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# 3-(4-Chloroanilino)-2,5-dimethylcyclohex-2-en-1-one

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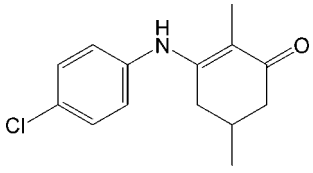
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.006 Å; disorder in main residue; $R$ factor = 0.054; $wR$ factor = 0.150; data-to-parameter ratio = 10.0.

In the title compound, $C_{14}H_{16}ClNO$, the dihedral angle between the benzene ring and the conjugated part of the cyclohexene ring is 61.7 (2)°. Part of the cyclohexene ring and one of the attached methyl groups are disordered over two orientations with occupancies of 0.602 (7) and 0.398 (7). In addition, the crystal studied was a racemic twin [Flack parameter = 0.58 (4)]. In the crystal, the molecules are linked into chains in the *b*-axis direction by intermolecular N—H···O hydrogen bonds. C—H···O and C—H···Cl interactions are also observed.

## Related literature

The title compound 3-(4-chlorophenylamino)-2,5-dimethylcyclohex-2-enone possesses significant anticonvulsant properties. For the anticonvulsant properties of enaminones, see: Edafiogho *et al.* (1992); Eddington *et al.* (2003); Scott *et al.* (1993, 1995). For related structures see: Alexander *et al.* (2010, 2011).




## Experimental

### Crystal data

$C_{14}H_{16}ClNO$
$M_r$ = 249.73
Monoclinic, $P2_1$
$a$ = 6.0775 (5) Å
$b$ = 8.8106 (5) Å
$c$ = 12.5794 (7) Å
$\beta$ = 99.904 (7)°
$V$ = 663.5 (1) Å$^3$
$Z$ = 2
Cu $K\alpha$ radiation
$\mu$ = 2.41 mm$^{-1}$
$T$ = 295 K
0.45 × 0.28 × 0.10 mm

### Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min}$ = 0.679, $T_{max}$ = 1.000
2292 measured reflections
1705 independent reflections
1417 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.150
$S$ = 1.00
1705 reflections
171 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.17 e Å$^{-3}$
Absolute structure: Flack (1983), 259 Friedel pairs
Flack parameter: 0.58 (4)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.86 | 2.10 | 2.897 (4) | 155 |
| C6—H6$A$···O1$^{ii}$ | 0.93 | 2.42 | 3.340 (4) | 172 |
| C12$A$—H12$A$···O1$^{ii}$ | 0.97 | 2.58 | 3.439 (9) | 147 |
| C12$B$—H12$D$···Cl1$^{iii}$ | 0.97 | 2.95 | 3.79 (3) | 146 |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2794).

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# supporting information

*Acta Cryst.* (2011). E**67**, o1283–o1284 [doi:10.1107/S1600536811005678]

## 3-(4-Chloroanilino)-2,5-dimethylcyclohex-2-en-1-one

**Henry North, Kwame Wutoh, M'egya K. Odoom, Pradeep Karla, Kenneth R. Scott and Ray J. Butcher**

### S1. Comment

The study of enaminones has led to several compounds possessing anticonvulsant properties (Edafiogho *et al.*, 1992; Eddington *et al.*, 2003; Scott *et al.*, 1993, 1995; Alexander *et al.*, 2010, 2011). Our group has extensively studied the effects of modification of the enaminone with substitutions at the methyl ester, ethyl ester, and without the ester group. Early in our work, the N–H binding site was confirmed when it was found that no compound was active with the N–H proton missing. All of the active compounds were *para*-substituted with an electron-withdrawing group. A series of compounds with vinyl proton substitution has recently synthesized. The title compound, 3-(4-chlorophenylamino)-2,5-dimethylcyclohex-2-enone was exclusively active in the maximal electroshock seizure evaluation (MES) in mice, indicative of protection against tonic-clonic convulsions in humans. The MES test with mice revealed no activity at the 30 mg $kg^{-1}$ dose, however in the 100 mg $kg^{-1}$ dose, 3/3 of the animals were protected at 30 minutes and 0/3 of the animals were protected at 4 h. At a dose of 300 mg $kg^{-1}$, 1/1 animals were protected 30 min and 4 h. There was toxicity at 30 min in 2/4 animals. In the rat MES study, at a dose of 30 mg $kg^{-1}$, 1/4 of the animals were protected at 1 and 2 h with no toxicity.

Since the shape of the molecule is important in determining binding to the receptor sites it is of interest to note that the dihedral angle between the phenyl ring and the conjugated part of the cyclohexene ring is 61.7 (2)°. The backbone of the cyclohexene ring is disordered over two conformations with occupancies of 0.602 (7) and 0.398 (7), respectively. In addition the compound is a racemic twin (Flack parameter of 0.58 (4)). The molecules are linked in chains in the b direction by intermolecular N—H···O hydrogen bonds.

### S2. Experimental

Iodomethane (11.2 ml, 0.18 mol, 1.5 equiv) was added to a solution of 5-methyl-1,3-cyclohexanedione (15.0 g, 0.119 mol) in 4 N aqueous sodium hydroxide (30 mL, 1.0 equiv of NaOH) in a two-neck 250 ml round bottom flask fitted with a magnetic stirrer and condenser. The solution was refluxed for 20 h and cooled to room temperature, then refrigerated at 0°C overnight. Vacuum filtration of the reaction mixture gave a crystalline mass dried to yield 9.24 g (54%). The crystalline mass, 2,5-dimethyl-1,3-cyclohexadione (2.10 g, 15 mmol), mp 170–172°C (lit. mp 130–131.5°C), 4-chloroaniline (2.32 g, 18 mmol), and toluene (60 ml) was added to a 150 ml single neck round bottom flask containing a stir bar. The solution was refluxed and stirred for 6 h with azeotropic removal of water by Dean-Stark trap. After standing overnight, crystals appeared. Evaporation under reduced pressure yielded crystals that were recrystallized from EtOAc, 44.3% yield (mp 183–185°C).

### S3. Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with a C—H distance of 0.93 and 0.98 Å $U_{iso}$(H) = 1.2$U_{eq}$(C) and 0.96 Å for $CH_3$ [$U_{iso}$(H) = 1.5$U_{eq}$(C)]. The H atoms attached to N were idealized with an N–H distance of 0.86 Å. The backbone of the cyclohexene was disordered over two conformations with occupancies of 0.602 (7) and 0.398 (7), respectively.

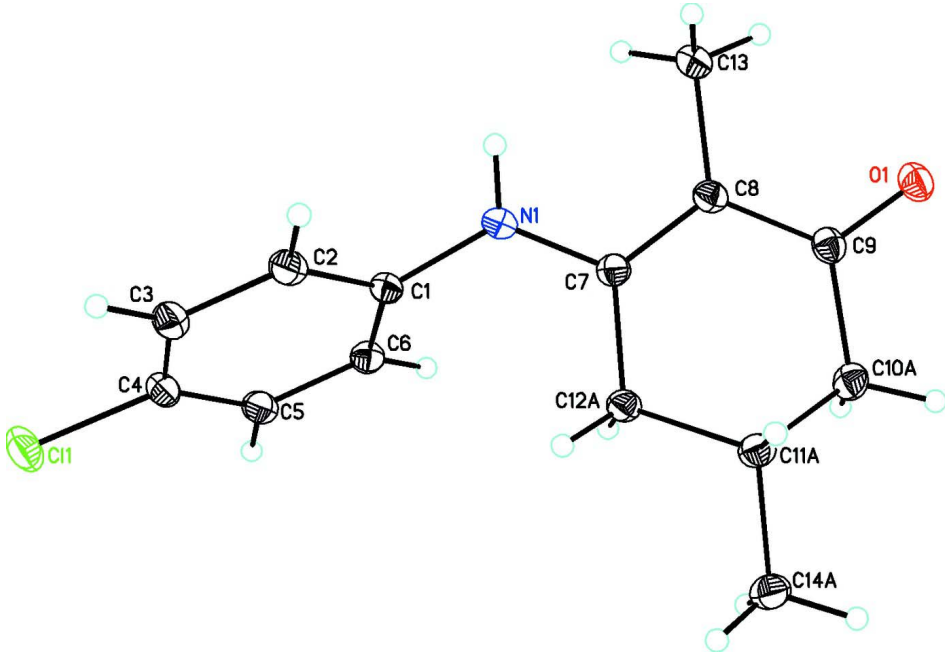


**Figure 1**

Diagram of 3-(4-Chlorophenylamino)-2,5-dimethylcyclohex-2-enone showing atom labeling scheme. Thermal ellipsoids drawn at the 30% probability level.

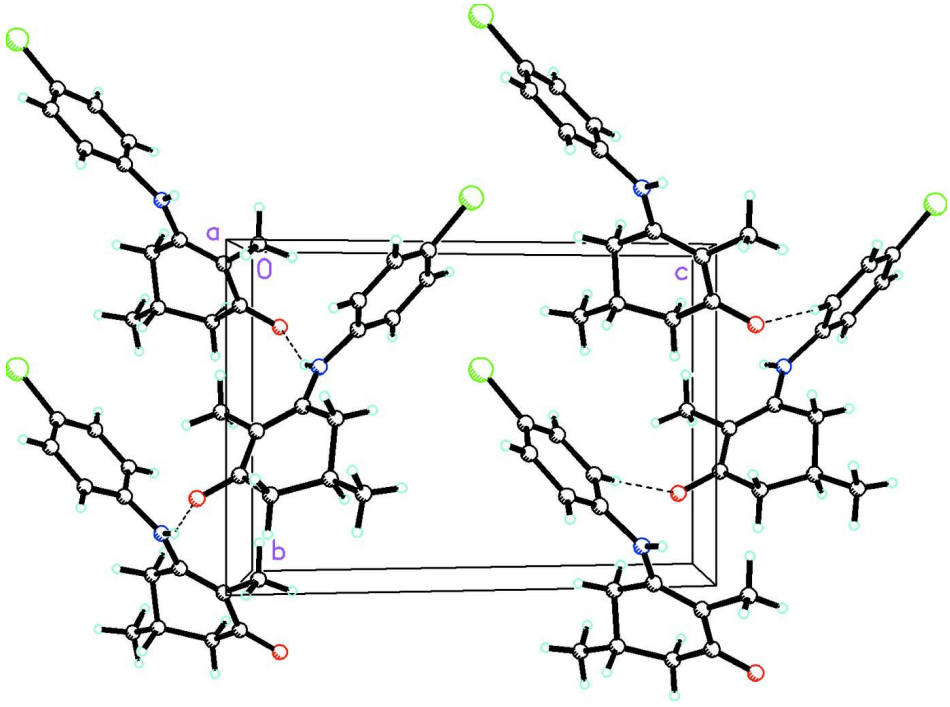


**Figure 2**

The molecular packing for 3-(4-Chlorophenylamino)-2,5-dimethylcyclohex-2-enone viewed down the *a* axis. Intermolecular interactions are shown by dashed lines.

### 3-(4-Chloroanilino)-2,5-dimethylcyclohex-2-en-1-one

*Crystal data*

$C_{14}H_{16}ClNO$
$M_r = 249.73$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.0775\ (5)$ Å
$b = 8.8106\ (5)$ Å
$c = 12.5794\ (7)$ Å
$\beta = 99.904\ (7)°$
$V = 663.5\ (1)$ Å$^3$
$Z = 2$
$F(000) = 264$
$D_x = 1.250$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 1339 reflections
$\theta = 5.0–73.9°$
$\mu = 2.41$ mm$^{-1}$
$T = 295$ K
Chunk, colorless
0.45 × 0.28 × 0.10 mm

*Data collection*

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.679$, $T_{max} = 1.000$
2292 measured reflections
1705 independent reflections
1417 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 74.1°$, $\theta_{min} = 6.2°$
$h = -7\rightarrow7$
$k = -10\rightarrow5$
$l = -13\rightarrow15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.150$
$S = 1.00$
1705 reflections
171 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1145P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å$^{-3}$
$\Delta\rho_{min} = -0.17$ e Å$^{-3}$
Absolute structure: Flack (1983), **259 Friedel pairs**
Absolute structure parameter: 0.58 (4)

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1 | 0.8577 (2) | −0.13354 (18) | 0.49024 (9) | 0.1059 (5) | |
| O1 | 0.7909 (4) | 0.7372 (4) | −0.0704 (2) | 0.0739 (7) | |
| N1 | 0.6375 (5) | 0.3591 (5) | 0.1708 (2) | 0.0660 (7) | |
| H1 | 0.5033 | 0.3524 | 0.1357 | 0.079* | |
| C1 | 0.7018 (5) | 0.2490 (4) | 0.2532 (2) | 0.0577 (8) | |
| C2 | 0.5554 (6) | 0.2139 (6) | 0.3223 (3) | 0.0706 (10) | |
| H2A | 0.4243 | 0.2693 | 0.3193 | 0.085* | |
| C3 | 0.6030 (7) | 0.0966 (6) | 0.3961 (3) | 0.0775 (11) | |
| H3A | 0.5036 | 0.0717 | 0.4419 | 0.093* | |
| C4 | 0.7978 (7) | 0.0183 (5) | 0.4006 (3) | 0.0716 (10) | |
| C5 | 0.9464 (6) | 0.0537 (5) | 0.3342 (3) | 0.0685 (9) | |
| H5A | 1.0803 | 0.0009 | 0.3397 | 0.082* | |
| C6 | 0.8969 (6) | 0.1680 (5) | 0.2591 (3) | 0.0630 (8) | |
| H6A | 0.9957 | 0.1904 | 0.2125 | 0.076* | |
| C7 | 0.7639 (6) | 0.4728 (4) | 0.1416 (3) | 0.0576 (8) | |
| C8 | 0.7074 (6) | 0.5456 (4) | 0.0452 (3) | 0.0576 (7) | |
| C9 | 0.8352 (5) | 0.6705 (5) | 0.0176 (3) | 0.0614 (8) | |
| C10A | 1.051 (3) | 0.719 (3) | 0.0899 (17) | 0.066 (3) | 0.602 (7) |
| H10A | 1.0710 | 0.8274 | 0.0847 | 0.080* | 0.602 (7) |
| H10B | 1.1773 | 0.6689 | 0.0664 | 0.080* | 0.602 (7) |
| C11A | 1.0433 (10) | 0.6749 (8) | 0.2071 (5) | 0.0654 (13) | 0.602 (7) |
| H11A | 0.9295 | 0.7376 | 0.2324 | 0.078* | 0.602 (7) |
| C12A | 0.977 (3) | 0.5058 (16) | 0.2158 (13) | 0.059 (2) | 0.602 (7) |

| | | | | | |
|---|---|---|---|---|---|
| H12A | 1.0945 | 0.4414 | 0.1976 | 0.070* | 0.602 (7) |
| H12B | 0.9599 | 0.4834 | 0.2894 | 0.070* | 0.602 (7) |
| C14A | 1.270 (3) | 0.708 (2) | 0.279 (2) | 0.081 (4) | 0.602 (7) |
| H14A | 1.3132 | 0.8111 | 0.2684 | 0.122* | 0.602 (7) |
| H14B | 1.3813 | 0.6404 | 0.2606 | 0.122* | 0.602 (7) |
| H14C | 1.2575 | 0.6937 | 0.3535 | 0.122* | 0.602 (7) |
| C10B | 1.015 (6) | 0.732 (5) | 0.110 (3) | 0.066 (3) | 0.398 (7) |
| H10C | 1.1249 | 0.7883 | 0.0783 | 0.080* | 0.398 (7) |
| H10D | 0.9443 | 0.8023 | 0.1529 | 0.080* | 0.398 (7) |
| C11B | 1.1314 (16) | 0.6105 (13) | 0.1820 (8) | 0.0654 (13) | 0.398 (7) |
| H11B | 1.1935 | 0.5344 | 0.1386 | 0.078* | 0.398 (7) |
| C12B | 0.950 (5) | 0.540 (3) | 0.233 (2) | 0.059 (2) | 0.398 (7) |
| H12C | 0.8849 | 0.6157 | 0.2745 | 0.070* | 0.398 (7) |
| H12D | 1.0119 | 0.4596 | 0.2822 | 0.070* | 0.398 (7) |
| C14B | 1.321 (6) | 0.673 (4) | 0.272 (4) | 0.081 (4) | 0.398 (7) |
| H14D | 1.4200 | 0.5920 | 0.3000 | 0.122* | 0.398 (7) |
| H14E | 1.2559 | 0.7160 | 0.3298 | 0.122* | 0.398 (7) |
| H14F | 1.4044 | 0.7502 | 0.2423 | 0.122* | 0.398 (7) |
| C13 | 0.5116 (7) | 0.4931 (5) | −0.0383 (3) | 0.0716 (10) | |
| H13A | 0.5080 | 0.3842 | −0.0402 | 0.107* | |
| H13B | 0.5279 | 0.5312 | −0.1080 | 0.107* | |
| H13C | 0.3749 | 0.5306 | −0.0196 | 0.107* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.1398 (11) | 0.0944 (8) | 0.0775 (6) | 0.0012 (8) | 0.0015 (6) | 0.0320 (6) |
| O1 | 0.0719 (15) | 0.0806 (17) | 0.0706 (14) | 0.0063 (14) | 0.0158 (11) | 0.0202 (13) |
| N1 | 0.0611 (14) | 0.0737 (18) | 0.0575 (14) | −0.0048 (16) | −0.0060 (11) | 0.0102 (15) |
| C1 | 0.0651 (18) | 0.0592 (19) | 0.0451 (14) | −0.0076 (16) | −0.0009 (12) | 0.0016 (13) |
| C2 | 0.0615 (18) | 0.085 (3) | 0.0630 (18) | −0.0002 (19) | 0.0042 (14) | 0.0081 (18) |
| C3 | 0.074 (2) | 0.100 (3) | 0.0582 (17) | −0.009 (2) | 0.0091 (15) | 0.015 (2) |
| C4 | 0.087 (3) | 0.068 (2) | 0.0535 (17) | −0.008 (2) | −0.0037 (15) | 0.0098 (16) |
| C5 | 0.074 (2) | 0.065 (2) | 0.0623 (18) | 0.003 (2) | −0.0017 (15) | −0.0009 (17) |
| C6 | 0.0663 (18) | 0.066 (2) | 0.0556 (15) | −0.0063 (19) | 0.0059 (13) | −0.0038 (16) |
| C7 | 0.0562 (16) | 0.0566 (19) | 0.0579 (16) | −0.0011 (15) | 0.0042 (12) | −0.0010 (15) |
| C8 | 0.0563 (16) | 0.0581 (18) | 0.0566 (16) | 0.0059 (16) | 0.0046 (12) | −0.0017 (15) |
| C9 | 0.0585 (16) | 0.0611 (19) | 0.0660 (18) | 0.0088 (17) | 0.0144 (14) | 0.0059 (17) |
| C10A | 0.057 (7) | 0.072 (6) | 0.073 (8) | −0.005 (5) | 0.021 (4) | 0.005 (6) |
| C11A | 0.062 (3) | 0.060 (4) | 0.070 (3) | 0.000 (3) | 0.001 (2) | −0.007 (3) |
| C12A | 0.065 (5) | 0.051 (7) | 0.056 (6) | 0.003 (5) | 0.000 (3) | −0.006 (4) |
| C14A | 0.070 (10) | 0.075 (10) | 0.092 (4) | −0.006 (6) | −0.007 (6) | −0.005 (7) |
| C10B | 0.057 (7) | 0.072 (6) | 0.073 (8) | −0.005 (5) | 0.021 (4) | 0.005 (6) |
| C11B | 0.062 (3) | 0.060 (4) | 0.070 (3) | 0.000 (3) | 0.001 (2) | −0.007 (3) |
| C12B | 0.065 (5) | 0.051 (7) | 0.056 (6) | 0.003 (5) | 0.000 (3) | −0.006 (4) |
| C14B | 0.070 (10) | 0.075 (10) | 0.092 (4) | −0.006 (6) | −0.007 (6) | −0.005 (7) |
| C13 | 0.072 (2) | 0.075 (2) | 0.0617 (19) | 0.000 (2) | −0.0045 (16) | 0.0077 (18) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.747 (4) | C10A—H10B | 0.9700 |
| O1—C9 | 1.241 (4) | C11A—C14A | 1.54 (2) |
| N1—C7 | 1.351 (5) | C11A—C12A | 1.552 (18) |
| N1—C1 | 1.424 (5) | C11A—H11A | 0.9800 |
| N1—H1 | 0.8600 | C12A—H12A | 0.9700 |
| C1—C6 | 1.375 (5) | C12A—H12B | 0.9700 |
| C1—C2 | 1.382 (5) | C14A—H14A | 0.9600 |
| C2—C3 | 1.386 (6) | C14A—H14B | 0.9600 |
| C2—H2A | 0.9300 | C14A—H14C | 0.9600 |
| C3—C4 | 1.363 (6) | C10B—C11B | 1.50 (4) |
| C3—H3A | 0.9300 | C10B—H10C | 0.9700 |
| C4—C5 | 1.367 (5) | C10B—H10D | 0.9700 |
| C5—C6 | 1.378 (6) | C11B—C12B | 1.50 (3) |
| C5—H5A | 0.9300 | C11B—C14B | 1.58 (5) |
| C6—H6A | 0.9300 | C11B—H11B | 0.9800 |
| C7—C8 | 1.363 (5) | C12B—H12C | 0.9700 |
| C7—C12A | 1.489 (19) | C12B—H12D | 0.9700 |
| C7—C12B | 1.58 (3) | C14B—H14D | 0.9600 |
| C8—C9 | 1.424 (5) | C14B—H14E | 0.9600 |
| C8—C13 | 1.518 (5) | C14B—H14F | 0.9600 |
| C9—C10A | 1.52 (3) | C13—H13A | 0.9600 |
| C9—C10B | 1.55 (4) | C13—H13B | 0.9600 |
| C10A—C11A | 1.53 (2) | C13—H13C | 0.9600 |
| C10A—H10A | 0.9700 | | |
| | | | |
| C7—N1—C1 | 127.3 (3) | C10A—C11A—C14A | 110.2 (14) |
| C7—N1—H1 | 116.4 | C10A—C11A—C12A | 111.1 (13) |
| C1—N1—H1 | 116.4 | C14A—C11A—C12A | 110.9 (12) |
| C6—C1—C2 | 119.5 (3) | C10A—C11A—H11A | 108.2 |
| C6—C1—N1 | 121.3 (3) | C14A—C11A—H11A | 108.2 |
| C2—C1—N1 | 119.0 (3) | C12A—C11A—H11A | 108.2 |
| C1—C2—C3 | 120.4 (4) | C7—C12A—C11A | 110.6 (11) |
| C1—C2—H2A | 119.8 | C7—C12A—H12A | 109.5 |
| C3—C2—H2A | 119.8 | C11A—C12A—H12A | 109.5 |
| C4—C3—C2 | 119.0 (4) | C7—C12A—H12B | 109.5 |
| C4—C3—H3A | 120.5 | C11A—C12A—H12B | 109.5 |
| C2—C3—H3A | 120.5 | H12A—C12A—H12B | 108.1 |
| C3—C4—C5 | 121.3 (4) | C11B—C10B—C9 | 114 (3) |
| C3—C4—Cl1 | 119.8 (3) | C11B—C10B—H10C | 108.8 |
| C5—C4—Cl1 | 118.9 (3) | C9—C10B—H10C | 108.8 |
| C4—C5—C6 | 119.8 (4) | C11B—C10B—H10D | 108.8 |
| C4—C5—H5A | 120.1 | C9—C10B—H10D | 108.8 |
| C6—C5—H5A | 120.1 | H10C—C10B—H10D | 107.7 |
| C1—C6—C5 | 120.1 (3) | C10B—C11B—C12B | 104.5 (18) |
| C1—C6—H6A | 120.0 | C10B—C11B—C14B | 113 (2) |
| C5—C6—H6A | 120.0 | C12B—C11B—C14B | 110 (2) |

| | | | |
|---|---|---|---|
| N1—C7—C8 | 121.5 (3) | C10B—C11B—H11B | 109.7 |
| N1—C7—C12A | 116.6 (8) | C12B—C11B—H11B | 109.7 |
| C8—C7—C12A | 121.7 (8) | C14B—C11B—H11B | 109.7 |
| N1—C7—C12B | 116.5 (12) | C11B—C12B—C7 | 109.0 (17) |
| C8—C7—C12B | 120.8 (12) | C11B—C12B—H12C | 109.9 |
| C12A—C7—C12B | 15.4 (10) | C7—C12B—H12C | 109.9 |
| C7—C8—C9 | 121.1 (3) | C11B—C12B—H12D | 109.9 |
| C7—C8—C13 | 121.3 (3) | C7—C12B—H12D | 109.9 |
| C9—C8—C13 | 117.6 (3) | H12C—C12B—H12D | 108.3 |
| O1—C9—C8 | 122.6 (3) | C11B—C14B—H14D | 109.5 |
| O1—C9—C10A | 115.7 (10) | C11B—C14B—H14E | 109.5 |
| C8—C9—C10A | 121.3 (10) | H14D—C14B—H14E | 109.5 |
| O1—C9—C10B | 121.3 (16) | C11B—C14B—H14F | 109.5 |
| C8—C9—C10B | 115.6 (15) | H14D—C14B—H14F | 109.5 |
| C10A—C9—C10B | 14.3 (11) | H14E—C14B—H14F | 109.5 |
| C9—C10A—C11A | 109.7 (13) | C8—C13—H13A | 109.5 |
| C9—C10A—H10A | 109.7 | C8—C13—H13B | 109.5 |
| C11A—C10A—H10A | 109.7 | H13A—C13—H13B | 109.5 |
| C9—C10A—H10B | 109.7 | C8—C13—H13C | 109.5 |
| C11A—C10A—H10B | 109.7 | H13A—C13—H13C | 109.5 |
| H10A—C10A—H10B | 108.2 | H13B—C13—H13C | 109.5 |

| | | | |
|---|---|---|---|
| C7—N1—C1—C6 | −49.7 (6) | C13—C8—C9—C10A | −172.1 (8) |
| C7—N1—C1—C2 | 136.0 (4) | C7—C8—C9—C10B | −9.3 (13) |
| C6—C1—C2—C3 | −1.0 (6) | C13—C8—C9—C10B | 173.0 (13) |
| N1—C1—C2—C3 | 173.4 (4) | O1—C9—C10A—C11A | 159.1 (10) |
| C1—C2—C3—C4 | 1.1 (6) | C8—C9—C10A—C11A | −27.6 (17) |
| C2—C3—C4—C5 | 0.3 (6) | C10B—C9—C10A—C11A | 43 (10) |
| C2—C3—C4—Cl1 | −178.1 (3) | C9—C10A—C11A—C14A | 174.3 (12) |
| C3—C4—C5—C6 | −1.7 (6) | C9—C10A—C11A—C12A | 50.9 (17) |
| Cl1—C4—C5—C6 | 176.7 (3) | N1—C7—C12A—C11A | −152.2 (7) |
| C2—C1—C6—C5 | −0.5 (5) | C8—C7—C12A—C11A | 32.7 (10) |
| N1—C1—C6—C5 | −174.8 (3) | C12B—C7—C12A—C11A | −59 (6) |
| C4—C5—C6—C1 | 1.9 (6) | C10A—C11A—C12A—C7 | −54.1 (13) |
| C1—N1—C7—C8 | 162.5 (3) | C14A—C11A—C12A—C7 | −177.0 (10) |
| C1—N1—C7—C12A | −12.5 (7) | O1—C9—C10B—C11B | −149.8 (12) |
| C1—N1—C7—C12B | −29.8 (10) | C8—C9—C10B—C11B | 37.9 (17) |
| N1—C7—C8—C9 | 176.7 (3) | C10A—C9—C10B—C11B | −79 (10) |
| C12A—C7—C8—C9 | −8.4 (7) | C9—C10B—C11B—C12B | −63.3 (18) |
| C12B—C7—C8—C9 | 9.6 (10) | C9—C10B—C11B—C14B | 177.3 (18) |
| N1—C7—C8—C13 | −5.6 (6) | C10B—C11B—C12B—C7 | 60 (2) |
| C12A—C7—C8—C13 | 169.2 (5) | C14B—C11B—C12B—C7 | −178.3 (16) |
| C12B—C7—C8—C13 | −172.8 (9) | N1—C7—C12B—C11B | 155.6 (11) |
| C7—C8—C9—O1 | 178.5 (4) | C8—C7—C12B—C11B | −36.6 (17) |
| C13—C8—C9—O1 | 0.7 (5) | C12A—C7—C12B—C11B | 61 (6) |
| C7—C8—C9—C10A | 5.6 (9) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.86 | 2.10 | 2.897 (4) | 155 |
| C6—H6*A*···O1$^{ii}$ | 0.93 | 2.42 | 3.340 (4) | 172 |
| C12*A*—H12*A*···O1$^{ii}$ | 0.97 | 2.58 | 3.439 (9) | 147 |
| C12*B*—H12*D*···Cl1$^{iii}$ | 0.97 | 2.95 | 3.79 (3) | 146 |

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