IUCrData

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(1*S*,2*R*,8*R*)-11-Amino-2,2-dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodec-10-en-9-one

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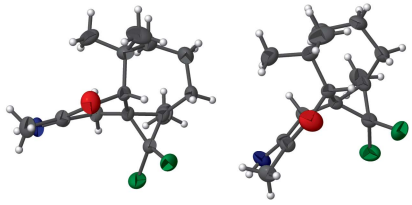
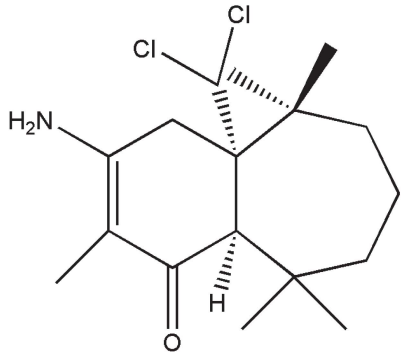
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{16}H_{23}Cl_2NO$, crystallizes in the monoclinic space group $P2_1$ with two independent molecules (*A* and *B*) in the asymmetric unit. They have essentially the same conformation. Each molecule is built up from fused six- and seven-membered rings and an additional three-membered ring. The six-membered ring has an envelope conformation, with the C atom belonging to the three-membered ring forming the flap, while the seven-membered ring displays a boat conformation. In the crystal, molecules are linked into chains propagating along the *a*-axis direction by N—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

As part of our studies of the essential oil of the Atlas cedar (*Cedrus atlantica*) made up mainly (50%) of β-himachalene (Benharref *et al.*, 2013; El Haib *et al.*, 2011), the reactivity of this sesquiterpene and its derivatives has been studied extensively by our team in order to prepare new products having biological properties (El Jamili *et al.*, 2002; Zaki *et al.*, 2014; Benharref *et al.*, 2015, 2016). Indeed, these compounds have been tested, using the food-poisoning technique, for their potential antifungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004).

The asymmetric unit (Fig. 1) includes two crystallographically independent molecules (*A* and *B*). A least-squares overlay of the two molecules (Spek, 2009) is shown in Fig. 2 and reveals that there is almost no difference between them. In both molecules, the six-membered rings has an envelope conformation as indicated by the total puckering amplitude Q_T = 0.438 (3) Å and spherical polar angle $\theta 2$ = 121.6 (4)° with

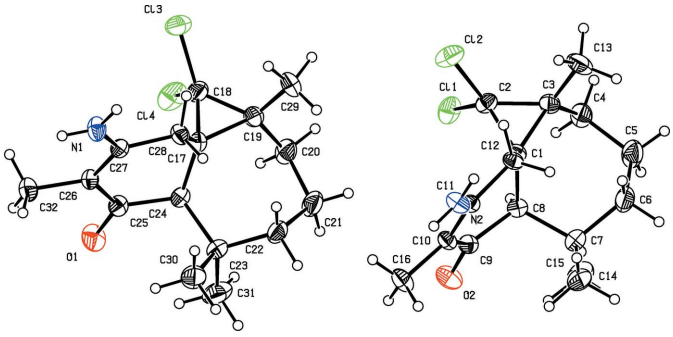


Figure 1
The molecular structure of the title compound, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

$\varphi 2 = -164.2\ (5)^\circ$, whereas the seven-membered ring displays a boat conformation with $Q_T = 1.133\ (3)$ Å, $\theta 2 = 88.37\ (17)^\circ$; $\varphi 2 = -49.66\ (16)^\circ$ and $\varphi 3 = -86.92\ (5)$.

In the crystal, molecules are linked *via* N—H···O hydrogen bonds (Table 1), forming chains along [100] as shown in Fig. 3. Owing to the presence of Cl atoms, the absolute configuration could be determined, by refining the Flack parameter as C1(S), C3(*R*), C8(*R*) and C17(S), C19(*R*), C24(*R*), respectively.

Synthesis and crystallization

To a solution of (1*S*,3*R*,8*R*)-2,2-dichloro-3,7,7,10-tetramethyltricyclo [6. 4.0.0^{1,3}]dodec-9-en-11-one (1 g, 3.32 mmol) (Benharref *et al.*, 2010) in 20 ml of trifuoroacetic acid at 10°C was added with stirring (500 mg, 7, 8 mmol) of NaN_3. After being stirred at room temperature for 24 h, the mixture was neutralized with Na_2CO_3 and extracted with diethyl ether

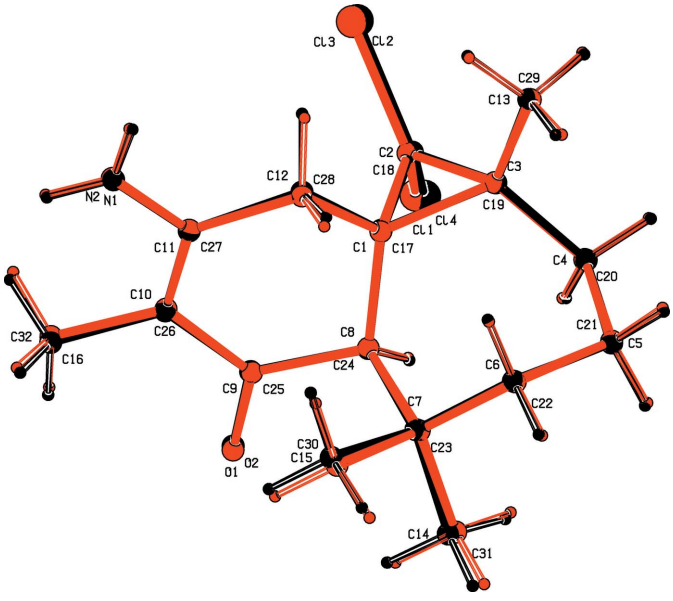


Figure 2
The AutoMolFit (*PLATON*; Spek, 2009) of molecule *B* (red) on molecule *A* (black).

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O1^{i}	0.86	2.00	2.816 (3)	159
N2—H2*B*···O2^{i}	0.86	2.06	2.873 (3)	159

Symmetry code: (i) $x-1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{23}Cl_2NO$
M_r	316.25
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	296
a, *b*, *c* (Å)	6.9962 (13), 18.654 (4), 12.426 (3)
β (°)	91.273 (5)
V (Å^3)	1621.4 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.40
Crystal size (mm)	0.24 × 0.2 × 0.15
Data collection	
Diffractometer	Bruker X8 *APEX*
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.629, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	58110, 8375, 6511
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.094, 1.02
No. of reflections	8375
No. of parameters	369
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.18
Absolute structure	Flack *x* determined using 2672 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.010 (18)

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

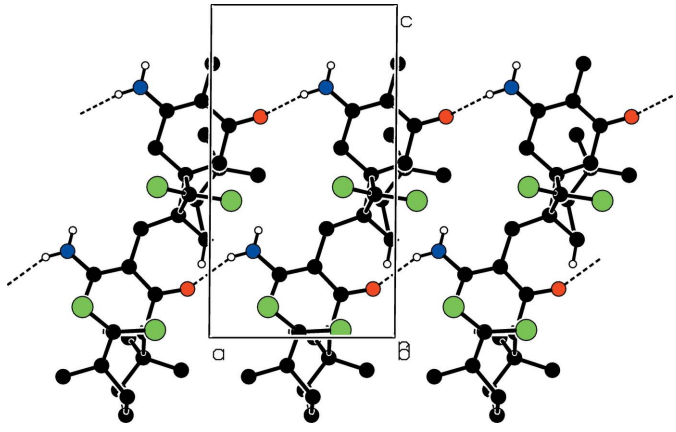


Figure 3
Crystal structure projection along the *b* axis of the title compound, showing molecules linked by N—H···O hydrogen bonds, forming chains along [100].

solution. The combined organic phases were dried on sodium sulfate and concentrated at reduced pressure to give the crude product, which was chromatographed on a silica gel column(cyclohexane–ether, 20–80 by volume) to give 630 mg (2 mmol) of the title product, which was recrystallized from diethyl ether.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170255 [https://doi.org/10.1107/S2414314617002553]

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(1*S*,2*R*,8*R*)-11-Amino-2,2-dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodec-10-en-9-one

Crystal data

$C_{16}H_{23}Cl_2NO$
M_r = 316.25
Monoclinic, $P2_1$
a = 6.9962 (13) Å
b = 18.654 (4) Å
c = 12.426 (3) Å
β = 91.273 (5)°
V = 1621.4 (5) Å^3
Z = 4
$F(000)$ = 672
D_x = 1.296 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6632 reflections
θ = 1.6–26.4°
μ = 0.40 mm^{-1}
T = 296 K
Prismatic, colourless
0.24 × 0.2 × 0.15 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.629, T_{max} = 0.747
58110 measured reflections
8375 independent reflections
6511 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 28.7°, θ_{min} = 2.7°
h = −9→8
k = −25→25
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.094
S = 1.02
8375 reflections
369 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.1774P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack x determined using 2672 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.010 (18)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3670 (4)	0.62609 (15)	0.5109 (2)	0.0342 (6)
C2	0.3868 (5)	0.69648 (17)	0.5700 (2)	0.0469 (7)
C3	0.3294 (5)	0.63016 (17)	0.6324 (2)	0.0452 (7)
C4	0.4790 (6)	0.59418 (18)	0.7054 (2)	0.0540 (8)
H4A	0.4594	0.6086	0.7793	0.065*
H4B	0.6054	0.6099	0.6852	0.065*
C5	0.4675 (6)	0.51212 (19)	0.6972 (3)	0.0610 (10)
H5A	0.5883	0.4923	0.7228	0.073*
H5B	0.3694	0.4955	0.7452	0.073*
C6	0.4241 (5)	0.48237 (17)	0.5853 (2)	0.0497 (8)
H6A	0.2932	0.4951	0.5664	0.060*
H6B	0.4292	0.4305	0.5902	0.060*
C7	0.5494 (4)	0.50495 (15)	0.4912 (2)	0.0419 (7)
C8	0.5481 (4)	0.58915 (14)	0.4760 (2)	0.0336 (6)
H8	0.6508	0.6076	0.5232	0.040*
C9	0.5984 (4)	0.61206 (16)	0.3623 (2)	0.0375 (6)
C10	0.4512 (4)	0.63564 (15)	0.2889 (2)	0.0345 (6)
C11	0.2637 (4)	0.63392 (14)	0.3179 (2)	0.0321 (5)
C12	0.2033 (4)	0.61729 (17)	0.4302 (2)	0.0374 (6)
H12A	0.1563	0.5684	0.4330	0.045*
H12B	0.0996	0.6490	0.4493	0.045*
C13	0.1274 (6)	0.6244 (2)	0.6731 (3)	0.0649 (10)
H13A	0.1181	0.6502	0.7396	0.097*
H13B	0.0963	0.5749	0.6846	0.097*
H13C	0.0397	0.6445	0.6208	0.097*
C14	0.7561 (6)	0.4808 (2)	0.5124 (4)	0.0719 (11)
H14A	0.7604	0.4294	0.5177	0.108*
H14B	0.8032	0.5014	0.5786	0.108*
H14C	0.8343	0.4962	0.4543	0.108*
C15	0.4721 (6)	0.46588 (19)	0.3904 (3)	0.0609 (9)
H15A	0.5429	0.4807	0.3290	0.091*
H15B	0.3395	0.4775	0.3791	0.091*
H15C	0.4857	0.4151	0.4002	0.091*
C16	0.5071 (5)	0.65700 (18)	0.1765 (2)	0.0463 (7)
H16A	0.4695	0.6200	0.1267	0.069*
H16B	0.6431	0.6636	0.1746	0.069*
H16C	0.4442	0.7010	0.1569	0.069*
C17	−0.0333 (4)	0.41311 (14)	−0.0173 (2)	0.0318 (6)
C18	−0.0172 (4)	0.49436 (15)	−0.0174 (2)	0.0393 (6)

C19	−0.0796 (4)	0.45709 (16)	0.0835 (2)	0.0415 (7)
C20	0.0609 (6)	0.45095 (18)	0.1779 (2)	0.0522 (8)
H20A	0.0326	0.4878	0.2303	0.063*
H20B	0.1892	0.4593	0.1528	0.063*
C21	0.0535 (7)	0.3772 (2)	0.2327 (3)	0.0652 (10)
H21A	0.1744	0.3691	0.2705	0.078*
H21B	−0.0455	0.3783	0.2861	0.078*
C22	0.0149 (6)	0.31356 (18)	0.1570 (3)	0.0562 (9)
H22A	−0.1151	0.3182	0.1292	0.067*
H22B	0.0194	0.2703	0.2004	0.067*
C23	0.1448 (5)	0.30144 (16)	0.0602 (2)	0.0447 (7)
C24	0.1500 (4)	0.36961 (14)	−0.0151 (2)	0.0324 (6)
H24	0.2501	0.4011	0.0148	0.039*
C25	0.2082 (4)	0.35152 (15)	−0.1298 (2)	0.0348 (6)
C26	0.0656 (4)	0.34250 (15)	−0.2124 (2)	0.0342 (6)
C27	−0.1239 (4)	0.35081 (14)	−0.1896 (2)	0.0306 (5)
C28	−0.1911 (4)	0.37905 (15)	−0.0838 (2)	0.0337 (6)
H28A	−0.2459	0.3400	−0.0433	0.040*
H28B	−0.2910	0.4142	−0.0971	0.040*
C29	−0.2869 (5)	0.4639 (2)	0.1167 (3)	0.0613 (9)
H29A	−0.3029	0.5074	0.1567	0.092*
H29B	−0.3201	0.4237	0.1608	0.092*
H29C	−0.3686	0.4648	0.0536	0.092*
C30	0.3499 (6)	0.2850 (3)	0.0979 (4)	0.0789 (13)
H30A	0.3518	0.2413	0.1386	0.118*
H30B	0.3970	0.3236	0.1423	0.118*
H30C	0.4296	0.2799	0.0365	0.118*
C31	0.0658 (6)	0.23478 (18)	−0.0001 (3)	0.0628 (10)
H31A	0.0671	0.1944	0.0478	0.094*
H31B	0.1441	0.2247	−0.0607	0.094*
H31C	−0.0629	0.2440	−0.0247	0.094*
C32	0.1267 (5)	0.31821 (19)	−0.3219 (2)	0.0491 (8)
H32A	0.0893	0.2692	−0.3330	0.074*
H32B	0.2631	0.3222	−0.3266	0.074*
H32C	0.0668	0.3478	−0.3762	0.074*
N1	0.1202 (4)	0.64687 (15)	0.24803 (19)	0.0442 (6)
H1A	0.1440	0.6567	0.1821	0.053*
H1B	0.0039	0.6453	0.2690	0.053*
N2	−0.2662 (3)	0.33465 (14)	−0.25976 (19)	0.0419 (6)
H2A	−0.2403	0.3182	−0.3224	0.050*
H2B	−0.3832	0.3407	−0.2420	0.050*
O1	0.7681 (3)	0.60721 (15)	0.3360 (2)	0.0583 (6)
O2	0.3815 (3)	0.34305 (14)	−0.14593 (19)	0.0532 (6)
Cl1	0.20775 (13)	0.53695 (4)	−0.02330 (7)	0.0559 (2)
Cl2	−0.18423 (14)	0.54562 (5)	−0.09233 (7)	0.0629 (2)
Cl3	0.61389 (17)	0.73707 (5)	0.58908 (8)	0.0714 (3)
Cl4	0.21570 (18)	0.76477 (5)	0.54750 (8)	0.0734 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0363 (15)	0.0378 (14)	0.0286 (12)	−0.0006 (11)	0.0015 (10)	0.0034 (10)
C2	0.064 (2)	0.0387 (16)	0.0383 (16)	0.0063 (14)	−0.0031 (14)	0.0014 (12)
C3	0.063 (2)	0.0415 (16)	0.0313 (14)	0.0066 (14)	0.0002 (13)	−0.0024 (12)
C4	0.081 (2)	0.0484 (18)	0.0322 (15)	0.0021 (17)	−0.0087 (15)	0.0000 (13)
C5	0.095 (3)	0.0488 (19)	0.0386 (17)	0.0041 (18)	−0.0074 (17)	0.0117 (14)
C6	0.066 (2)	0.0350 (15)	0.0483 (17)	−0.0053 (14)	0.0016 (15)	0.0064 (13)
C7	0.0438 (18)	0.0338 (14)	0.0480 (17)	−0.0002 (12)	−0.0011 (13)	0.0021 (12)
C8	0.0306 (15)	0.0324 (13)	0.0375 (14)	−0.0032 (11)	−0.0042 (11)	0.0023 (11)
C9	0.0280 (15)	0.0396 (15)	0.0450 (15)	−0.0056 (12)	0.0046 (12)	0.0011 (12)
C10	0.0317 (15)	0.0381 (14)	0.0339 (13)	−0.0042 (11)	0.0042 (11)	0.0034 (11)
C11	0.0319 (15)	0.0307 (13)	0.0337 (13)	−0.0025 (10)	0.0006 (11)	0.0022 (10)
C12	0.0305 (14)	0.0486 (16)	0.0334 (13)	0.0018 (12)	0.0043 (11)	0.0030 (11)
C13	0.082 (3)	0.072 (2)	0.0418 (18)	0.012 (2)	0.0242 (17)	0.0027 (17)
C14	0.055 (2)	0.050 (2)	0.111 (3)	0.0146 (17)	0.002 (2)	0.015 (2)
C15	0.086 (3)	0.0411 (18)	0.0554 (19)	−0.0044 (18)	0.0070 (18)	−0.0075 (15)
C16	0.0466 (19)	0.0526 (18)	0.0400 (16)	−0.0035 (14)	0.0112 (13)	0.0070 (13)
C17	0.0338 (15)	0.0302 (13)	0.0314 (13)	−0.0025 (11)	0.0028 (11)	−0.0043 (10)
C18	0.0471 (17)	0.0319 (14)	0.0385 (15)	−0.0022 (12)	−0.0060 (12)	−0.0032 (11)
C19	0.0534 (19)	0.0340 (14)	0.0374 (14)	−0.0024 (13)	0.0043 (12)	−0.0089 (12)
C20	0.079 (2)	0.0444 (17)	0.0331 (14)	−0.0099 (16)	−0.0053 (14)	−0.0059 (13)
C21	0.105 (3)	0.055 (2)	0.0358 (17)	−0.013 (2)	−0.0030 (18)	0.0026 (15)
C22	0.084 (3)	0.0430 (18)	0.0419 (17)	−0.0079 (17)	0.0071 (17)	0.0063 (13)
C23	0.0509 (19)	0.0364 (15)	0.0468 (17)	−0.0008 (13)	0.0002 (14)	0.0063 (13)
C24	0.0300 (14)	0.0326 (13)	0.0346 (13)	−0.0033 (11)	−0.0029 (11)	−0.0023 (10)
C25	0.0276 (15)	0.0336 (13)	0.0432 (15)	−0.0025 (11)	0.0049 (11)	−0.0008 (11)
C26	0.0316 (15)	0.0363 (14)	0.0351 (13)	−0.0040 (11)	0.0053 (11)	−0.0061 (11)
C27	0.0307 (14)	0.0290 (12)	0.0323 (12)	−0.0040 (10)	0.0011 (10)	−0.0028 (10)
C28	0.0252 (13)	0.0375 (14)	0.0386 (14)	−0.0015 (11)	0.0040 (11)	−0.0087 (11)
C29	0.067 (2)	0.059 (2)	0.059 (2)	0.0056 (19)	0.0206 (17)	−0.0196 (18)
C30	0.071 (3)	0.081 (3)	0.084 (3)	0.017 (2)	−0.016 (2)	0.030 (2)
C31	0.091 (3)	0.0307 (15)	0.067 (2)	0.0023 (17)	0.012 (2)	−0.0013 (15)
C32	0.0436 (18)	0.060 (2)	0.0441 (17)	−0.0082 (15)	0.0144 (14)	−0.0128 (15)
N1	0.0338 (14)	0.0620 (17)	0.0367 (13)	−0.0003 (12)	−0.0009 (10)	0.0118 (12)
N2	0.0331 (13)	0.0545 (15)	0.0379 (12)	−0.0010 (11)	−0.0001 (10)	−0.0134 (11)
O1	0.0245 (11)	0.0809 (18)	0.0700 (15)	−0.0016 (11)	0.0104 (10)	0.0140 (13)
O2	0.0244 (11)	0.0735 (16)	0.0620 (14)	0.0005 (10)	0.0066 (9)	−0.0087 (12)
Cl1	0.0660 (5)	0.0450 (4)	0.0565 (4)	−0.0231 (4)	−0.0068 (4)	0.0036 (4)
Cl2	0.0772 (6)	0.0421 (4)	0.0684 (5)	0.0142 (4)	−0.0204 (4)	−0.0017 (4)
Cl3	0.0953 (8)	0.0444 (4)	0.0734 (6)	−0.0183 (5)	−0.0224 (5)	−0.0053 (4)
Cl4	0.1074 (8)	0.0535 (5)	0.0593 (5)	0.0355 (5)	0.0064 (5)	0.0030 (4)

Geometric parameters (Å, º)

C1—C2	1.509 (4)	C17—C18	1.520 (4)
C1—C8	1.514 (4)	C17—C19	1.538 (4)

C1—C12	1.515 (4)	C18—C19	1.506 (4)
C1—C3	1.540 (4)	C18—Cl2	1.760 (3)
C2—C3	1.519 (4)	C18—Cl1	1.766 (3)
C2—Cl4	1.766 (3)	C19—C20	1.518 (4)
C2—Cl3	1.771 (4)	C19—C29	1.522 (5)
C3—C13	1.515 (5)	C20—C21	1.536 (5)
C3—C4	1.525 (4)	C20—H20A	0.9700
C4—C5	1.536 (5)	C20—H20B	0.9700
C4—H4A	0.9700	C21—C22	1.534 (5)
C4—H4B	0.9700	C21—H21A	0.9700
C5—C6	1.521 (4)	C21—H21B	0.9700
C5—H5A	0.9700	C22—C23	1.541 (5)
C5—H5B	0.9700	C22—H22A	0.9700
C6—C7	1.536 (4)	C22—H22B	0.9700
C6—H6A	0.9700	C23—C30	1.530 (5)
C6—H6B	0.9700	C23—C31	1.547 (5)
C7—C14	1.533 (5)	C23—C24	1.580 (4)
C7—C15	1.537 (5)	C24—C25	1.529 (4)
C7—C8	1.582 (4)	C24—H24	0.9800
C8—C9	1.524 (4)	C25—O2	1.243 (3)
C8—H8	0.9800	C25—C26	1.425 (4)
C9—O1	1.241 (3)	C26—C27	1.371 (4)
C9—C10	1.430 (4)	C26—C32	1.506 (4)
C10—C11	1.369 (4)	C27—N2	1.343 (3)
C10—C16	1.512 (4)	C27—C28	1.501 (3)
C11—N1	1.335 (3)	C28—H28A	0.9700
C11—C12	1.499 (4)	C28—H28B	0.9700
C12—H12A	0.9700	C29—H29A	0.9600
C12—H12B	0.9700	C29—H29B	0.9600
C13—H13A	0.9600	C29—H29C	0.9600
C13—H13B	0.9600	C30—H30A	0.9600
C13—H13C	0.9600	C30—H30B	0.9600
C14—H14A	0.9600	C30—H30C	0.9600
C14—H14B	0.9600	C31—H31A	0.9600
C14—H14C	0.9600	C31—H31B	0.9600
C15—H15A	0.9600	C31—H31C	0.9600
C15—H15B	0.9600	C32—H32A	0.9600
C15—H15C	0.9600	C32—H32B	0.9600
C16—H16A	0.9600	C32—H32C	0.9600
C16—H16B	0.9600	N1—H1A	0.8600
C16—H16C	0.9600	N1—H1B	0.8600
C17—C28	1.505 (4)	N2—H2A	0.8600
C17—C24	1.517 (4)	N2—H2B	0.8600

C2—C1—C8	117.9 (2)	C28—C17—C19	120.5 (2)
C2—C1—C12	118.4 (3)	C24—C17—C19	117.7 (2)
C8—C1—C12	112.7 (2)	C18—C17—C19	59.03 (18)
C2—C1—C3	59.76 (19)	C19—C18—C17	61.08 (18)

C8—C1—C3	117.8 (2)	C19—C18—Cl2	119.3 (2)
C12—C1—C3	120.8 (2)	C17—C18—Cl2	119.6 (2)
C1—C2—C3	61.13 (19)	C19—C18—Cl1	121.2 (2)
C1—C2—Cl4	119.8 (2)	C17—C18—Cl1	121.0 (2)
C3—C2—Cl4	118.8 (2)	Cl2—C18—Cl1	108.38 (16)
C1—C2—Cl3	120.6 (2)	C18—C19—C20	118.9 (3)
C3—C2—Cl3	121.7 (2)	C18—C19—C29	118.9 (3)
Cl4—C2—Cl3	108.40 (18)	C20—C19—C29	113.6 (3)
C13—C3—C2	119.1 (3)	C18—C19—C17	59.90 (17)
C13—C3—C4	113.8 (3)	C20—C19—C17	116.4 (3)
C2—C3—C4	118.4 (3)	C29—C19—C17	119.0 (3)
C13—C3—C1	120.3 (3)	C19—C20—C21	112.5 (3)
C2—C3—C1	59.10 (19)	C19—C20—H20A	109.1
C4—C3—C1	115.5 (3)	C21—C20—H20A	109.1
C3—C4—C5	111.4 (3)	C19—C20—H20B	109.1
C3—C4—H4A	109.3	C21—C20—H20B	109.1
C5—C4—H4A	109.3	H20A—C20—H20B	107.8
C3—C4—H4B	109.3	C22—C21—C20	115.4 (3)
C5—C4—H4B	109.3	C22—C21—H21A	108.4
H4A—C4—H4B	108.0	C20—C21—H21A	108.4
C6—C5—C4	115.7 (3)	C22—C21—H21B	108.4
C6—C5—H5A	108.4	C20—C21—H21B	108.4
C4—C5—H5A	108.4	H21A—C21—H21B	107.5
C6—C5—H5B	108.4	C21—C22—C23	119.5 (3)
C4—C5—H5B	108.4	C21—C22—H22A	107.4
H5A—C5—H5B	107.4	C23—C22—H22A	107.4
C5—C6—C7	119.3 (3)	C21—C22—H22B	107.4
C5—C6—H6A	107.5	C23—C22—H22B	107.4
C7—C6—H6A	107.5	H22A—C22—H22B	107.0
C5—C6—H6B	107.5	C30—C23—C22	110.8 (3)
C7—C6—H6B	107.5	C30—C23—C31	108.1 (3)
H6A—C6—H6B	107.0	C22—C23—C31	106.6 (3)
C14—C7—C6	109.9 (3)	C30—C23—C24	108.0 (3)
C14—C7—C15	108.3 (3)	C22—C23—C24	111.5 (3)
C6—C7—C15	107.1 (3)	C31—C23—C24	111.9 (3)
C14—C7—C8	108.4 (3)	C17—C24—C25	110.1 (2)
C6—C7—C8	111.2 (2)	C17—C24—C23	114.2 (2)
C15—C7—C8	111.9 (3)	C25—C24—C23	112.6 (2)
C1—C8—C9	110.5 (2)	C17—C24—H24	106.4
C1—C8—C7	114.8 (2)	C25—C24—H24	106.4
C9—C8—C7	112.9 (2)	C23—C24—H24	106.4
C1—C8—H8	106.0	O2—C25—C26	122.6 (2)
C9—C8—H8	106.0	O2—C25—C24	117.3 (2)
C7—C8—H8	106.0	C26—C25—C24	120.0 (2)
O1—C9—C10	122.3 (3)	C27—C26—C25	120.2 (2)
O1—C9—C8	117.7 (3)	C27—C26—C32	121.1 (2)
C10—C9—C8	120.0 (2)	C25—C26—C32	118.5 (2)
C11—C10—C9	120.4 (2)	N2—C27—C26	123.2 (2)

C11—C10—C16	121.2 (3)	N2—C27—C28	113.9 (2)
C9—C10—C16	118.2 (2)	C26—C27—C28	122.9 (2)
N1—C11—C10	122.4 (2)	C27—C28—C17	112.9 (2)
N1—C11—C12	114.8 (2)	C27—C28—H28A	109.0
C10—C11—C12	122.8 (2)	C17—C28—H28A	109.0
C11—C12—C1	111.7 (2)	C27—C28—H28B	109.0
C11—C12—H12A	109.3	C17—C28—H28B	109.0
C1—C12—H12A	109.3	H28A—C28—H28B	107.8
C11—C12—H12B	109.3	C19—C29—H29A	109.5
C1—C12—H12B	109.3	C19—C29—H29B	109.5
H12A—C12—H12B	107.9	H29A—C29—H29B	109.5
C3—C13—H13A	109.5	C19—C29—H29C	109.5
C3—C13—H13B	109.5	H29A—C29—H29C	109.5
H13A—C13—H13B	109.5	H29B—C29—H29C	109.5
C3—C13—H13C	109.5	C23—C30—H30A	109.5
H13A—C13—H13C	109.5	C23—C30—H30B	109.5
H13B—C13—H13C	109.5	H30A—C30—H30B	109.5
C7—C14—H14A	109.5	C23—C30—H30C	109.5
C7—C14—H14B	109.5	H30A—C30—H30C	109.5
H14A—C14—H14B	109.5	H30B—C30—H30C	109.5
C7—C14—H14C	109.5	C23—C31—H31A	109.5
H14A—C14—H14C	109.5	C23—C31—H31B	109.5
H14B—C14—H14C	109.5	H31A—C31—H31B	109.5
C7—C15—H15A	109.5	C23—C31—H31C	109.5
C7—C15—H15B	109.5	H31A—C31—H31C	109.5
H15A—C15—H15B	109.5	H31B—C31—H31C	109.5
C7—C15—H15C	109.5	C26—C32—H32A	109.5
H15A—C15—H15C	109.5	C26—C32—H32B	109.5
H15B—C15—H15C	109.5	H32A—C32—H32B	109.5
C10—C16—H16A	109.5	C26—C32—H32C	109.5
C10—C16—H16B	109.5	H32A—C32—H32C	109.5
H16A—C16—H16B	109.5	H32B—C32—H32C	109.5
C10—C16—H16C	109.5	C11—N1—H1A	120.0
H16A—C16—H16C	109.5	C11—N1—H1B	120.0
H16B—C16—H16C	109.5	H1A—N1—H1B	120.0
C28—C17—C24	113.2 (2)	C27—N2—H2A	120.0
C28—C17—C18	118.3 (2)	C27—N2—H2B	120.0
C24—C17—C18	118.1 (2)	H2A—N2—H2B	120.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O1^{i}	0.86	2.00	2.816 (3)	159
N2—H2*B*···O2^{i}	0.86	2.06	2.873 (3)	159

Symmetry code: (i) $x-1, y, z$.