—C1	117.2 (4)	C12—C13—C15	103.6 (4)
C3—C2—H2	121.4	C12—C13—C14	102.9 (3)
C1—C2—H2	121.4	C15—C13—C14	103.7 (4)

C4—C3—C2	118.3 (4)	C12—C13—H13	115.0
C4—C3—C7	120.2 (4)	C15—C13—H13	115.0
C2—C3—C7	121.5 (4)	C14—C13—H13	115.0
C5—C4—C3	122.4 (5)	C10—C14—C17	111.0 (3)
C5—C4—H4	118.8	C10—C14—C13	102.3 (4)
C3—C4—H4	118.8	C17—C14—C13	103.8 (4)
C6—C5—C4	121.3 (5)	C10—C14—H14	113.0
C6—C5—H5	119.3	C17—C14—H14	113.0
C4—C5—H5	119.3	C13—C14—H14	113.0
C5—C6—C1	118.7 (5)	C16—C15—C13	92.6 (3)
C5—C6—H6	120.6	C16—C15—H15A	113.2
C1—C6—H6	120.6	C13—C15—H15A	113.2
C3—C7—C8	112.0 (3)	C16—C15—H15B	113.2
C3—C7—H7A	109.2	C13—C15—H15B	113.2
C8—C7—H7A	109.2	H15A—C15—H15B	110.5
C3—C7—H7B	109.2	C20—C16—C15	104.1 (4)
C8—C7—H7B	109.2	C20—C16—C17	103.6 (4)
H7A—C7—H7B	107.9	C15—C16—C17	105.2 (4)
N1—C8—C7	116.0 (3)	C20—C16—H16	114.2
N1—C8—H8A	108.3	C15—C16—H16	114.2
C7—C8—H8A	108.3	C17—C16—H16	114.2
N1—C8—H8B	108.3	C18—C17—C14	112.3 (3)
C7—C8—H8B	108.3	C18—C17—C16	99.1 (4)
H8A—C8—H8B	107.4	C14—C17—C16	100.2 (4)
N1—C9—H9A	109.5	C18—C17—H17	114.4
N1—C9—H9B	109.5	C14—C17—H17	114.4
H9A—C9—H9B	109.5	C16—C17—H17	114.4
N1—C9—H9C	109.5	O1—C18—C17	127.6 (4)
H9A—C9—H9C	109.5	O1—C18—C19	126.7 (4)
H9B—C9—H9C	109.5	C17—C18—C19	103.9 (4)
N1—C10—C14	114.6 (3)	C18—C19—C20	103.2 (4)
N1—C10—C11	112.0 (3)	C18—C19—C11	112.2 (4)
C14—C10—C11	99.4 (3)	C20—C19—C11	90.4 (3)
N1—C10—H10	110.1	C18—C19—H19	115.9
C14—C10—H10	110.1	C20—C19—H19	115.9
C11—C10—H10	110.1	C11—C19—H19	115.9
C10—C11—C12	107.3 (4)	C16—C20—C19	106.5 (4)
C10—C11—C19	111.3 (3)	C16—C20—C12	102.4 (4)
C12—C11—C19	89.7 (3)	C19—C20—C12	89.6 (3)
C10—C11—H11	115.2	C16—C20—H20	118.0
C12—C11—H11	115.2	C19—C20—H20	118.0
C19—C11—H11	115.2	C12—C20—H20	118.0
C13—C12—C11	105.8 (4)	C10—N1—C9	113.5 (3)
C13—C12—C20	103.8 (4)	C10—N1—C8	113.1 (3)
C11—C12—C20	90.3 (3)	C9—N1—C8	112.3 (3)
F1—C1—C2—C3	-179.7 (4)	C13—C14—C17—C18	104.4 (4)
C6—C1—C2—C3	0.5 (6)	C10—C14—C17—C16	-109.2 (4)

C1—C2—C3—C4	-0.6 (6)	C13—C14—C17—C16	0.0 (4)
C1—C2—C3—C7	179.2 (4)	C20—C16—C17—C18	-41.0 (4)
C2—C3—C4—C5	-0.4 (6)	C15—C16—C17—C18	-150.0 (4)
C7—C3—C4—C5	179.8 (4)	C20—C16—C17—C14	73.7 (4)
C3—C4—C5—C6	1.6 (7)	C15—C16—C17—C14	-35.3 (5)
C4—C5—C6—C1	-1.6 (8)	C14—C17—C18—O1	137.7 (5)
F1—C1—C6—C5	-179.2 (5)	C16—C17—C18—O1	-117.2 (5)
C2—C1—C6—C5	0.6 (8)	C14—C17—C18—C19	-56.7 (5)
C4—C3—C7—C8	-85.8 (5)	C16—C17—C18—C19	48.4 (4)
C2—C3—C7—C8	94.4 (5)	O1—C18—C19—C20	127.7 (4)
C3—C7—C8—N1	167.1 (4)	C17—C18—C19—C20	-38.0 (4)
N1—C10—C11—C12	154.8 (3)	O1—C18—C19—C11	-136.4 (4)
C14—C10—C11—C12	33.3 (4)	C17—C18—C19—C11	58.0 (4)
N1—C10—C11—C19	58.2 (4)	C10—C11—C19—C18	3.5 (5)
C14—C10—C11—C19	-63.3 (4)	C12—C11—C19—C18	-105.0 (4)
C10—C11—C12—C13	-7.4 (5)	C10—C11—C19—C20	108.1 (4)
C19—C11—C12—C13	104.9 (4)	C12—C11—C19—C20	-0.5 (4)
C10—C11—C12—C20	-111.8 (4)	C15—C16—C20—C19	128.1 (4)
C19—C11—C12—C20	0.5 (4)	C17—C16—C20—C19	18.3 (4)
C11—C12—C13—C15	-128.9 (4)	C15—C16—C20—C12	34.8 (4)
C20—C12—C13—C15	-34.7 (4)	C17—C16—C20—C12	-75.0 (4)
C11—C12—C13—C14	-21.1 (4)	C18—C19—C20—C16	10.6 (4)
C20—C12—C13—C14	73.1 (4)	C11—C19—C20—C16	-102.3 (4)
N1—C10—C14—C17	-55.1 (5)	C18—C19—C20—C12	113.4 (4)
C11—C10—C14—C17	64.4 (5)	C11—C19—C20—C12	0.5 (4)
N1—C10—C14—C13	-165.3 (3)	C13—C12—C20—C16	0.0 (4)
C11—C10—C14—C13	-45.8 (4)	C11—C12—C20—C16	106.3 (4)
C12—C13—C14—C10	42.8 (4)	C13—C12—C20—C19	-106.8 (4)
C15—C13—C14—C10	150.5 (4)	C11—C12—C20—C19	-0.5 (4)
C12—C13—C14—C17	-72.8 (4)	C14—C10—N1—C9	-58.3 (5)
C15—C13—C14—C17	34.9 (5)	C11—C10—N1—C9	-170.6 (3)
C12—C13—C15—C16	53.3 (4)	C14—C10—N1—C8	172.2 (3)
C14—C13—C15—C16	-53.9 (4)	C11—C10—N1—C8	59.9 (4)
C13—C15—C16—C20	-53.6 (4)	C7—C8—N1—C10	61.1 (5)
C13—C15—C16—C17	55.0 (4)	C7—C8—N1—C9	-69.0 (5)
C10—C14—C17—C18	-4.9 (6)		
