Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# *N*,*N*-Dimethyldehydroabietylammonium chloride ethanol monosolvate

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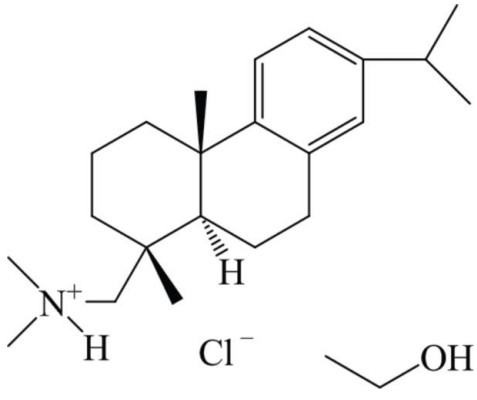
Received 25 April 2013; accepted 20 May 2013

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.008 Å; $R$ factor = 0.074; $wR$ factor = 0.190; data-to-parameter ratio = 17.9.

The title compound {systematic name: 1-[(1*R*,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl]-*N*,*N*-dimethylmethanaminium chloride ethanol monosolvate}, $C_{22}H_{36}N^+\cdot Cl^-\cdot C_2H_6O$, was synthesized from dehydroabietylamine by *N*-methylation with formaldehyde/formic acid and transformation into the hydrochloride. The dehydroabietyl moiety exhibits the usual conformation with the two cyclohexane rings in chair and half-chair conformations and a *trans*-ring junction. The crystal structure is built up from columns of the dehydroabietyl moieties stacked along the *a* axis. These columns are held together by the chloride ions *via* N—H···Cl and C—H···Cl interactions, which establish a two-dimensional network parallel to (010). The ethanol solvent molecules are located between the columns and anchored *via* O—H···Cl hydrogen bonds.

## Related literature

For the biological activity of dehydroabietylamine derivatives, see: Goodson *et al.* (1999); Rao *et al.* (2008); Wilkerson *et al.* (1993); For the crystal structures of dehydroabietic acid derivatives, see Rao *et al.* (2006, 2009).




## Experimental

### Crystal data

$C_{22}H_{36}N^+\cdot Cl^-\cdot C_2H_6O$
$M_r$ = 396.04
Monoclinic, $P2_1$
$a$ = 6.0560 (12) Å
$b$ = 10.963 (2) Å
$c$ = 18.554 (4) Å
$\beta$ = 98.62 (3)°
$V$ = 1217.9 (4) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.17 mm$^{-1}$
$T$ = 293 K
0.30 × 0.20 × 0.10 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.951, $T_{max}$ = 0.983
4924 measured reflections
4476 independent reflections
2605 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
3 standard reflections every 200 reflections
intensity decay: 1%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.190
$S$ = 0.99
4476 reflections
250 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Absolute structure: Flack (1983), 2102 Friedel pairs
Flack parameter: −0.02 (13)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N—H0*B*···Cl | 0.91 | 2.27 | 3.097 (4) | 152 |
| O—H0*A*···Cl | 0.82 | 2.27 | 3.092 (9) | 178 |
| C18—H18*B*···Cl$^{i}$ | 0.96 | 2.78 | 3.694 (6) | 160 |
| C18—H18*C*···Cl$^{ii}$ | 0.96 | 2.84 | 3.693 (6) | 149 |
| C2—H2*B*···Cl | 0.97 | 2.86 | 3.775 (5) | 158 |

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+1$; (ii) $x+1, y, z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This research was supported financially by grants from the National Forestry Bureau 948 (2012–4–13).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QK2059).

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# supplementary materials

*Acta Cryst.* (2013). E**69**, o959 [doi:10.1107/S1600536813013846]

## *N,N*-Dimethyldehydroabietylammonium chloride ethanol monosolvate

**Xiu-Zhi Huang, Xiao-Ping Rao and Yan-Jie Cui**

### Comment

Dehydroabietylamine is widely used as starting material for design and synthesis of biological compounds (Goodson *et al.*, 1999; Rao *et al.*, 2008; Wilkerson *et al.*, 1993). In continuation of previous investigations (Rao *et al.*, 2006, 2009) the title compound was studied. The overall geometry of dehydroabietyl moiety in the title compound is comparable to that found for dehydroabietic acid and related compounds (Rao *et al.*, 2009). There are three six-membered rings, which form planar, half-chair and chair conformations, respectively. The absolute structure of the title compound could be secured via anomalous dispersion effects (Flack parameter -0.02 (13)) and is in accordance with expectations. Thus the three chiral centers in the molecule have R–, S– and R–configurations, respectively.

### Experimental

Dehydroabietylamine (13.57 g, 0.05 mol, Hangzhou Wanjing Company), formaldehyde (11.95 g, 36%) and formic acid (12.83 g, 85%) were added to ethanol solution (13.6 g, 95%), the mixture was stirred for 4h at 60–70 °C to form N,N-dimethydehydroabietylamine. 4.0 ml concentrated hydrochloric acid was added to a solution (10.0 g, 0.032 mol) of N,N-dimethydehydroabietylamine in 50 ml water and the mixture was stirred for 4h at 60–70 °C. After cooling to room temperature, the solvent was distilled off under vacuum. Crystals were obtained by recrystallization from ethanol.

### Refinement

H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.96 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms, and C—H = 0.97–0.98 Å, N—H = 0.91 Å, O—H = 0.85 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C,N,H) for all other H atoms. Methyl groups were refined in orientation (AFIX 137 of program SHELXL97), O—H group was generated with AFIX 83.

### Computing details

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software* (Enraf–Nonius, 1989); data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

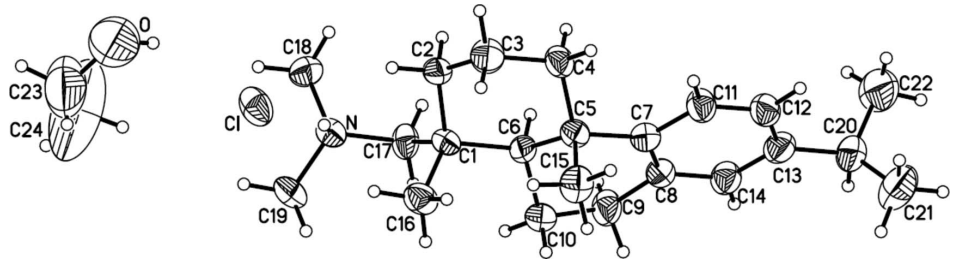


**Figure 1**

Molecular structure of the title compound, with H atoms represented by small spheres of arbitrary radius and displacement ellipsoids at the 30% probability level.

## 1-[(1*R*,4a*S*,10a*R*)-7-isopropyl-1,4a-dimethyl-1,2,3,4,4a,9,10,10a-octahydrophenanthren-1-yl]-*N*,*N*-dimethylmethanaminium chloride ethanol monosolvate

*Crystal data*

$C_{22}H_{36}N^+\cdot Cl^-\cdot C_2H_6O$
$M_r$ = 396.04
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a$ = 6.0560 (12) Å
$b$ = 10.963 (2) Å
$c$ = 18.554 (4) Å
$\beta$ = 98.62 (3)°
$V$ = 1217.9 (4) Å$^3$
$Z$ = 2
$F(000)$ = 436
$D_x$ = 1.080 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 25 reflections
$\theta$ = 9–13°
$\mu$ = 0.17 mm$^{-1}$
$T$ = 293 K
Block, white
0.30 × 0.20 × 0.10 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.951, $T_{max}$ = 0.983
4924 measured reflections
4476 independent reflections
2605 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 25.4°, $\theta_{min}$ = 1.1°
$h$ = 0→7
$k$ = −13→13
$l$ = −22→22
3 standard reflections every 200 reflections
intensity decay: 1%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.190
$S$ = 0.99
4476 reflections
250 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.080P)^2 + 0.5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$
Absolute structure: Flack (1983), 2102 Friedel pairs
Flack parameter: −0.02 (13)

*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.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl | 0.1469 (2) | 0.07595 (14) | 0.50514 (8) | 0.0890 (5) |
| C1 | 0.4016 (7) | 0.1919 (4) | 0.3221 (2) | 0.0511 (11) |
| C2 | 0.4933 (8) | 0.0669 (5) | 0.3522 (2) | 0.0642 (12) |
| H2A | 0.6522 | 0.0747 | 0.3689 | 0.077* |
| H2B | 0.4221 | 0.0456 | 0.3939 | 0.077* |
| C3 | 0.4563 (11) | −0.0354 (5) | 0.2971 (3) | 0.0810 (17) |
| H3A | 0.2973 | −0.0480 | 0.2826 | 0.097* |
| H3B | 0.5197 | −0.1103 | 0.3191 | 0.097* |
| C4 | 0.5646 (9) | −0.0053 (4) | 0.2303 (3) | 0.0669 (14) |
| H4A | 0.5429 | −0.0730 | 0.1963 | 0.080* |
| H4B | 0.7239 | 0.0056 | 0.2449 | 0.080* |
| C5 | 0.4646 (7) | 0.1118 (4) | 0.1920 (3) | 0.0554 (12) |
| C6 | 0.5008 (8) | 0.2166 (4) | 0.2491 (2) | 0.0567 (11) |
| H6A | 0.6628 | 0.2228 | 0.2635 | 0.068* |
| C7 | 0.5873 (8) | 0.1477 (5) | 0.1286 (3) | 0.0652 (13) |
| C8 | 0.6409 (8) | 0.2667 (5) | 0.1122 (3) | 0.0654 (13) |
| C9 | 0.5920 (11) | 0.3702 (5) | 0.1597 (3) | 0.0817 (17) |
| H9A | 0.5321 | 0.4376 | 0.1290 | 0.098* |
| H9B | 0.7310 | 0.3974 | 0.1881 | 0.098* |
| C10 | 0.4279 (9) | 0.3391 (5) | 0.2118 (3) | 0.0700 (14) |
| H10A | 0.2779 | 0.3322 | 0.1851 | 0.084* |
| H10B | 0.4284 | 0.4028 | 0.2481 | 0.084* |
| C11 | 0.6423 (9) | 0.0552 (5) | 0.0795 (3) | 0.0705 (14) |
| H11A | 0.6097 | −0.0259 | 0.0881 | 0.085* |
| C12 | 0.7413 (8) | 0.0836 (6) | 0.0204 (3) | 0.0741 (14) |
| H12A | 0.7769 | 0.0214 | −0.0099 | 0.089* |
| C13 | 0.7905 (9) | 0.2049 (6) | 0.0044 (3) | 0.0745 (15) |
| C14 | 0.7355 (9) | 0.2915 (5) | 0.0515 (3) | 0.0710 (14) |
| H14A | 0.7643 | 0.3726 | 0.0416 | 0.085* |
| C15 | 0.2191 (8) | 0.0887 (6) | 0.1565 (3) | 0.0806 (15) |
| H15A | 0.2172 | 0.0378 | 0.1144 | 0.121* |
| H15B | 0.1484 | 0.1652 | 0.1423 | 0.121* |
| H15C | 0.1400 | 0.0490 | 0.1911 | 0.121* |
| C16 | 0.1456 (8) | 0.1985 (5) | 0.3140 (3) | 0.0741 (14) |
| H16A | 0.1003 | 0.1992 | 0.3614 | 0.111* |
| H16B | 0.0821 | 0.1287 | 0.2872 | 0.111* |
| H16C | 0.0945 | 0.2716 | 0.2882 | 0.111* |

| | | | | |
|---|---|---|---|---|
| C17 | 0.5076 (9) | 0.2897 (4) | 0.3771 (2) | 0.0643 (13) |
| H17A | 0.6689 | 0.2834 | 0.3819 | 0.077* |
| H17B | 0.4655 | 0.3699 | 0.3577 | 0.077* |
| N | 0.4394 (6) | 0.2782 (3) | 0.4510 (2) | 0.0579 (10) |
| H0B | 0.3563 | 0.2090 | 0.4508 | 0.069* |
| C18 | 0.6406 (8) | 0.2623 (6) | 0.5085 (3) | 0.0756 (15) |
| H18A | 0.5928 | 0.2483 | 0.5549 | 0.113* |
| H18B | 0.7308 | 0.3346 | 0.5111 | 0.113* |
| H18C | 0.7265 | 0.1937 | 0.4963 | 0.113* |
| C19 | 0.3003 (9) | 0.3800 (5) | 0.4710 (3) | 0.0760 (15) |
| H19A | 0.1697 | 0.3878 | 0.4349 | 0.114* |
| H19B | 0.3847 | 0.4544 | 0.4733 | 0.114* |
| H19C | 0.2561 | 0.3640 | 0.5176 | 0.114* |
| C20 | 0.9025 (11) | 0.2391 (7) | −0.0617 (3) | 0.0890 (18) |
| H20A | 0.9040 | 0.3285 | −0.0630 | 0.107* |
| C21 | 0.7518 (13) | 0.1982 (8) | −0.1330 (4) | 0.124 (3) |
| H21A | 0.8099 | 0.2308 | −0.1744 | 0.186* |
| H21B | 0.6026 | 0.2277 | −0.1330 | 0.186* |
| H21C | 0.7499 | 0.1107 | −0.1357 | 0.186* |
| C22 | 1.1396 (12) | 0.2007 (9) | −0.0568 (4) | 0.136 (3) |
| H22A | 1.2276 | 0.2417 | −0.0167 | 0.204* |
| H22B | 1.1939 | 0.2214 | −0.1013 | 0.204* |
| H22C | 1.1503 | 0.1141 | −0.0494 | 0.204* |
| O | 0.1487 (19) | 0.0080 (8) | 0.6670 (5) | 0.246 (5) |
| H0A | 0.1446 | 0.0264 | 0.6239 | 0.295* |
| C23 | 0.117 (4) | 0.2011 (17) | 0.6937 (7) | 0.332 (14) |
| H23A | 0.0267 | 0.2620 | 0.7123 | 0.498* |
| H23B | 0.1269 | 0.2182 | 0.6436 | 0.498* |
| H23C | 0.2642 | 0.2020 | 0.7216 | 0.498* |
| C24 | 0.022 (3) | 0.0876 (13) | 0.6992 (7) | 0.255 (8) |
| H24B | 0.0224 | 0.0660 | 0.7499 | 0.306* |
| H24A | −0.1309 | 0.0870 | 0.6744 | 0.306* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl | 0.0935 (10) | 0.0739 (8) | 0.1121 (11) | −0.0040 (8) | 0.0564 (9) | 0.0053 (9) |
| C1 | 0.044 (2) | 0.047 (3) | 0.063 (3) | 0.003 (2) | 0.013 (2) | −0.001 (2) |
| C2 | 0.069 (3) | 0.062 (3) | 0.065 (3) | 0.017 (3) | 0.021 (2) | 0.015 (3) |
| C3 | 0.108 (5) | 0.055 (3) | 0.083 (4) | 0.004 (3) | 0.022 (4) | 0.001 (3) |
| C4 | 0.083 (4) | 0.055 (3) | 0.066 (3) | 0.006 (3) | 0.021 (3) | −0.006 (2) |
| C5 | 0.052 (3) | 0.055 (3) | 0.059 (3) | 0.000 (2) | 0.007 (2) | −0.003 (2) |
| C6 | 0.070 (3) | 0.050 (2) | 0.052 (2) | 0.009 (2) | 0.015 (2) | 0.005 (2) |
| C7 | 0.062 (3) | 0.072 (3) | 0.061 (3) | 0.010 (3) | 0.008 (3) | 0.003 (3) |
| C8 | 0.073 (3) | 0.064 (3) | 0.062 (3) | 0.010 (3) | 0.020 (3) | 0.013 (3) |
| C9 | 0.118 (5) | 0.062 (3) | 0.073 (3) | 0.006 (3) | 0.039 (3) | 0.011 (3) |
| C10 | 0.077 (4) | 0.064 (3) | 0.070 (3) | 0.017 (3) | 0.015 (3) | 0.007 (3) |
| C11 | 0.087 (4) | 0.061 (3) | 0.067 (3) | 0.001 (3) | 0.023 (3) | −0.004 (3) |
| C12 | 0.070 (3) | 0.089 (4) | 0.061 (3) | 0.007 (4) | 0.006 (3) | −0.013 (3) |
| C13 | 0.068 (3) | 0.088 (4) | 0.068 (3) | 0.003 (3) | 0.011 (3) | 0.020 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.077 (4) | 0.071 (3) | 0.067 (3) | −0.003 (3) | 0.016 (3) | 0.005 (3) |
| C15 | 0.071 (3) | 0.093 (4) | 0.076 (3) | −0.015 (3) | 0.007 (3) | −0.008 (3) |
| C16 | 0.053 (3) | 0.084 (4) | 0.084 (3) | 0.005 (3) | 0.007 (3) | −0.007 (3) |
| C17 | 0.078 (3) | 0.060 (3) | 0.061 (3) | −0.013 (3) | 0.028 (3) | 0.004 (2) |
| N | 0.053 (2) | 0.052 (2) | 0.070 (2) | 0.0007 (18) | 0.015 (2) | −0.0016 (19) |
| C18 | 0.067 (3) | 0.087 (4) | 0.070 (3) | 0.009 (3) | 0.000 (3) | 0.008 (3) |
| C19 | 0.070 (4) | 0.070 (3) | 0.091 (4) | 0.016 (3) | 0.022 (3) | −0.015 (3) |
| C20 | 0.094 (4) | 0.109 (5) | 0.069 (3) | 0.006 (4) | 0.026 (3) | 0.020 (3) |
| C21 | 0.113 (5) | 0.172 (8) | 0.081 (4) | −0.020 (5) | −0.001 (4) | 0.028 (5) |
| C22 | 0.099 (5) | 0.192 (9) | 0.124 (6) | 0.007 (6) | 0.043 (5) | 0.050 (6) |
| O | 0.380 (14) | 0.188 (7) | 0.170 (7) | 0.130 (8) | 0.042 (8) | 0.004 (6) |
| C23 | 0.54 (3) | 0.29 (2) | 0.171 (12) | −0.32 (2) | 0.062 (15) | −0.073 (12) |
| C24 | 0.45 (3) | 0.182 (13) | 0.162 (10) | 0.083 (18) | 0.139 (14) | 0.020 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C16 | 1.537 (6) | C15—H15B | 0.9600 |
| C1—C2 | 1.551 (6) | C15—H15C | 0.9600 |
| C1—C17 | 1.551 (6) | C16—H16A | 0.9600 |
| C1—C6 | 1.585 (6) | C16—H16B | 0.9600 |
| C2—C3 | 1.511 (7) | C16—H16C | 0.9600 |
| C2—H2A | 0.9700 | C17—N | 1.496 (5) |
| C2—H2B | 0.9700 | C17—H17A | 0.9700 |
| C3—C4 | 1.523 (7) | C17—H17B | 0.9700 |
| C3—H3A | 0.9700 | N—C19 | 1.479 (6) |
| C3—H3B | 0.9700 | N—C18 | 1.505 (6) |
| C4—C5 | 1.547 (6) | N—H0B | 0.9100 |
| C4—H4A | 0.9700 | C18—H18A | 0.9600 |
| C4—H4B | 0.9700 | C18—H18B | 0.9600 |
| C5—C7 | 1.533 (7) | C18—H18C | 0.9600 |
| C5—C15 | 1.554 (6) | C19—H19A | 0.9600 |
| C5—C6 | 1.556 (6) | C19—H19B | 0.9600 |
| C6—C10 | 1.544 (6) | C19—H19C | 0.9600 |
| C6—H6A | 0.9800 | C20—C22 | 1.486 (9) |
| C7—C8 | 1.390 (7) | C20—C21 | 1.557 (9) |
| C7—C11 | 1.436 (7) | C20—H20A | 0.9800 |
| C8—C14 | 1.365 (7) | C21—H21A | 0.9600 |
| C8—C9 | 1.494 (7) | C21—H21B | 0.9600 |
| C9—C10 | 1.524 (7) | C21—H21C | 0.9600 |
| C9—H9A | 0.9700 | C22—H22A | 0.9600 |
| C9—H9B | 0.9700 | C22—H22B | 0.9600 |
| C10—H10A | 0.9700 | C22—H22C | 0.9600 |
| C10—H10B | 0.9700 | O—C24 | 1.358 (13) |
| C11—C12 | 1.361 (7) | O—H0A | 0.8200 |
| C11—H11A | 0.9300 | C23—C24 | 1.382 (13) |
| C12—C13 | 1.404 (8) | C23—H23A | 0.9600 |
| C12—H12A | 0.9300 | C23—H23B | 0.9600 |
| C13—C14 | 1.365 (7) | C23—H23C | 0.9600 |
| C13—C20 | 1.534 (7) | C24—H24B | 0.9700 |
| C14—H14A | 0.9300 | C24—H24A | 0.9700 |

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| C15—H15A | 0.9600 |

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| C16—C1—C2 | 112.2 (4) | C5—C15—H15B | 109.5 |
| C16—C1—C17 | 110.1 (4) | H15A—C15—H15B | 109.5 |
| C2—C1—C17 | 106.4 (4) | C5—C15—H15C | 109.5 |
| C16—C1—C6 | 114.5 (4) | H15A—C15—H15C | 109.5 |
| C2—C1—C6 | 107.3 (3) | H15B—C15—H15C | 109.5 |
| C17—C1—C6 | 105.8 (3) | C1—C16—H16A | 109.5 |
| C3—C2—C1 | 113.8 (4) | C1—C16—H16B | 109.5 |
| C3—C2—H2A | 108.8 | H16A—C16—H16B | 109.5 |
| C1—C2—H2A | 108.8 | C1—C16—H16C | 109.5 |
| C3—C2—H2B | 108.8 | H16A—C16—H16C | 109.5 |
| C1—C2—H2B | 108.8 | H16B—C16—H16C | 109.5 |
| H2A—C2—H2B | 107.7 | N—C17—C1 | 113.7 (4) |
| C2—C3—C4 | 110.5 (4) | N—C17—H17A | 108.8 |
| C2—C3—H3A | 109.5 | C1—C17—H17A | 108.8 |
| C4—C3—H3A | 109.5 | N—C17—H17B | 108.8 |
| C2—C3—H3B | 109.5 | C1—C17—H17B | 108.8 |
| C4—C3—H3B | 109.5 | H17A—C17—H17B | 107.7 |
| H3A—C3—H3B | 108.1 | C19—N—C17 | 114.3 (4) |
| C3—C4—C5 | 111.6 (4) | C19—N—C18 | 109.9 (4) |
| C3—C4—H4A | 109.3 | C17—N—C18 | 110.8 (4) |
| C5—C4—H4A | 109.3 | C19—N—H0B | 107.1 |
| C3—C4—H4B | 109.3 | C17—N—H0B | 107.1 |
| C5—C4—H4B | 109.3 | C18—N—H0B | 107.1 |
| H4A—C4—H4B | 108.0 | N—C18—H18A | 109.5 |
| C7—C5—C4 | 111.3 (4) | N—C18—H18B | 109.5 |
| C7—C5—C15 | 105.1 (4) | H18A—C18—H18B | 109.5 |
| C4—C5—C15 | 110.1 (4) | N—C18—H18C | 109.5 |
| C7—C5—C6 | 107.3 (4) | H18A—C18—H18C | 109.5 |
| C4—C5—C6 | 107.1 (4) | H18B—C18—H18C | 109.5 |
| C15—C5—C6 | 115.9 (4) | N—C19—H19A | 109.5 |
| C10—C6—C5 | 109.6 (4) | N—C19—H19B | 109.5 |
| C10—C6—C1 | 114.3 (4) | H19A—C19—H19B | 109.5 |
| C5—C6—C1 | 115.0 (4) | N—C19—H19C | 109.5 |
| C10—C6—H6A | 105.7 | H19A—C19—H19C | 109.5 |
| C5—C6—H6A | 105.7 | H19B—C19—H19C | 109.5 |
| C1—C6—H6A | 105.7 | C22—C20—C13 | 114.9 (5) |
| C8—C7—C11 | 116.1 (5) | C22—C20—C21 | 114.0 (6) |
| C8—C7—C5 | 124.4 (4) | C13—C20—C21 | 109.6 (5) |
| C11—C7—C5 | 119.5 (4) | C22—C20—H20A | 105.8 |
| C14—C8—C7 | 120.6 (5) | C13—C20—H20A | 105.8 |
| C14—C8—C9 | 118.7 (5) | C21—C20—H20A | 105.8 |
| C7—C8—C9 | 120.7 (4) | C20—C21—H21A | 109.5 |
| C8—C9—C10 | 114.2 (5) | C20—C21—H21B | 109.5 |
| C8—C9—H9A | 108.7 | H21A—C21—H21B | 109.5 |
| C10—C9—H9A | 108.7 | C20—C21—H21C | 109.5 |
| C8—C9—H9B | 108.7 | H21A—C21—H21C | 109.5 |
| C10—C9—H9B | 108.7 | H21B—C21—H21C | 109.5 |

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| H9A—C9—H9B | 107.6 | C20—C22—H22A | 109.5 |
| C9—C10—C6 | 108.0 (4) | C20—C22—H22B | 109.5 |
| C9—C10—H10A | 110.1 | H22A—C22—H22B | 109.5 |
| C6—C10—H10A | 110.1 | C20—C22—H22C | 109.5 |
| C9—C10—H10B | 110.1 | H22A—C22—H22C | 109.5 |
| C6—C10—H10B | 110.1 | H22B—C22—H22C | 109.5 |
| H10A—C10—H10B | 108.4 | C24—O—H0A | 109.5 |
| C12—C11—C7 | 121.5 (5) | C24—C23—H23A | 109.5 |
| C12—C11—H11A | 119.3 | C24—C23—H23B | 109.5 |
| C7—C11—H11A | 119.3 | H23A—C23—H23B | 109.5 |
| C11—C12—C13 | 121.3 (5) | C24—C23—H23C | 109.5 |
| C11—C12—H12A | 119.3 | H23A—C23—H23C | 109.5 |
| C13—C12—H12A | 119.3 | H23B—C23—H23C | 109.5 |
| C14—C13—C12 | 116.3 (5) | O—C24—C23 | 106.1 (17) |
| C14—C13—C20 | 121.4 (5) | O—C24—H24B | 110.5 |
| C12—C13—C20 | 122.3 (5) | C23—C24—H24B | 110.5 |
| C8—C14—C13 | 124.2 (5) | O—C24—H24A | 110.5 |
| C8—C14—H14A | 117.9 | C23—C24—H24A | 110.5 |
| C13—C14—H14A | 117.9 | H24B—C24—H24A | 108.7 |
| C5—C15—H15A | 109.5 | | |

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| C16—C1—C2—C3 | 74.3 (5) | C5—C7—C8—C14 | −175.0 (5) |
| C17—C1—C2—C3 | −165.2 (4) | C11—C7—C8—C9 | −179.8 (5) |
| C6—C1—C2—C3 | −52.3 (5) | C5—C7—C8—C9 | 3.7 (8) |
| C1—C2—C3—C4 | 58.2 (6) | C14—C8—C9—C10 | 162.8 (5) |
| C2—C3—C4—C5 | −60.7 (6) | C7—C8—C9—C10 | −16.0 (7) |
| C3—C4—C5—C7 | 175.3 (4) | C8—C9—C10—C6 | 47.1 (6) |
| C3—C4—C5—C15 | −68.6 (5) | C5—C6—C10—C9 | −68.4 (5) |
| C3—C4—C5—C6 | 58.2 (5) | C1—C6—C10—C9 | 160.8 (4) |
| C7—C5—C6—C10 | 54.1 (5) | C8—C7—C11—C12 | 0.0 (7) |
| C4—C5—C6—C10 | 173.8 (4) | C5—C7—C11—C12 | 176.7 (4) |
| C15—C5—C6—C10 | −63.0 (5) | C7—C11—C12—C13 | −1.0 (8) |
| C7—C5—C6—C1 | −175.4 (4) | C11—C12—C13—C14 | 0.5 (7) |
| C4—C5—C6—C1 | −55.8 (5) | C11—C12—C13—C20 | 179.8 (5) |
| C15—C5—C6—C1 | 67.5 (5) | C7—C8—C14—C13 | −2.1 (8) |
| C16—C1—C6—C10 | 55.3 (5) | C9—C8—C14—C13 | 179.1 (5) |
| C2—C1—C6—C10 | −179.4 (4) | C12—C13—C14—C8 | 1.0 (8) |
| C17—C1—C6—C10 | −66.1 (5) | C20—C13—C14—C8 | −178.3 (5) |
| C16—C1—C6—C5 | −72.8 (5) | C16—C1—C17—N | 57.9 (5) |
| C2—C1—C6—C5 | 52.5 (5) | C2—C1—C17—N | −64.0 (5) |
| C17—C1—C6—C5 | 165.8 (4) | C6—C1—C17—N | −178.0 (4) |
| C4—C5—C7—C8 | −139.8 (5) | C1—C17—N—C19 | −113.0 (5) |
| C15—C5—C7—C8 | 101.1 (6) | C1—C17—N—C18 | 122.1 (4) |
| C6—C5—C7—C8 | −22.8 (6) | C14—C13—C20—C22 | 110.1 (7) |
| C4—C5—C7—C11 | 43.9 (6) | C12—C13—C20—C22 | −69.2 (8) |
| C15—C5—C7—C11 | −75.3 (5) | C14—C13—C20—C21 | −120.0 (6) |
| C6—C5—C7—C11 | 160.8 (4) | C12—C13—C20—C21 | 60.8 (8) |
| C11—C7—C8—C14 | 1.5 (7) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N—H0*B*···Cl | 0.91 | 2.27 | 3.097 (4) | 152 |
| O—H0*A*···Cl | 0.82 | 2.27 | 3.092 (9) | 178 |
| C18—H18*B*···Cl$^{i}$ | 0.96 | 2.78 | 3.694 (6) | 160 |
| C18—H18*C*···Cl$^{ii}$ | 0.96 | 2.84 | 3.693 (6) | 149 |
| C2—H2*B*···Cl | 0.97 | 2.86 | 3.775 (5) | 158 |

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1; (ii) *x*+1, *y*, *z*.