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N′-[(1*S*,3*R*,8*R*)-2,2-Dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodecan-11-yl]acetohydrazide

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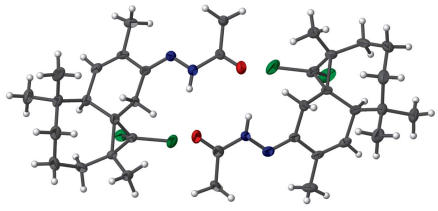
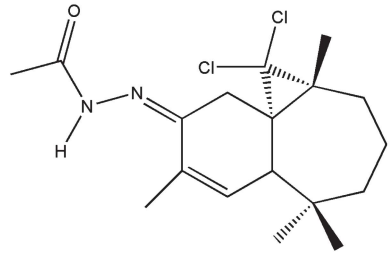
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{18}H_{26}Cl_2N_2O$, was synthesized in four steps from β-himachalene (3,5,5,9-tetramethyl-2,4a,5,6,7,8-hexahydro-1*H*-benzocycloheptene), which was isolated from an essential oil of the Atlas cedar (*Cedrus atlantica*). It crystallizes with two independent molecules in the asymmetric unit. Each molecule is built up from fused six- and seven-membered rings and an appended three-membered ring. An acetylhydrazone substituent is attached to the six-membered ring. In both molecules the six-membered rings display half-chair conformations, whereas the seven-membered rings have boat conformations. In the two molecules, the mean planes of the two rings are inclined to one another by 59.9 (3) and 59.1 (3)°. In the crystal, the two molecules are linked *via* N—H···O hydrogen bonds, forming dimers with an $R_2^2(8)$ loop. Within the dimer there are also C—H···O hydrogen bonds present. The dimers are linked *via* C—H···Cl hydrogen bonds, forming slabs parallel to the *ab* plane.

3D view



Chemical scheme



Structure description

The bicyclic sesquiterpene β-himachalene is the main constituent of the essential oil of the Atlas cedar (*Cedrus atlantica*) (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; Benharref *et al.*, 2015). These compounds have been tested, using the food-poisoning technique, for their potential antifungal activity against phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004). In this paper we present the crystal structure of the title compound, *N*′-[(1*S*,3*R*,8*R*)-2,2-dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodecan-11-yl]acetohydrazide.

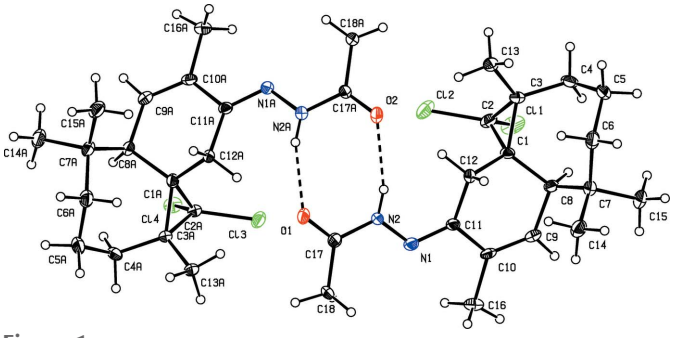


Figure 1
A view of the molecular structure of the two independent molecules of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

The title compound, Fig. 1, crystallizes with two independent molecules in the asymmetric unit. Each molecule is built up from fused six- and seven-membered rings and an appended three-membered ring. The six-membered rings each display a half-chair conformation, with puckering parameters of θ = 126.7 (7)° and $\varphi2$ = 169.4 (8) ° for the first molecule (C1/C8–C12), and θ = 53.4 (5)°, $\varphi2$ = 169.3 (7) ° for the second molecule (C1*A*/C8*A*–C12*A*). The seven-membered rings have boat conformations with θ = 88.2 (3) °, $\varphi2$ = −50.2 (3)° and $\varphi3$ = −93.40 (8)° for the first molecule (C1/C3–C8), and θ = 87.9 (3)°, $\varphi2$ =-50.6 (3)° and $\varphi3$ = −90.68 (8)° for the other molecule (C1*A*/C3*A*–C8*A*). In the first molecule, the mean planes of the two rings are inclined to one another by 59.9 (3)° [59.1 (3)° in the second molecule].

In the crystal, the two molecules are linked by two N—H···O hydrogen bonds, forming a dimer with an $R_2^2(8)$ ring motif (Fig. 2 and Table 1). Within the dimer there are also C—H···O hydrogen bonds present (Table 1). The dimers are linked *via* C—H···Cl hydrogen bonds, forming slabs lying parallel to the *ab* plane (Fig. 2 and Table 1).

Synthesis and crystallization

To a solution of an equimolecular quantity of (1*S*,3*R*,8*R*)-2,2-dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodecan-11-one (Ourhriss *et al.*, 2013) and thiosemicarbazide dissolved in ethanol, several drops of concentrated HCl were added. The reaction mixture was heated at reflux for 5 h and then evaporated under reduced pressure. The residue obtained was chromatographed on a silica gel column with a mixture of hexane and ethyl acetate as eluent (80/20). 1 g (2.7 mmol) of thiosemicarbazone was dissolved in 2 ml of pyridine and 2 ml of acetic anhydride. The mixture was heated at reflux during 1 h with magnetic stirring, and then evaporated under reduced pressure. Chromatography of the residue on silica (hexane/ethyl acetate, 90:10 *v*/*v*) allowed the isolation of the title compound with a yield of 90% (867 mg, 2.43 mmol). The product was recrystallized from ethyl acetate.

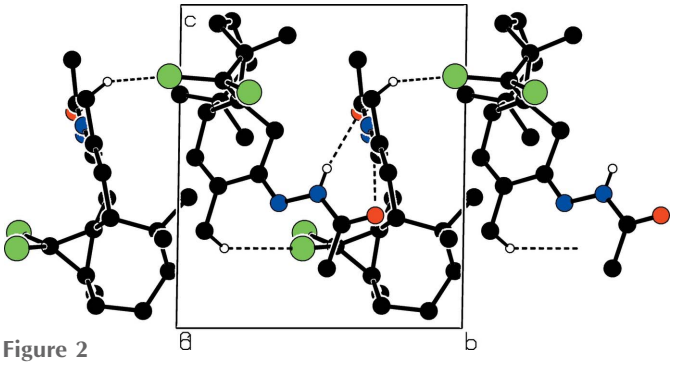


Figure 2
A partial view along the *a* axis of the crystal packing of the title compound, showing the N—H···O and C—H···Cl hydrogen bonds as dashed lines (see Table 1). H atoms not involved in these interactions have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	2.14	2.977 (6)	163
N2*A*—H2*A*···O1	0.86	2.14	2.968 (6)	163
C12—H12*B*···O2	0.97	2.41	3.315 (8)	154
C12*A*—H12*D*···O1	0.97	2.41	3.328 (8)	158
C16—H16*C*···Cl4^{i}	0.96	2.77	3.580 (7)	143
C16*A*—H16*F*···Cl1^{ii}	0.96	2.77	3.604 (6)	146

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y+1, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{26}Cl_2N_2O$
M_r	357.33
Crystal system, space group	Triclinic, *P*1
Temperature (K)	173
a, *b*, *c* (Å)	9.9172 (5), 10.0010 (5), 10.4124 (5)
α, β, γ (°)	86.863 (4), 84.173 (4), 66.037 (4)
V (Å^3)	938.73 (9)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.35
Crystal size (mm)	0.45 × 0.2 × 0.15
Data collection	
Diffractometer	Agilent Xcalibur (Eos, Gemini ultra)
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.785, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15916, 6506, 6259
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.053, 0.146, 1.06
No. of reflections	6506
No. of parameters	425
No. of restraints	3
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.09, −0.29
Absolute structure	Flack *x* determined using 2891 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.00 (3)

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SIR97* (Altomare *et al.*, 1999), *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Owing to the presence of the Cl atoms, the absolute configuration of the molecules in the crystal was confirmed by resonant scattering to be C1(*S*),C3(*R*),C8(*R*).

Acknowledgements

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References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Abingdon, England.

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Benharref, A., El Ammari, L., Saadi, M. & Berraho, M. (2015). *Acta Cryst.* E**71**, o284–o285.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Daoubi, M., Durán-Patrón, R., Hmamouchi, M., Hernández-Galán, R., Benharref, A. & Collado, I. G. (2004). *Pest. Manag. Sci.* **60**, 927–932.

El Haib, A., Benharref, A., Parrès-Maynadié, S., Manoury, E., Urrutigoïty, M. & Gouygou, M. (2011). *Tetrahedron Asymmetry*, **22**, 101–108.

El Jamili, H., Auhmani, A., Dakir, M., Lassaba, E., Benharref, A., Pierrot, M., Chiaroni, A. & Riche, C. (2002). *Tetrahedron Lett.* **43**, 6645–6648.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Ourhriss, N., Benharref, A., Oukhrib, A., Daran, J.-C. & Berraho, M. (2013). *Acta Cryst.* E**69**, o830.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

full crystallographic data

IUCrData (2016). **1**, x160554 [doi:10.1107/S241431461600554X]

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Crystal data

$C_{18}H_{26}Cl_2N_2O$
M_r = 357.33
Triclinic, $P\overline{1}$
a = 9.9172 (5) Å
b = 10.0010 (5) Å
c = 10.4124 (5) Å
α = 86.863 (4)°
β = 84.173 (4)°
γ = 66.037 (4)°
V = 938.73 (9) Å^3
Z = 2
$F(000)$ = 380
D_x = 1.264 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6506 reflections
θ = 3–25°
μ = 0.35 mm^{-1}
T = 173 K
Needle, colourless
0.45 × 0.2 × 0.15 mm

Data collection

Agilent Xcalibur (Eos, Gemini ultra) diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1978 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.785, T_{max} = 1.000
15916 measured reflections
6506 independent reflections
6259 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.0°, θ_{min} = 3.0°
h = −11→11
k = −11→11
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.146
S = 1.06
6506 reflections
425 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0973P)^2 + 0.5962P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Flack x determined using 2891 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.00 (3)

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}
Cl1	0.52732 (18)	−0.03725 (16)	0.77911 (17)	0.0499 (4)
Cl2	0.53714 (16)	0.24288 (17)	0.72961 (16)	0.0452 (4)
O1	0.3707 (5)	0.6906 (5)	0.3487 (4)	0.0372 (9)
N1	0.3445 (5)	0.3523 (5)	0.3868 (4)	0.0281 (10)
N2	0.3379 (5)	0.4884 (5)	0.4154 (4)	0.0278 (9)
H2	0.3151	0.5190	0.4935	0.033*
C1	0.2723 (6)	0.2065 (5)	0.7052 (5)	0.0229 (10)
C2	0.4178 (6)	0.1518 (6)	0.7655 (5)	0.0321 (12)
C3	0.2756 (6)	0.2247 (6)	0.8506 (5)	0.0298 (11)
C4	0.2213 (9)	0.1289 (7)	0.9419 (5)	0.0435 (16)
H4A	0.2683	0.0286	0.9127	0.052*
H4B	0.2508	0.1311	1.0275	0.052*
C5	0.0536 (9)	0.1785 (8)	0.9497 (6)	0.0490 (18)
H5A	0.0089	0.2542	1.0140	0.059*
H5B	0.0296	0.0964	0.9790	0.059*
C6	−0.0162 (8)	0.2381 (7)	0.8212 (6)	0.0425 (14)
H6A	−0.1214	0.2614	0.8362	0.051*
H6B	−0.0059	0.3295	0.8008	0.051*
C7	0.0426 (7)	0.1424 (6)	0.7007 (5)	0.0338 (12)
C8	0.2149 (6)	0.0950 (6)	0.6676 (5)	0.0268 (11)
H8	0.2646	0.0056	0.7180	0.032*
C9	0.2637 (6)	0.0570 (6)	0.5276 (5)	0.0278 (11)
H9	0.2646	−0.0302	0.4998	0.033*
C10	0.3061 (5)	0.1389 (6)	0.4399 (5)	0.0256 (10)
C11	0.3026 (5)	0.2807 (6)	0.4764 (5)	0.0239 (10)
C12	0.2408 (6)	0.3346 (6)	0.6115 (5)	0.0252 (10)
H12A	0.1347	0.3910	0.6129	0.030*
H12B	0.2854	0.3979	0.6374	0.030*
C13	0.2407 (7)	0.3735 (6)	0.9055 (5)	0.0358 (13)
H13A	0.2908	0.3615	0.9822	0.054*
H13B	0.1357	0.4229	0.9262	0.054*
H13C	0.2733	0.4305	0.8428	0.054*
C14	−0.0476 (7)	0.2294 (7)	0.5903 (6)	0.0384 (13)
H14A	−0.0117	0.1743	0.5123	0.058*
H14B	−0.0376	0.3209	0.5783	0.058*
H14C	−0.1500	0.2476	0.6112	0.058*
C15	0.0164 (8)	0.0004 (8)	0.7230 (7)	0.0468 (16)
H15A	0.0766	−0.0586	0.7884	0.070*
H15B	0.0425	−0.0532	0.6440	0.070*

H15C	−0.0861	0.0247	0.7503	0.070*
C16	0.3500 (7)	0.0933 (7)	0.3009 (5)	0.0341 (12)
H16A	0.3495	−0.0014	0.2912	0.051*
H16B	0.4476	0.0891	0.2762	0.051*
H16C	0.2809	0.1633	0.2468	0.051*
C17	0.3672 (6)	0.5734 (7)	0.3206 (5)	0.0331 (12)
C18	0.3901 (9)	0.5238 (10)	0.1849 (5)	0.0508 (19)
H18A	0.4160	0.5908	0.1288	0.076*
H18B	0.3006	0.5209	0.1606	0.076*
H18C	0.4686	0.4279	0.1777	0.076*
Cl3	0.75890 (15)	0.44374 (14)	0.29511 (13)	0.0356 (3)
Cl4	1.05043 (14)	0.43749 (14)	0.24643 (13)	0.0347 (3)
O2	0.2944 (5)	0.6314 (5)	0.6686 (4)	0.0369 (9)
N1A	0.6333 (5)	0.6647 (5)	0.6269 (4)	0.0252 (9)
N2A	0.4988 (5)	0.6664 (5)	0.5988 (4)	0.0287 (9)
H2A	0.4728	0.6844	0.5213	0.034*
C1A	0.8036 (5)	0.7056 (5)	0.3058 (5)	0.0233 (10)
C2A	0.8595 (6)	0.5531 (6)	0.2536 (5)	0.0252 (10)
C3A	0.7986 (6)	0.6809 (6)	0.1622 (5)	0.0256 (10)
C4A	0.9069 (6)	0.7168 (6)	0.0693 (5)	0.0314 (12)
H4A1	0.9118	0.6757	−0.0142	0.038*
H4A2	1.0046	0.6715	0.1005	0.038*
C5A	0.8634 (7)	0.8827 (7)	0.0531 (6)	0.0393 (14)
H5A1	0.9512	0.8990	0.0225	0.047*
H5A2	0.7941	0.9198	−0.0127	0.047*
C6A	0.7933 (7)	0.9705 (7)	0.1763 (6)	0.0361 (13)
H6A1	0.6977	0.9660	0.1973	0.043*
H6A2	0.7746	1.0719	0.1556	0.043*
C7A	0.8758 (6)	0.9293 (6)	0.3003 (5)	0.0306 (11)
C8A	0.9160 (6)	0.7635 (6)	0.3408 (5)	0.0243 (10)
H8A	1.0101	0.7058	0.2920	0.029*
C9A	0.9407 (6)	0.7329 (6)	0.4811 (5)	0.0268 (11)
H9A	1.0256	0.7365	0.5084	0.032*
C10A	0.8511 (6)	0.7011 (5)	0.5701 (5)	0.0256 (10)
C11A	0.7098 (6)	0.7009 (5)	0.5359 (5)	0.0230 (10)
C12A	0.6664 (5)	0.7511 (5)	0.4015 (4)	0.0222 (10)
H12C	0.6137	0.8568	0.3993	0.027*
H12D	0.6007	0.7085	0.3772	0.027*
C13A	0.6534 (6)	0.7120 (7)	0.1061 (5)	0.0314 (12)
H13D	0.6178	0.8078	0.0667	0.047*
H13E	0.5819	0.7076	0.1737	0.047*
H13F	0.6687	0.6403	0.0423	0.047*
C14A	1.0227 (7)	0.9487 (7)	0.2765 (7)	0.0423 (14)
H14D	1.0024	1.0495	0.2563	0.063*
H14E	1.0820	0.8889	0.2057	0.063*
H14F	1.0752	0.9198	0.3527	0.063*
C15A	0.7790 (7)	1.0353 (6)	0.4049 (6)	0.0379 (13)
H15D	0.8310	1.0175	0.4811	0.057*

H15E	0.6889	1.0211	0.4247	0.057*
H15F	0.7558	1.1340	0.3749	0.057*
C16A	0.8864 (6)	0.6713 (6)	0.7088 (5)	0.0307 (11)
H16D	0.9812	0.6731	0.7174	0.046*
H16E	0.8889	0.5769	0.7351	0.046*
H16F	0.8117	0.7451	0.7624	0.046*
C17A	0.4081 (7)	0.6395 (6)	0.6944 (5)	0.0306 (12)
C18A	0.4541 (9)	0.6207 (9)	0.8302 (5)	0.0500 (18)
H18D	0.3766	0.6128	0.8892	0.075*
H18E	0.4719	0.7038	0.8519	0.075*
H18F	0.5430	0.5335	0.8360	0.075*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0515 (9)	0.0321 (7)	0.0603 (10)	−0.0059 (7)	−0.0253 (8)	−0.0053 (7)
Cl2	0.0364 (8)	0.0523 (9)	0.0568 (9)	−0.0263 (7)	−0.0037 (6)	−0.0166 (7)
O1	0.043 (2)	0.054 (3)	0.032 (2)	−0.038 (2)	−0.0114 (17)	0.0112 (18)
N1	0.026 (2)	0.040 (3)	0.023 (2)	−0.017 (2)	−0.0027 (17)	−0.0044 (18)
N2	0.034 (2)	0.044 (3)	0.017 (2)	−0.028 (2)	−0.0016 (17)	0.0007 (18)
C1	0.032 (3)	0.019 (2)	0.017 (2)	−0.010 (2)	−0.0020 (19)	−0.0036 (18)
C2	0.039 (3)	0.030 (3)	0.032 (3)	−0.017 (2)	−0.009 (2)	−0.006 (2)
C3	0.041 (3)	0.035 (3)	0.019 (2)	−0.020 (2)	−0.005 (2)	−0.002 (2)
C4	0.086 (5)	0.042 (3)	0.016 (2)	−0.039 (3)	−0.004 (3)	0.000 (2)
C5	0.084 (5)	0.063 (4)	0.023 (3)	−0.055 (4)	0.011 (3)	−0.008 (3)
C6	0.051 (4)	0.047 (3)	0.042 (3)	−0.034 (3)	0.010 (3)	−0.010 (3)
C7	0.043 (3)	0.039 (3)	0.030 (3)	−0.029 (3)	0.001 (2)	−0.002 (2)
C8	0.038 (3)	0.026 (3)	0.022 (3)	−0.017 (2)	−0.008 (2)	−0.0005 (19)
C9	0.032 (3)	0.028 (3)	0.027 (3)	−0.013 (2)	−0.008 (2)	−0.006 (2)
C10	0.024 (2)	0.034 (3)	0.022 (2)	−0.012 (2)	−0.0064 (19)	−0.008 (2)
C11	0.022 (2)	0.035 (3)	0.018 (2)	−0.014 (2)	−0.0031 (18)	−0.002 (2)
C12	0.032 (3)	0.027 (3)	0.023 (2)	−0.018 (2)	0.001 (2)	−0.001 (2)
C13	0.054 (4)	0.037 (3)	0.026 (3)	−0.028 (3)	0.001 (2)	−0.006 (2)
C14	0.034 (3)	0.045 (3)	0.044 (3)	−0.023 (3)	−0.005 (3)	−0.003 (3)
C15	0.066 (4)	0.049 (4)	0.045 (4)	−0.045 (4)	0.001 (3)	−0.003 (3)
C16	0.034 (3)	0.040 (3)	0.023 (3)	−0.008 (2)	−0.006 (2)	−0.008 (2)
C17	0.030 (3)	0.054 (4)	0.027 (3)	−0.028 (3)	−0.009 (2)	0.006 (2)
C18	0.074 (5)	0.096 (6)	0.017 (3)	−0.069 (5)	−0.005 (3)	0.009 (3)
Cl3	0.0456 (8)	0.0336 (7)	0.0393 (7)	−0.0267 (6)	−0.0121 (6)	0.0041 (5)
Cl4	0.0318 (7)	0.0338 (7)	0.0348 (7)	−0.0085 (5)	−0.0050 (5)	−0.0048 (5)
O2	0.047 (2)	0.045 (2)	0.032 (2)	−0.034 (2)	0.0073 (17)	−0.0058 (17)
N1A	0.035 (2)	0.026 (2)	0.021 (2)	−0.0181 (19)	−0.0035 (17)	0.0007 (16)
N2A	0.036 (2)	0.036 (2)	0.023 (2)	−0.024 (2)	−0.0012 (17)	0.0003 (17)
C1A	0.022 (2)	0.027 (2)	0.025 (3)	−0.013 (2)	−0.0037 (19)	0.0036 (19)
C2A	0.029 (3)	0.029 (3)	0.021 (2)	−0.015 (2)	−0.006 (2)	−0.001 (2)
C3A	0.032 (3)	0.033 (3)	0.018 (2)	−0.020 (2)	−0.004 (2)	−0.001 (2)
C4A	0.035 (3)	0.046 (3)	0.018 (2)	−0.021 (3)	−0.002 (2)	0.002 (2)
C5A	0.046 (3)	0.052 (4)	0.032 (3)	−0.033 (3)	−0.007 (2)	0.013 (3)

C6A	0.043 (3)	0.037 (3)	0.038 (3)	−0.025 (3)	−0.009 (3)	0.009 (2)
C7A	0.037 (3)	0.032 (3)	0.033 (3)	−0.023 (2)	−0.007 (2)	0.001 (2)
C8A	0.023 (2)	0.028 (3)	0.027 (3)	−0.015 (2)	−0.0019 (19)	−0.0014 (19)
C9A	0.027 (3)	0.029 (3)	0.028 (3)	−0.014 (2)	−0.007 (2)	−0.004 (2)
C10A	0.035 (3)	0.024 (2)	0.019 (2)	−0.011 (2)	−0.007 (2)	−0.0028 (18)
C11A	0.031 (3)	0.019 (2)	0.021 (2)	−0.011 (2)	−0.005 (2)	−0.0018 (18)
C12A	0.023 (2)	0.028 (3)	0.021 (2)	−0.016 (2)	−0.0034 (19)	0.0022 (19)
C13A	0.038 (3)	0.044 (3)	0.023 (3)	−0.026 (3)	−0.012 (2)	0.004 (2)
C14A	0.046 (4)	0.047 (4)	0.049 (4)	−0.034 (3)	−0.011 (3)	0.008 (3)
C15A	0.047 (3)	0.032 (3)	0.043 (3)	−0.023 (3)	−0.009 (3)	−0.003 (2)
C16A	0.035 (3)	0.034 (3)	0.023 (3)	−0.012 (2)	−0.008 (2)	−0.002 (2)
C17A	0.048 (3)	0.035 (3)	0.020 (2)	−0.029 (3)	0.004 (2)	−0.003 (2)
C18A	0.087 (5)	0.076 (5)	0.021 (3)	−0.069 (4)	0.001 (3)	−0.003 (3)

Geometric parameters (Å, º)

Cl1—C2	1.763 (6)	Cl3—C2A	1.768 (5)
Cl2—C2	1.766 (6)	Cl4—C2A	1.769 (5)
O1—C17	1.238 (8)	O2—C17A	1.220 (7)
N1—C11	1.283 (7)	N1A—C11A	1.282 (7)
N1—N2	1.383 (7)	N1A—N2A	1.387 (6)
N2—C17	1.353 (7)	N2A—C17A	1.363 (7)
N2—H2	0.8600	N2A—H2A	0.8600
C1—C2	1.510 (7)	C1A—C2A	1.507 (7)
C1—C12	1.513 (7)	C1A—C12A	1.524 (7)
C1—C8	1.527 (7)	C1A—C8A	1.529 (7)
C1—C3	1.540 (7)	C1A—C3A	1.538 (7)
C2—C3	1.512 (8)	C2A—C3A	1.504 (7)
C3—C13	1.515 (8)	C3A—C4A	1.510 (7)
C3—C4	1.526 (8)	C3A—C13A	1.516 (7)
C4—C5	1.525 (11)	C4A—C5A	1.539 (9)
C4—H4A	0.9700	C4A—H4A1	0.9700
C4—H4B	0.9700	C4A—H4A2	0.9700
C5—C6	1.546 (10)	C5A—C6A	1.531 (9)
C5—H5A	0.9700	C5A—H5A1	0.9700
C5—H5B	0.9700	C5A—H5A2	0.9700
C6—C7	1.536 (8)	C6A—C7A	1.545 (8)
C6—H6A	0.9700	C6A—H6A1	0.9700
C6—H6B	0.9700	C6A—H6A2	0.9700
C7—C14	1.531 (9)	C7A—C15A	1.523 (8)
C7—C15	1.546 (8)	C7A—C14A	1.540 (8)
C7—C8	1.582 (8)	C7A—C8A	1.584 (7)
C8—C9	1.506 (7)	C8A—C9A	1.501 (7)
C8—H8	0.9800	C8A—H8A	0.9800
C9—C10	1.342 (8)	C9A—C10A	1.333 (8)
C9—H9	0.9300	C9A—H9A	0.9300
C10—C11	1.474 (7)	C10A—C11A	1.482 (7)
C10—C16	1.508 (7)	C10A—C16A	1.507 (7)

C11—C12	1.512 (7)	C11A—C12A	1.506 (7)
C12—H12A	0.9700	C12A—H12C	0.9700
C12—H12B	0.9700	C12A—H12D	0.9700
C13—H13A	0.9600	C13A—H13D	0.9600
C13—H13B	0.9600	C13A—H13E	0.9600
C13—H13C	0.9600	C13A—H13F	0.9600
C14—H14A	0.9600	C14A—H14D	0.9600
C14—H14B	0.9600	C14A—H14E	0.9600
C14—H14C	0.9600	C14A—H14F	0.9600
C15—H15A	0.9600	C15A—H15D	0.9600
C15—H15B	0.9600	C15A—H15E	0.9600
C15—H15C	0.9600	C15A—H15F	0.9600
C16—H16A	0.9600	C16A—H16D	0.9600
C16—H16B	0.9600	C16A—H16E	0.9600
C16—H16C	0.9600	C16A—H16F	0.9600
C17—C18	1.488 (8)	C17A—C18A	1.509 (8)
C18—H18A	0.9600	C18A—H18D	0.9600
C18—H18B	0.9600	C18A—H18E	0.9600
C18—H18C	0.9600	C18A—H18F	0.9600
C11—N1—N2	118.3 (4)	C11A—N1A—N2A	117.5 (4)
C17—N2—N1	120.1 (4)	C17A—N2A—N1A	119.9 (4)
C17—N2—H2	119.9	C17A—N2A—H2A	120.1
N1—N2—H2	119.9	N1A—N2A—H2A	120.1
C2—C1—C12	116.9 (5)	C2A—C1A—C12A	116.1 (4)
C2—C1—C8	118.6 (4)	C2A—C1A—C8A	118.8 (4)
C12—C1—C8	113.5 (4)	C12A—C1A—C8A	112.7 (4)
C2—C1—C3	59.4 (4)	C2A—C1A—C3A	59.2 (3)
C12—C1—C3	121.4 (4)	C12A—C1A—C3A	122.4 (4)
C8—C1—C3	116.7 (4)	C8A—C1A—C3A	117.6 (4)
C1—C2—C3	61.3 (4)	C3A—C2A—C1A	61.4 (3)
C1—C2—Cl1	120.8 (4)	C3A—C2A—Cl3	119.3 (4)
C3—C2—Cl1	121.4 (4)	C1A—C2A—Cl3	119.9 (4)
C1—C2—Cl2	119.4 (4)	C3A—C2A—Cl4	121.9 (4)
C3—C2—Cl2	119.6 (4)	C1A—C2A—Cl4	120.5 (4)
Cl1—C2—Cl2	108.2 (3)	Cl3—C2A—Cl4	107.9 (3)
C2—C3—C13	118.9 (5)	C2A—C3A—C4A	118.1 (5)
C2—C3—C4	118.5 (5)	C2A—C3A—C13A	119.3 (4)
C13—C3—C4	113.0 (5)	C4A—C3A—C13A	113.0 (4)
C2—C3—C1	59.3 (3)	C2A—C3A—C1A	59.4 (3)
C13—C3—C1	120.8 (5)	C4A—C3A—C1A	115.9 (4)
C4—C3—C1	116.4 (5)	C13A—C3A—C1A	121.3 (4)
C5—C4—C3	112.6 (5)	C3A—C4A—C5A	112.5 (5)
C5—C4—H4A	109.1	C3A—C4A—H4A1	109.1
C3—C4—H4A	109.1	C5A—C4A—H4A1	109.1
C5—C4—H4B	109.1	C3A—C4A—H4A2	109.1
C3—C4—H4B	109.1	C5A—C4A—H4A2	109.1
H4A—C4—H4B	107.8	H4A1—C4A—H4A2	107.8

C4—C5—C6	114.7 (5)	C6A—C5A—C4A	114.6 (5)
C4—C5—H5A	108.6	C6A—C5A—H5A1	108.6
C6—C5—H5A	108.6	C4A—C5A—H5A1	108.6
C4—C5—H5B	108.6	C6A—C5A—H5A2	108.6
C6—C5—H5B	108.6	C4A—C5A—H5A2	108.6
H5A—C5—H5B	107.6	H5A1—C5A—H5A2	107.6
C7—C6—C5	119.0 (6)	C5A—C6A—C7A	120.0 (5)
C7—C6—H6A	107.6	C5A—C6A—H6A1	107.3
C5—C6—H6A	107.6	C7A—C6A—H6A1	107.3
C7—C6—H6B	107.6	C5A—C6A—H6A2	107.3
C5—C6—H6B	107.6	C7A—C6A—H6A2	107.3
H6A—C6—H6B	107.0	H6A1—C6A—H6A2	106.9
C14—C7—C6	107.0 (5)	C15A—C7A—C14A	108.1 (5)
C14—C7—C15	108.1 (5)	C15A—C7A—C6A	107.6 (5)
C6—C7—C15	110.0 (5)	C14A—C7A—C6A	109.7 (5)
C14—C7—C8	112.4 (4)	C15A—C7A—C8A	112.8 (4)
C6—C7—C8	112.2 (5)	C14A—C7A—C8A	107.1 (5)
C15—C7—C8	107.0 (5)	C6A—C7A—C8A	111.5 (4)
C9—C8—C1	109.0 (4)	C9A—C8A—C1A	109.4 (4)
C9—C8—C7	112.9 (4)	C9A—C8A—C7A	113.2 (4)
C1—C8—C7	114.2 (4)	C1A—C8A—C7A	113.8 (4)
C9—C8—H8	106.8	C9A—C8A—H8A	106.7
C1—C8—H8	106.8	C1A—C8A—H8A	106.7
C7—C8—H8	106.8	C7A—C8A—H8A	106.7
C10—C9—C8	125.3 (5)	C10A—C9A—C8A	125.5 (5)
C10—C9—H9	117.4	C10A—C9A—H9A	117.3
C8—C9—H9	117.4	C8A—C9A—H9A	117.3
C9—C10—C11	120.2 (4)	C9A—C10A—C11A	120.6 (5)
C9—C10—C16	121.4 (5)	C9A—C10A—C16A	121.4 (5)
C11—C10—C16	118.3 (5)	C11A—C10A—C16A	118.0 (5)
N1—C11—C10	116.3 (4)	N1A—C11A—C10A	116.0 (4)
N1—C11—C12	125.4 (5)	N1A—C11A—C12A	126.7 (5)
C10—C11—C12	118.2 (4)	C10A—C11A—C12A	117.2 (4)
C11—C12—C1	110.2 (4)	C11A—C12A—C1A	110.3 (4)
C11—C12—H12A	109.6	C11A—C12A—H12C	109.6
C1—C12—H12A	109.6	C1A—C12A—H12C	109.6
C11—C12—H12B	109.6	C11A—C12A—H12D	109.6
C1—C12—H12B	109.6	C1A—C12A—H12D	109.6
H12A—C12—H12B	108.1	H12C—C12A—H12D	108.1
C3—C13—H13A	109.5	C3A—C13A—H13D	109.5
C3—C13—H13B	109.5	C3A—C13A—H13E	109.5
H13A—C13—H13B	109.5	H13D—C13A—H13E	109.5
C3—C13—H13C	109.5	C3A—C13A—H13F	109.5
H13A—C13—H13C	109.5	H13D—C13A—H13F	109.5
H13B—C13—H13C	109.5	H13E—C13A—H13F	109.5
C7—C14—H14A	109.5	C7A—C14A—H14D	109.5
C7—C14—H14B	109.5	C7A—C14A—H14E	109.5
H14A—C14—H14B	109.5	H14D—C14A—H14E	109.5

C7—C14—H14C	109.5	C7A—C14A—H14F	109.5
H14A—C14—H14C	109.5	H14D—C14A—H14F	109.5
H14B—C14—H14C	109.5	H14E—C14A—H14F	109.5
C7—C15—H15A	109.5	C7A—C15A—H15D	109.5
C7—C15—H15B	109.5	C7A—C15A—H15E	109.5
H15A—C15—H15B	109.5	H15D—C15A—H15E	109.5
C7—C15—H15C	109.5	C7A—C15A—H15F	109.5
H15A—C15—H15C	109.5	H15D—C15A—H15F	109.5
H15B—C15—H15C	109.5	H15E—C15A—H15F	109.5
C10—C16—H16A	109.5	C10A—C16A—H16D	109.5
C10—C16—H16B	109.5	C10A—C16A—H16E	109.5
H16A—C16—H16B	109.5	H16D—C16A—H16E	109.5
C10—C16—H16C	109.5	C10A—C16A—H16F	109.5
H16A—C16—H16C	109.5	H16D—C16A—H16F	109.5
H16B—C16—H16C	109.5	H16E—C16A—H16F	109.5
O1—C17—N2	119.5 (5)	O2—C17A—N2A	120.1 (5)
O1—C17—C18	122.2 (5)	O2—C17A—C18A	122.4 (5)
N2—C17—C18	118.3 (5)	N2A—C17A—C18A	117.6 (5)
C17—C18—H18A	109.5	C17A—C18A—H18D	109.5
C17—C18—H18B	109.5	C17A—C18A—H18E	109.5
H18A—C18—H18B	109.5	H18D—C18A—H18E	109.5
C17—C18—H18C	109.5	C17A—C18A—H18F	109.5
H18A—C18—H18C	109.5	H18D—C18A—H18F	109.5
H18B—C18—H18C	109.5	H18E—C18A—H18F	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	2.14	2.977 (6)	163
N2*A*—H2*A*···O1	0.86	2.14	2.968 (6)	163
C12—H12*B*···O2	0.97	2.41	3.315 (8)	154
C12*A*—H12*D*···O1	0.97	2.41	3.328 (8)	158
C16—H16*C*···Cl4^{i}	0.96	2.77	3.580 (7)	143
C16*A*—H16*F*···Cl1^{ii}	0.96	2.77	3.604 (6)	146

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y+1, z$.