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3β-Chloro-*N*-methoxy-*N*-methylcholest-5-ene-24-carboxamide

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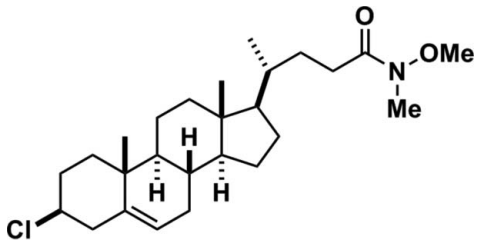
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.157; data-to-parameter ratio = 22.6.

The title compound, $C_{26}H_{42}ClNO_2$, is a 3β-chloro steroid with a Weinreb amide at the C-24 position. The two cyclohexane and the cyclohexene rings adopt chair and boat conformations, respectively. The cyclopentane ring has an envelope conformation.

Related literature

The title compound was obtained as part of our studies on the synthesis of chlorinated steroids as antimalarial agents. For chlorination of 3β-hydroxyl-5-Δ steroids, see: Liu *et al.* (2005). For antimalarial steroids, see: Corrales *et al.* (2011); Sharma *et al.* (2008). For the emerging role of chlorinated lipids and fatty acids in pathology, see: Spickett (2007). For the use of steryl chlorides as synthetic intermediates, see: Ochi *et al.* (1977). For liquid crystal properties of steryl chlorides, see: Leder (1971). For chloroquine-resistant malaria, see: Wellems & Plowe (2001). For drug resistance in malaria, see: Bloland (2001).



Experimental

Crystal data

$C_{26}H_{42}ClNO_2$ M_r = 436.06
Orthorhombic, $P2_12_12_1$
a = 7.5263 (2) Å
b = 16.2157 (4) Å
c = 20.8850 (5) Å
V = 2548.89 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 296 K
0.31 × 0.09 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.949, T_{max} = 0.987
23203 measured reflections
6244 independent reflections
3307 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.157
S = 1.01
6244 reflections
276 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983), 2662 Friedel pairs
Flack parameter: −0.04 (9)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 1999); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2071).

References

Bloland, P. B. (2001). World Health Organization, WHO/CDS/CSR/DRS/2001.4. http://www.who.int/emc.
Bruker (1999). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Corrales, R. C. N. R., de Souza, N. B., Pinheiro, L. S., Abramo, C., Coimbra, E. S. & Da Silva, A. D. (2011). *Biomed. Pharmacother.* **65**, 198–203.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Leder, L. B. (1971). *J. Chem. Phys.* **54**, 4671–4675.
Liu, F.-W., Liu, H.-M., Zhang, Y.-B., Zhang, J.-Y. & Tian, L.-H. (2005). *Steroids*, **70**, 825–830.
Ochi, K., Matsunaga, I. & Shindo, M. (1977). *Steroids*, **30**, 795–803.
Sharma, U., Srivastava, K., Puri, S. K. & Singh, C. (2008). *Med. Chem. Res.* **17**, 326–334.
Sheldrick, G. M. (2008*a*). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008*b*). *Acta Cryst.* A**64**, 112–122.
Spickett, C. M. (2007). *Pharmacol. Ther.* **115**, 400–409.
Wellems, T. E. & Plowe, C. V. (2001). *J. Infect. Dis.* **184**, 770–776.

supplementary materials

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3*β*-Chloro-*N*-methoxy-*N*-methylcholest-5-ene-24-carboxamide

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Comment

The synthesis of the title compound started with the alcohol protection of 5-cholenic acid 3*β*-ol methyl ester with *tert*-butyldimethyl silyl chloride. Then it was derivatized to the Weinreb amide in the presence of *N*-methoxy *N*-methyl hydroxylamine hydrochloride and dimethyl aluminium chloride with the concomitant chlorination of the 3*β*-silyl ether to afford compound **1** with retention of configuration at the C-3 position.

This synthetic route is part of our studies on the synthesis of 3*β*-chloro steroids as antimalarial agents. There is an urgent need to develop new classes of antimalarial drugs due to the development of resistance by the parasite to the commonly used drugs, such as chloroquine [Bloland (2001) and Wellems and Plowe (2001)]. Compound **1** was screened against *Plasmodium falciparum*, but unfortunely, it showed negligible activity (37.4% inhibition). Our future plans include the synthesis of additional derivatives with different functional groups at C-24. The structure of the title compound was determined by one-dimensional and two-dimensional NMR spectra. To confirm the structure of compound **1**, X-ray crystallography was performed.

Experimental

Compound **1** was synthesized by the reaction of 5-cholenic acid 3*β*-ol methyl ester (259 mg, 0.67 mmol) and imidazole (114 mg, 1.68 mmol) in 15 ml of CH_2Cl_2/DMF (2:1) with 1.0 *M tert*-butyldimethylsilyl chloride (TBSCl) in CH_2Cl_2 (1.0 ml, 1.0 mmol). The reaction mixture was stirred for 24 h at room temperature, quenched with $NH_4Cl_{(aq)}$ (20 ml), and extracted with CH_2Cl_2 (3 *x* 15 ml). The combined organic layers were dried ($MgSO_4$) and concentrated *in vacuo*. The crude (378.1 mg) was dissolved in CH_2Cl_2 (10 ml), then (OMe)NH(Me).HCl (87.8 mg, 0.9 mmol) and 1.0 *M* Me_2AlCl in hexane (1.5 ml, 1.5 mmol) were added. The reaction mixture was stirred at room temperature for 24 h, quenched with a solution of 1.0 *M* $NaOH_{(aq)}$, and extracted with CH_2Cl_2 (3 *x* 10 ml). The combined organic layers were dried ($MgSO_4$) and concentrated *in vacuo*. Purification by flash-Silica Gel column chromatography [Hex/EtOAc (4:1)] afforded the chlorinated steroid **1** as a white solid in 75% yield (217 mg). Crystals of **1** were obtained by slow evaporation from Hex/CH_2Cl_2 (2:1) at room temperature. Mp 111–113°C; $[\alpha]_D^{20}$ -27.0 (*c* 1.0, $CHCl_3$); IR (film) ν_{max} 2948, 1662, 1386, 994 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 5.33 (br d, 1H), 3.71 (m, 1H), 3.65 (s, 3H), 3.13 (s, 3H), 2.53–2.25 (broad envelope, 4H), 2.04–1.71 (broad envelope, 7H), 1.58–0.82 (broad envelope, 14H), 0.98 (s, 3H), 0.91 (d, *J* = 6.5 Hz, 3H), 0.64 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 175.1 (C),140.6 (C), 122.3 (CH), 61.1 (CH_3), 60.1 (CH), 56.5 (CH), 55.8 (CH), 49.9(CH), 43.3 (CH_2), 42.2 (C), 39.5 (CH_2), 39.0 (CH_2), 36.2 (C), 35.4 (CH), 33.2 (CH_2), 32.1 (CH_3), 31.7 (CH_2), 31.6 (CH), 30.6 (CH_2), 28.7 (CH_3), 28.0 (CH_2), 24.1 (CH_2), 20.8 (CH_2), 19.1 (CH_3), 18.4 (CH_3), 11.7 (CH_3); HRESIMS *m/z* $[M+ H]^+$ 436.2977 (calcd for $C_{26}H_{43}NO_2Cl$, 436.2982).

Refinement

All non-H atoms were refined anisotropically. H atoms were positioned geometrically with C—H=0.96 (CH_3), 0.97 (CH_2), 0.98 (CH) Å and constrained with U_{iso}(H) = 1.5 U_{eq}(parent) for methyl H and U_{iso}(H) = 1.2 U_{eq}(parent) for all other atoms.

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT* (Bruker, 1999); 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* (Sheldrick, 2008*b*).

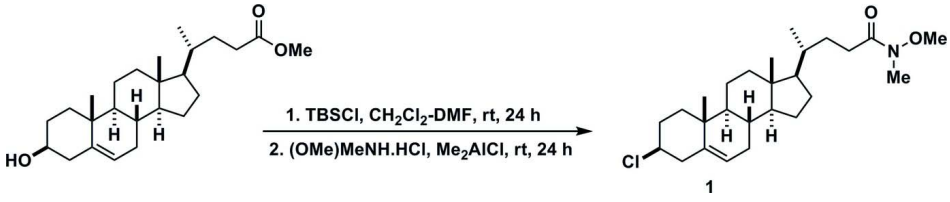

Figure 1

Reaction scheme showing the synthesis of compound **1** from 5-cholenic acid 3β-ol methyl ester with *tert*-butyldimethyl silyl chloride, *N*-methoxy *N*-methyl hydroxylamine hydrochloride, and dimethyl aluminium chloride.

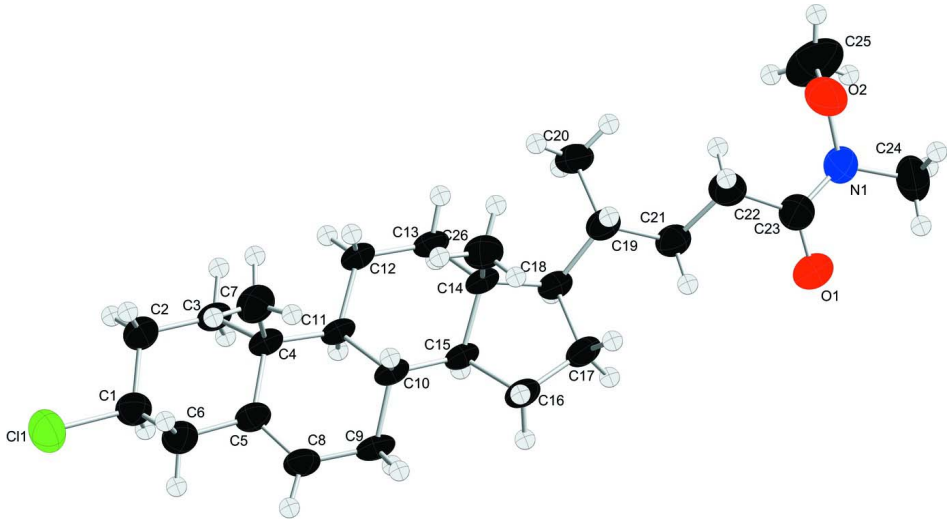

Figure 2

Asymmetric unit of compound **1** with 50% probability displacement ellipsoid for non-hydrogen atoms.

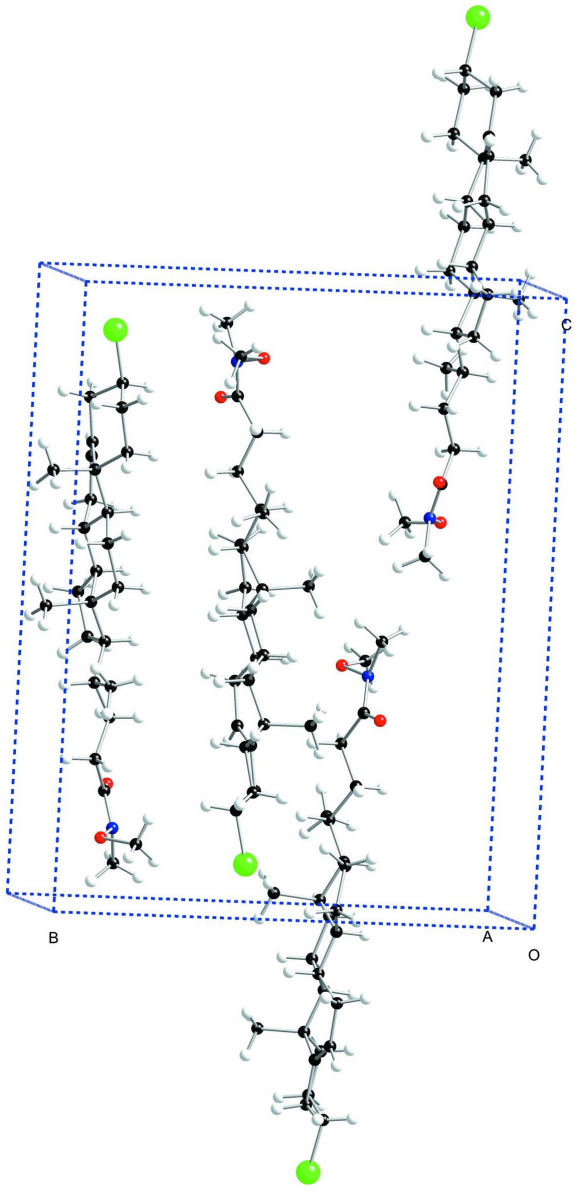

Figure 3

Packing diagram of compound **1**.

3*β*-Chloro-*N*-methoxy-*N*-methylcholest-5-ene-24-carboxamide

Crystal data

$C_{26}H_{42}ClNO_2$
$M_r = 436.06$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.5263$ (2) Å
$b = 16.2157$ (4) Å
$c = 20.8850$ (5) Å
$V = 2548.89$ (11) Å^3
$Z = 4$

$F(000) = 952$
$D_x = 1.136$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5845 reflections
$\theta = 2.7$–$20.9°$
$\mu = 0.17$ mm^{-1}
$T = 296$ K
Needle, colourless
0.31 × 0.09 × 0.08 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} = 0.949, T_{max} = 0.987
23203 measured reflections
6244 independent reflections
3307 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 28.3°, θ_{min} = 1.6°
h = −9→10
k = −19→21
l = −27→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.157
S = 1.01
6244 reflections
276 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
$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.2353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983), 2662 Friedel pairs
Flack parameter: −0.04 (9)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4754 (4)	0.8660 (2)	0.82833 (14)	0.0888 (8)
H1A	0.5059	0.8075	0.8234	0.107*
C2	0.3024 (4)	0.8823 (2)	0.79438 (14)	0.0873 (8)
H2A	0.2679	0.9395	0.8001	0.105*
H2B	0.2095	0.8476	0.8120	0.105*
C3	0.3269 (3)	0.86371 (18)	0.72355 (14)	0.0786 (7)
H3A	0.2150	0.8735	0.7018	0.094*
H3B	0.3551	0.8057	0.7188	0.094*
C4	0.4729 (3)	0.91473 (15)	0.69012 (13)	0.0666 (6)
C5	0.6421 (3)	0.90625 (15)	0.72909 (14)	0.0721 (7)
C6	0.6235 (4)	0.9177 (2)	0.80067 (14)	0.0901 (9)
H6A	0.7345	0.9028	0.8213	0.108*
H6B	0.6005	0.9753	0.8098	0.108*
C7	0.4179 (4)	1.00624 (17)	0.68853 (16)	0.0969 (9)
H7A	0.4991	1.0365	0.6620	0.145*

H7B	0.4205	1.0283	0.7312	0.145*
H7C	0.2999	1.0110	0.6715	0.145*
C8	0.7984 (3)	0.89137 (17)	0.70284 (15)	0.0805 (7)
H8	0.8945	0.8844	0.7303	0.097*
C9	0.8336 (3)	0.88485 (18)	0.63301 (14)	0.0823 (8)
H9A	0.8636	0.8282	0.6228	0.099*
H9B	0.9354	0.9189	0.6224	0.099*
C10	0.6762 (3)	0.91138 (15)	0.59167 (13)	0.0655 (6)
H10	0.6733	0.9718	0.5903	0.079*
C11	0.5021 (3)	0.88085 (15)	0.62200 (13)	0.0653 (6)
H11	0.5154	0.8211	0.6268	0.078*
C12	0.3419 (3)	0.8934 (2)	0.57734 (13)	0.0838 (8)
H12A	0.3119	0.9516	0.5768	0.101*
H12B	0.2410	0.8640	0.5950	0.101*
C13	0.3702 (3)	0.86456 (19)	0.50792 (14)	0.0820 (8)
H13A	0.3805	0.8049	0.5071	0.098*
H13B	0.2676	0.8798	0.4824	0.098*
C14	0.5371 (3)	0.90269 (14)	0.47859 (13)	0.0650 (6)
C15	0.6915 (3)	0.87954 (15)	0.52369 (13)	0.0661 (6)
H15	0.6880	0.8193	0.5272	0.079*
C16	0.8581 (3)	0.89897 (19)	0.48523 (13)	0.0841 (8)
H16A	0.9568	0.8647	0.4989	0.101*
H16B	0.8911	0.9565	0.4899	0.101*
C17	0.8063 (3)	0.87974 (18)	0.41608 (15)	0.0838 (8)
H17A	0.8700	0.8315	0.4010	0.101*
H17B	0.8355	0.9259	0.3885	0.101*
C18	0.6013 (3)	0.86341 (15)	0.41517 (13)	0.0683 (7)
H18	0.5847	0.8037	0.4193	0.082*
C19	0.5143 (4)	0.88930 (16)	0.35166 (13)	0.0748 (7)
H19	0.5342	0.9486	0.3463	0.090*
C20	0.3108 (4)	0.8747 (2)	0.35159 (16)	0.0934 (9)
H20A	0.2551	0.9125	0.3807	0.140*
H20B	0.2863	0.8191	0.3648	0.140*
H20C	0.2650	0.8834	0.3092	0.140*
C21	0.6022 (4)	0.84559 (17)	0.29503 (14)	0.0828 (8)
H21A	0.7299	0.8516	0.2989	0.099*
H21B	0.5753	0.7872	0.2978	0.099*
C22	0.5466 (4)	0.87628 (18)	0.22971 (15)	0.0924 (9)
H22A	0.4238	0.8610	0.2222	0.111*
H22B	0.5535	0.9360	0.2292	0.111*
C23	0.6578 (5)	0.84295 (18)	0.17692 (17)	0.0880 (9)
C24	0.6782 (7)	0.8149 (3)	0.06221 (17)	0.169 (2)
H24A	0.6617	0.8600	0.0331	0.253*
H24B	0.6373	0.7648	0.0426	0.253*
H24C	0.8021	0.8098	0.0725	0.253*
C25	0.2820 (7)	0.8083 (4)	0.1041 (3)	0.206 (3)
H25A	0.2553	0.7869	0.1459	0.308*
H25B	0.3244	0.7644	0.0773	0.308*
H25C	0.1766	0.8316	0.0857	0.308*

C26	0.5199 (4)	0.99713 (16)	0.47055 (16)	0.0927 (9)
H26A	0.4993	1.0220	0.5116	0.139*
H26B	0.4221	1.0092	0.4426	0.139*
H26C	0.6275	1.0187	0.4525	0.139*
Cl1	0.45336 (14)	0.88904 (7)	0.91266 (4)	0.1218 (4)
N1	0.5778 (4)	0.83019 (19)	0.12042 (14)	0.1117 (10)
O1	0.8116 (3)	0.82332 (15)	0.18387 (12)	0.1129 (7)
O2	0.4135 (4)	0.86944 (18)	0.10918 (12)	0.1320 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0777 (19)	0.097 (2)	0.091 (2)	−0.0030 (17)	−0.0064 (16)	−0.0126 (16)
C2	0.0667 (16)	0.102 (2)	0.093 (2)	−0.0062 (16)	0.0050 (15)	0.0007 (17)
C3	0.0428 (12)	0.1019 (19)	0.091 (2)	−0.0067 (12)	0.0009 (12)	0.0023 (15)
C4	0.0453 (12)	0.0642 (14)	0.0902 (18)	0.0023 (10)	0.0011 (12)	−0.0030 (13)
C5	0.0516 (14)	0.0700 (15)	0.0948 (19)	−0.0081 (11)	−0.0044 (13)	−0.0106 (14)
C6	0.0706 (17)	0.107 (2)	0.092 (2)	−0.0107 (16)	−0.0056 (15)	−0.0163 (17)
C7	0.094 (2)	0.0828 (19)	0.114 (2)	0.0204 (16)	0.0149 (19)	−0.0036 (16)
C8	0.0492 (14)	0.0975 (19)	0.095 (2)	−0.0045 (14)	−0.0098 (13)	−0.0044 (16)
C9	0.0358 (12)	0.0944 (19)	0.117 (2)	0.0002 (12)	−0.0061 (13)	−0.0015 (17)
C10	0.0367 (11)	0.0642 (13)	0.0956 (18)	−0.0018 (10)	0.0029 (11)	−0.0019 (13)
C11	0.0356 (11)	0.0671 (14)	0.0932 (17)	−0.0009 (11)	0.0039 (11)	0.0005 (13)
C12	0.0402 (12)	0.121 (2)	0.0902 (19)	0.0062 (13)	0.0041 (12)	−0.0024 (18)
C13	0.0367 (11)	0.111 (2)	0.098 (2)	−0.0021 (13)	−0.0015 (12)	0.0061 (16)
C14	0.0389 (11)	0.0667 (14)	0.0894 (17)	0.0043 (10)	0.0049 (11)	0.0033 (13)
C15	0.0379 (11)	0.0656 (13)	0.0947 (18)	0.0012 (10)	0.0050 (11)	0.0048 (14)
C16	0.0430 (12)	0.110 (2)	0.099 (2)	−0.0041 (13)	0.0065 (13)	0.0074 (17)
C17	0.0508 (13)	0.0987 (19)	0.102 (2)	0.0067 (14)	0.0164 (14)	0.0180 (17)
C18	0.0510 (13)	0.0613 (14)	0.0927 (18)	0.0052 (10)	0.0045 (13)	0.0092 (13)
C19	0.0675 (15)	0.0658 (15)	0.0912 (19)	0.0138 (13)	0.0064 (14)	0.0027 (14)
C20	0.0644 (16)	0.110 (2)	0.106 (2)	0.0140 (17)	−0.0067 (16)	0.0072 (18)
C21	0.0738 (17)	0.0792 (18)	0.095 (2)	0.0127 (14)	0.0052 (16)	0.0098 (14)
C22	0.092 (2)	0.0893 (19)	0.096 (2)	0.0216 (18)	−0.0097 (18)	−0.0166 (18)
C23	0.089 (2)	0.0821 (19)	0.093 (2)	0.0076 (16)	0.0044 (19)	0.0093 (16)
C24	0.187 (5)	0.238 (5)	0.080 (3)	0.081 (4)	0.019 (3)	0.009 (3)
C25	0.130 (4)	0.230 (6)	0.257 (7)	−0.053 (4)	−0.007 (4)	−0.138 (6)
C26	0.091 (2)	0.0758 (17)	0.112 (2)	0.0209 (15)	−0.0037 (18)	−0.0001 (16)
Cl1	0.1189 (7)	0.1578 (9)	0.0887 (5)	−0.0178 (6)	−0.0027 (5)	−0.0117 (6)
N1	0.111 (2)	0.138 (2)	0.0864 (19)	0.045 (2)	0.0087 (18)	−0.0013 (17)
O1	0.0845 (16)	0.1295 (17)	0.1247 (19)	0.0151 (14)	0.0060 (14)	−0.0026 (15)
O2	0.140 (2)	0.146 (2)	0.1100 (18)	0.051 (2)	−0.0210 (16)	−0.0116 (15)

Geometric parameters (Å, º)

C1—C2	1.506 (4)	C14—C18	1.547 (4)
C1—C6	1.510 (4)	C15—C16	1.522 (3)
C1—Cl1	1.808 (3)	C15—H15	0.9800
C1—H1A	0.9800	C16—C17	1.528 (4)
C2—C3	1.521 (4)	C16—H16A	0.9700

C2—H2A	0.9700	C16—H16B	0.9700
C2—H2B	0.9700	C17—C18	1.566 (4)
C3—C4	1.543 (4)	C17—H17A	0.9700
C3—H3A	0.9700	C17—H17B	0.9700
C3—H3B	0.9700	C18—C19	1.538 (4)
C4—C5	1.518 (3)	C18—H18	0.9800
C4—C7	1.541 (4)	C19—C21	1.529 (4)
C4—C11	1.541 (4)	C19—C20	1.550 (4)
C5—C8	1.320 (4)	C19—H19	0.9800
C5—C6	1.513 (4)	C20—H20A	0.9600
C6—H6A	0.9700	C20—H20B	0.9600
C6—H6B	0.9700	C20—H20C	0.9600
C7—H7A	0.9600	C21—C22	1.511 (4)
C7—H7B	0.9600	C21—H21A	0.9700
C7—H7C	0.9600	C21—H21B	0.9700
C8—C9	1.486 (4)	C22—C23	1.486 (4)
C8—H8	0.9300	C22—H22A	0.9700
C9—C10	1.527 (3)	C22—H22B	0.9700
C9—H9A	0.9700	C23—O1	1.209 (4)
C9—H9B	0.9700	C23—N1	1.341 (4)
C10—C15	1.515 (4)	C24—N1	1.453 (4)
C10—C11	1.537 (3)	C24—H24A	0.9600
C10—H10	0.9800	C24—H24B	0.9600
C11—C12	1.538 (3)	C24—H24C	0.9600
C11—H11	0.9800	C25—O2	1.405 (5)
C12—C13	1.538 (4)	C25—H25A	0.9600
C12—H12A	0.9700	C25—H25B	0.9600
C12—H12B	0.9700	C25—H25C	0.9600
C13—C14	1.529 (3)	C26—H26A	0.9600
C13—H13A	0.9700	C26—H26B	0.9600
C13—H13B	0.9700	C26—H26C	0.9600
C14—C15	1.542 (3)	N1—O2	1.410 (4)
C14—C26	1.546 (3)		

C2—C1—C6	111.1 (2)	C15—C14—C18	100.81 (18)
C2—C1—Cl1	110.1 (2)	C26—C14—C18	109.9 (2)
C6—C1—Cl1	109.0 (2)	C10—C15—C16	119.1 (2)
C2—C1—H1A	108.9	C10—C15—C14	115.59 (19)
C6—C1—H1A	108.9	C16—C15—C14	104.4 (2)
Cl1—C1—H1A	108.9	C10—C15—H15	105.5
C1—C2—C3	108.6 (2)	C16—C15—H15	105.5
C1—C2—H2A	110.0	C14—C15—H15	105.5
C3—C2—H2A	110.0	C15—C16—C17	104.3 (2)
C1—C2—H2B	110.0	C15—C16—H16A	110.9
C3—C2—H2B	110.0	C17—C16—H16A	110.9
H2A—C2—H2B	108.4	C15—C16—H16B	110.9
C2—C3—C4	114.8 (2)	C17—C16—H16B	110.9
C2—C3—H3A	108.6	H16A—C16—H16B	108.9
C4—C3—H3A	108.6	C16—C17—C18	107.3 (2)

C2—C3—H3B	108.6	C16—C17—H17A	110.3
C4—C3—H3B	108.6	C18—C17—H17A	110.3
H3A—C3—H3B	107.5	C16—C17—H17B	110.3
C5—C4—C7	108.9 (2)	C18—C17—H17B	110.3
C5—C4—C11	110.07 (19)	H17A—C17—H17B	108.5
C7—C4—C11	111.2 (2)	C19—C18—C14	119.5 (2)
C5—C4—C3	107.8 (2)	C19—C18—C17	112.6 (2)
C7—C4—C3	109.6 (2)	C14—C18—C17	103.2 (2)
C11—C4—C3	109.2 (2)	C19—C18—H18	106.9
C8—C5—C6	121.0 (2)	C14—C18—H18	106.9
C8—C5—C4	122.8 (3)	C17—C18—H18	106.9
C6—C5—C4	116.2 (2)	C21—C19—C18	110.9 (2)
C1—C6—C5	112.2 (2)	C21—C19—C20	110.9 (2)
C1—C6—H6A	109.2	C18—C19—C20	112.3 (2)
C5—C6—H6A	109.2	C21—C19—H19	107.5
C1—C6—H6B	109.2	C18—C19—H19	107.5
C5—C6—H6B	109.2	C20—C19—H19	107.5
H6A—C6—H6B	107.9	C19—C20—H20A	109.5
C4—C7—H7A	109.5	C19—C20—H20B	109.5
C4—C7—H7B	109.5	H20A—C20—H20B	109.5
H7A—C7—H7B	109.5	C19—C20—H20C	109.5
C4—C7—H7C	109.5	H20A—C20—H20C	109.5
H7A—C7—H7C	109.5	H20B—C20—H20C	109.5
H7B—C7—H7C	109.5	C22—C21—C19	115.2 (2)
C5—C8—C9	125.4 (2)	C22—C21—H21A	108.5
C5—C8—H8	117.3	C19—C21—H21A	108.5
C9—C8—H8	117.3	C22—C21—H21B	108.5
C8—C9—C10	113.4 (2)	C19—C21—H21B	108.5
C8—C9—H9A	108.9	H21A—C21—H21B	107.5
C10—C9—H9A	108.9	C23—C22—C21	113.2 (2)
C8—C9—H9B	108.9	C23—C22—H22A	108.9
C10—C9—H9B	108.9	C21—C22—H22A	108.9
H9A—C9—H9B	107.7	C23—C22—H22B	108.9
C15—C10—C9	112.0 (2)	C21—C22—H22B	108.9
C15—C10—C11	109.96 (19)	H22A—C22—H22B	107.7
C9—C10—C11	109.7 (2)	O1—C23—N1	119.7 (3)
C15—C10—H10	108.4	O1—C23—C22	123.1 (3)
C9—C10—H10	108.4	N1—C23—C22	117.1 (3)
C11—C10—H10	108.4	N1—C24—H24A	109.5
C10—C11—C12	112.1 (2)	N1—C24—H24B	109.5
C10—C11—C4	112.8 (2)	H24A—C24—H24B	109.5
C12—C11—C4	113.62 (19)	N1—C24—H24C	109.5
C10—C11—H11	105.9	H24A—C24—H24C	109.5
C12—C11—H11	105.9	H24B—C24—H24C	109.5
C4—C11—H11	105.9	O2—C25—H25A	109.5
C11—C12—C13	115.00 (19)	O2—C25—H25B	109.5
C11—C12—H12A	108.5	H25A—C25—H25B	109.5
C13—C12—H12A	108.5	O2—C25—H25C	109.5
C11—C12—H12B	108.5	H25A—C25—H25C	109.5

C13—C12—H12B	108.5	H25B—C25—H25C	109.5
H12A—C12—H12B	107.5	C14—C26—H26A	109.5
C14—C13—C12	111.6 (2)	C14—C26—H26B	109.5
C14—C13—H13A	109.3	H26A—C26—H26B	109.5
C12—C13—H13A	109.3	C14—C26—H26C	109.5
C14—C13—H13B	109.3	H26A—C26—H26C	109.5
C12—C13—H13B	109.3	H26B—C26—H26C	109.5
H13A—C13—H13B	108.0	C23—N1—O2	118.1 (3)
C13—C14—C15	106.0 (2)	C23—N1—C24	121.9 (3)
C13—C14—C26	112.0 (2)	O2—N1—C24	113.2 (3)
C15—C14—C26	111.8 (2)	C25—O2—N1	108.1 (3)
C13—C14—C18	115.7 (2)		