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

# 4-(4-Chlorophenyl)-4-hydroxypiperidinium benzoate

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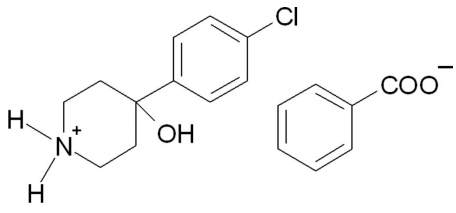
Received 5 May 2011; accepted 11 May 2011

Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.046; $wR$ factor = 0.125; data-to-parameter ratio = 19.3.

In the title salt, $C_{11}H_{15}ClNO^{+}\cdot C_7H_5O_2^{-}$, the dihedral angle between the mean planes of the chlorophenyl ring of the cation and the benzene ring of the anion is 74.4 (1)°. In the cation, the six-membered piperazine ring adopts a chair conformation. The crystal packing is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds, and weak intermolecular C—H···O, C—H···Cl and C—H···$\pi$ interactions.

## Related literature

For the synthesis and biological activity of uncondensed cyclic derivatives of piperidine, see: Vartanyan (1984). For puckering parameters, see: Cremer & Pople (1975) For related structures, see: Jasinski *et al.* (2009). For ring-motif pattterns, see: Bernstein *et al.* (1994).




## Experimental

### Crystal data

$C_{11}H_{15}ClNO^{+}\cdot C_7H_5O_2^{-}$
$M_r$ = 333.80
Triclinic, $P\overline{1}$
$a$ = 9.6235 (12) Å
$b$ = 10.0971 (16) Å
$c$ = 10.2251 (14) Å
$\alpha$ = 99.608 (12)°
$\beta$ = 108.748 (13)°
$\gamma$ = 113.357 (14)°
$V$ = 812.7 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.25 mm$^{-1}$
$T$ = 173 K
0.34 × 0.30 × 0.13 mm

### Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010)
$T_{min}$ = 0.920, $T_{max}$ = 0.968
7857 measured reflections
4184 independent reflections
3222 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.018

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.125
$S$ = 1.05
4184 reflections
217 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

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

*Cg*3 is the centroid of the C13–C18 ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*NA*···O3$^{i}$ | 0.87 (1) | 1.84 (1) | 2.6964 (17) | 166 (2) |
| O1—H1*O*···O3 | 0.82 (2) | 2.05 (2) | 2.7780 (16) | 147 (2) |
| N1—H1*NB*···O2$^{ii}$ | 0.86 (1) | 1.92 (1) | 2.7609 (18) | 166 (2) |
| C16—H16*A*···Cl1$^{iii}$ | 0.95 | 2.78 | 3.5268 (17) | 136 |
| C9—H9*B*···O1$^{i}$ | 0.99 | 2.46 | 3.3008 (19) | 143 |
| C1—H1*A*···*Cg*3$^{iv}$ | 0.95 | 2.70 | 3.554 (2) | 150 |

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x, y-1, z$; (iii) $-x, -y+1, -z$; (iv) $-x, -y+1, -z+1$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2010); 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*.

BPS thanks the University of Mysore for the research facilities. HSY is grateful to RL Fine Chem., Bengaluru, India, for a pure sample of 4-(4-chlorophynyl)-piperidin-4-ol. JPJ acknowledges the NSF–MRI program (grant No. CHE1039027) for funds to purchase the X-ray diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2415).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, o1436 [ **doi:10.1107/S1600536811017855** ]

# 4-(4-Chlorophenyl)-4-hydroxypiperidinium benzoate

**J. P. Jasinski, J. A. Golen, B. P. Siddaraju, A. S. Dayananda and H. S. Yathirajan**

## Comment

4-(4-Chlorophenyl)-4-hydroxypiperidine is used as an intermediate for the synthesis of pharmaceuticals such as haloperidol (a neuroleptic drug used to treat patients with psychotic illnesses, extreme agitation, or Tourette's syndrome) and loperamide (a synthetic piperidine derivative, effective against diarrhea resulting from gastroenteritis or inflammatory bowel disease). A review on the synthesis and biological activities of uncondensed cyclic derivatives of piperidine is reported (Vartanyan, 1984). The crystal structure of a related compound, 4-[(E)-(2,4-difluorophenyl) (hydroxyimino)methyl]piperidinium picrate (Jasinski *et al.*, 2009) has been reported. In this paperwe report the crystal structure of $C_{11}H_{15}ONCl^{+}$ . $C_{15}H_{12}O_2^{-}$.

In the title salt (Fig. 1), the 6-membered piperazine ring in the cation adopts a chair conformation with puckering parameters (Cremer & Pople, 1975) *Q*, θ and φ, 0.568 (2) Å, 0.00 (19)° and 278 (9)°, respectively . The dihedral angle between the mean planes of the chlorophenyl ring of the cation and the benzene ring of the anion is 74.4 (1)°. The crystal structure is stabilized by N1—H1NA···O3 and N1—H1NB···O2, hydrogen bonds forming $R_4^4(12)$ ring-motif patttern (Bernstein *et al.*, 1994) and N1—H1NA···O3, O1—H10···O3 hydrogen bonds resulting in $R_2^4(16)$ ring-motif, generating one dimensional chains along the *c* axis (Fig. 2). The structure is further consolidated by weak C9—H9B···O1, C16—H16A···Cl1 and C1—H1A···Cg3 π-ring intermolecular interactions.

## Experimental

Solutions of 4-(4-chlorophenyl)-piperidin-4-ol (2.12 g, 0.01 mol) in methanol (10 ml) and benzoic acid (1.226 g, 0.01 mol) in methanol (10 ml) were mixed and stirred in a beaker at 333 K for 30 minutes. The mixture was kept aside for three days at room temperature. The salt thus obtained was filtered and dried in a vaccum desiccator over phosphorous pentoxide. The compound was recrystallized from N,N-dimethylformamide by slow evaporation (m.p: 498 - 501 K).

## Refinement

Hydrogen atoms on O1 and N1 were found from a Fourier difference map and were refined using DFIX 0.84(0.02) and 0.86(0.01) values for O–H and N–H distances, respectively, and $U_{iso}$(H) = 1.2 times $U_{eq}$ (O/N). The rest of the H atoms were positioned geometrically, and allowed to ride on their parent atoms, with C—H distances 0.95 Å (CH) or 0.99 Å ($CH_2$) and $U_{iso}$(H) = 1.18-1.21 times $U_{eq}$ (C).

## Figures

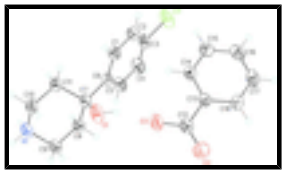

Fig. 1. Molecular structure of the title compound showing the atom labeling scheme and 50% probability displacement ellipsoids.

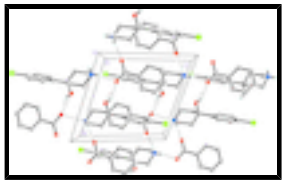

Fig. 2. Packing diagram for the title compound viewed down the *a* axis. Dashed lines indicate N—H···O and O—H···O hydrogen bonds generating one dimensional chains along the *c* axis.

## 4-(4-Chlorophenyl)-4-hydroxypiperidinium benzoate

*Crystal data*

| | |
|---|---|
| $C_{11}H_{15}ClNO^{+}\cdot C_7H_5O_2^{-}$ | $Z = 2$ |
| $M_r = 333.80$ | $F(000) = 352$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.364$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.6235$ (12) Å | Cell parameters from 3721 reflections |
| $b = 10.0971$ (16) Å | $\theta = 3.7$–$32.3°$ |
| $c = 10.2251$ (14) Å | $\mu = 0.25$ mm$^{-1}$ |
| $\alpha = 99.608$ (12)° | $T = 173$ K |
| $\beta = 108.748$ (13)° | Block, colorless |
| $\gamma = 113.357$ (14)° | 0.34 × 0.30 × 0.13 mm |
| $V = 812.7$ (2) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Xcalibur Eos Gemini diffractometer | 4184 independent reflections |
| Radiation source: Enhance (Mo) X-ray Source | 3222 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.018$ |
| Detector resolution: 16.1500 pixels mm$^{-1}$ | $\theta_{max} = 28.7°$, $\theta_{min} = 3.7°$ |
| ω scans | $h = -12 \rightarrow 12$ |
| Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2010) | $k = -13 \rightarrow 13$ |
| $T_{min} = 0.920$, $T_{max} = 0.968$ | $l = -13 \rightarrow 13$ |
| 7857 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.125$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.05$ | $w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.1776P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 4184 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 217 parameters | $\Delta\rho_{max} = 0.29$ e Å$^{-3}$ |
| 4 restraints | $\Delta\rho_{min} = -0.34$ e Å$^{-3}$ |

*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.

**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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.01350 (8) | 0.19249 (7) | 0.06335 (5) | 0.06605 (19) |
| O1 | 0.37436 (18) | 0.44147 (14) | 0.79300 (12) | 0.0470 (3) |
| H1O | 0.360 (3) | 0.498 (2) | 0.746 (2) | 0.056* |
| O2 | 0.46446 (18) | 0.94763 (13) | 0.77613 (13) | 0.0553 (4) |
| O3 | 0.36182 (17) | 0.70222 (13) | 0.75160 (12) | 0.0504 (3) |
| N1 | 0.51659 (18) | 0.21683 (15) | 0.95370 (13) | 0.0372 (3) |
| H1NB | 0.514 (2) | 0.1342 (16) | 0.9106 (18) | 0.045* |
| H1NA | 0.561 (2) | 0.231 (2) | 1.0473 (12) | 0.045* |
| C1 | 0.1141 (2) | 0.24120 (18) | 0.48209 (16) | 0.0351 (3) |
| H1A | 0.0551 | 0.2344 | 0.5416 | 0.042* |
| C2 | 0.0314 (2) | 0.21412 (19) | 0.33410 (17) | 0.0393 (4) |
| H2A | −0.0832 | 0.1888 | 0.2920 | 0.047* |
| C3 | 0.1181 (2) | 0.22451 (18) | 0.24891 (16) | 0.0387 (4) |
| C4 | 0.2839 (2) | 0.2611 (2) | 0.30731 (18) | 0.0474 (4) |
| H4A | 0.3419 | 0.2678 | 0.2470 | 0.057* |
| C5 | 0.3653 (2) | 0.2881 (2) | 0.45636 (17) | 0.0404 (4) |
| H5A | 0.4802 | 0.3140 | 0.4978 | 0.048* |
| C6 | 0.28210 (18) | 0.27817 (15) | 0.54568 (14) | 0.0278 (3) |
| C7 | 0.36689 (18) | 0.30769 (15) | 0.70961 (14) | 0.0282 (3) |
| C8 | 0.54504 (19) | 0.32943 (17) | 0.76015 (15) | 0.0331 (3) |
| H8A | 0.5421 | 0.2395 | 0.7006 | 0.040* |
| H8B | 0.6156 | 0.4212 | 0.7432 | 0.040* |
| C9 | 0.6232 (2) | 0.34823 (18) | 0.92121 (16) | 0.0373 (3) |

| | | | | |
|---|---|---|---|---|
| H9A | 0.7351 | 0.3556 | 0.9474 | 0.045* |
| H9B | 0.6383 | 0.4445 | 0.9818 | 0.045* |
| C10 | 0.3447 (2) | 0.19647 (19) | 0.91069 (16) | 0.0388 (4) |
| H10A | 0.3496 | 0.2880 | 0.9697 | 0.047* |
| H10B | 0.2763 | 0.1064 | 0.9310 | 0.047* |
| C11 | 0.26339 (19) | 0.17363 (18) | 0.74911 (16) | 0.0346 (3) |
| H11A | 0.1503 | 0.1632 | 0.7235 | 0.042* |
| H11B | 0.2503 | 0.0774 | 0.6904 | 0.042* |
| C12 | 0.37590 (19) | 0.80987 (17) | 0.70139 (15) | 0.0324 (3) |
| C13 | 0.27602 (18) | 0.76646 (16) | 0.53903 (15) | 0.0283 (3) |
| C14 | 0.1622 (2) | 0.61404 (17) | 0.45411 (16) | 0.0363 (3) |
| H14A | 0.1524 | 0.5367 | 0.4977 | 0.044* |
| C15 | 0.0633 (2) | 0.5739 (2) | 0.30681 (18) | 0.0447 (4) |
| H15A | −0.0151 | 0.4695 | 0.2498 | 0.054* |
| C16 | 0.0782 (2) | 0.6857 (2) | 0.24240 (18) | 0.0463 (4) |
| H16A | 0.0093 | 0.6580 | 0.1413 | 0.056* |
| C17 | 0.1924 (2) | 0.8366 (2) | 0.32407 (19) | 0.0452 (4) |
| H17A | 0.2042 | 0.9130 | 0.2790 | 0.054* |
| C18 | 0.2902 (2) | 0.87754 (17) | 0.47237 (17) | 0.0360 (3) |
| H18A | 0.3677 | 0.9823 | 0.5289 | 0.043* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0874 (4) | 0.0854 (4) | 0.0294 (2) | 0.0485 (3) | 0.0179 (2) | 0.0257 (2) |
| O1 | 0.0826 (9) | 0.0447 (7) | 0.0316 (6) | 0.0468 (7) | 0.0235 (6) | 0.0149 (5) |
| O2 | 0.0710 (9) | 0.0364 (6) | 0.0391 (6) | 0.0248 (6) | 0.0082 (6) | 0.0014 (5) |
| O3 | 0.0727 (9) | 0.0397 (6) | 0.0299 (5) | 0.0272 (6) | 0.0101 (6) | 0.0147 (5) |
| N1 | 0.0512 (8) | 0.0349 (7) | 0.0253 (6) | 0.0256 (6) | 0.0101 (6) | 0.0098 (5) |
| C1 | 0.0360 (8) | 0.0427 (8) | 0.0311 (7) | 0.0198 (7) | 0.0169 (6) | 0.0158 (6) |
| C2 | 0.0362 (8) | 0.0442 (9) | 0.0344 (7) | 0.0189 (7) | 0.0109 (7) | 0.0159 (7) |
| C3 | 0.0526 (10) | 0.0401 (8) | 0.0270 (7) | 0.0256 (8) | 0.0151 (7) | 0.0155 (6) |
| C4 | 0.0594 (11) | 0.0691 (12) | 0.0375 (8) | 0.0397 (10) | 0.0314 (8) | 0.0291 (8) |
| C5 | 0.0406 (9) | 0.0589 (10) | 0.0371 (8) | 0.0300 (8) | 0.0225 (7) | 0.0243 (7) |
| C6 | 0.0346 (8) | 0.0273 (6) | 0.0269 (6) | 0.0175 (6) | 0.0146 (6) | 0.0119 (5) |
| C7 | 0.0360 (8) | 0.0289 (7) | 0.0260 (6) | 0.0192 (6) | 0.0153 (6) | 0.0109 (5) |
| C8 | 0.0326 (8) | 0.0347 (7) | 0.0294 (7) | 0.0152 (6) | 0.0119 (6) | 0.0102 (6) |
| C9 | 0.0364 (8) | 0.0387 (8) | 0.0287 (7) | 0.0172 (7) | 0.0075 (6) | 0.0080 (6) |
| C10 | 0.0466 (9) | 0.0422 (8) | 0.0329 (7) | 0.0217 (7) | 0.0192 (7) | 0.0192 (6) |
| C11 | 0.0342 (8) | 0.0379 (8) | 0.0320 (7) | 0.0159 (7) | 0.0142 (6) | 0.0164 (6) |
| C12 | 0.0366 (8) | 0.0331 (7) | 0.0290 (7) | 0.0210 (6) | 0.0116 (6) | 0.0083 (6) |
| C13 | 0.0298 (7) | 0.0322 (7) | 0.0300 (7) | 0.0187 (6) | 0.0150 (6) | 0.0129 (5) |
| C14 | 0.0387 (8) | 0.0330 (7) | 0.0326 (7) | 0.0155 (7) | 0.0110 (6) | 0.0139 (6) |
| C15 | 0.0445 (10) | 0.0408 (9) | 0.0335 (8) | 0.0149 (8) | 0.0079 (7) | 0.0089 (7) |
| C16 | 0.0498 (10) | 0.0602 (11) | 0.0316 (8) | 0.0290 (9) | 0.0141 (7) | 0.0216 (8) |
| C17 | 0.0557 (11) | 0.0527 (10) | 0.0442 (9) | 0.0311 (9) | 0.0271 (8) | 0.0316 (8) |
| C18 | 0.0406 (9) | 0.0327 (7) | 0.0407 (8) | 0.0191 (7) | 0.0205 (7) | 0.0166 (6) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cl1—C3 | 1.7400 (15) | C8—C9 | 1.5167 (19) |
| O1—C7 | 1.4338 (17) | C8—H8A | 0.9900 |
| O1—H1O | 0.823 (15) | C8—H8B | 0.9900 |
| O2—C12 | 1.2376 (19) | C9—H9A | 0.9900 |
| O3—C12 | 1.2560 (18) | C9—H9B | 0.9900 |
| N1—C10 | 1.485 (2) | C10—C11 | 1.514 (2) |
| N1—C9 | 1.487 (2) | C10—H10A | 0.9900 |
| N1—H1NB | 0.861 (10) | C10—H10B | 0.9900 |
| N1—H1NA | 0.873 (10) | C11—H11A | 0.9900 |
| C1—C2 | 1.383 (2) | C11—H11B | 0.9900 |
| C1—C6 | 1.393 (2) | C12—C13 | 1.5064 (19) |
| C1—H1A | 0.9500 | C13—C14 | 1.389 (2) |
| C2—C3 | 1.376 (2) | C13—C18 | 1.391 (2) |
| C2—H2A | 0.9500 | C14—C15 | 1.381 (2) |
| C3—C4 | 1.373 (3) | C14—H14A | 0.9500 |
| C4—C5 | 1.391 (2) | C15—C16 | 1.381 (2) |
| C4—H4A | 0.9500 | C15—H15A | 0.9500 |
| C5—C6 | 1.387 (2) | C16—C17 | 1.373 (3) |
| C5—H5A | 0.9500 | C16—H16A | 0.9500 |
| C6—C7 | 1.5250 (18) | C17—C18 | 1.386 (2) |
| C7—C8 | 1.533 (2) | C17—H17A | 0.9500 |
| C7—C11 | 1.536 (2) | C18—H18A | 0.9500 |
| C7—O1—H1O | 114.0 (15) | N1—C9—H9A | 109.4 |
| C10—N1—C9 | 111.70 (12) | C8—C9—H9A | 109.4 |
| C10—N1—H1NB | 110.7 (13) | N1—C9—H9B | 109.4 |
| C9—N1—H1NB | 110.2 (13) | C8—C9—H9B | 109.4 |
| C10—N1—H1NA | 108.5 (13) | H9A—C9—H9B | 108.0 |
| C9—N1—H1NA | 110.3 (13) | N1—C10—C11 | 110.57 (13) |
| H1NB—N1—H1NA | 105.3 (16) | N1—C10—H10A | 109.5 |
| C2—C1—C6 | 121.38 (14) | C11—C10—H10A | 109.5 |
| C2—C1—H1A | 119.3 | N1—C10—H10B | 109.5 |
| C6—C1—H1A | 119.3 | C11—C10—H10B | 109.5 |
| C3—C2—C1 | 118.85 (15) | H10A—C10—H10B | 108.1 |
| C3—C2—H2A | 120.6 | C10—C11—C7 | 112.01 (13) |
| C1—C2—H2A | 120.6 | C10—C11—H11A | 109.2 |
| C4—C3—C2 | 121.62 (14) | C7—C11—H11A | 109.2 |
| C4—C3—Cl1 | 119.92 (13) | C10—C11—H11B | 109.2 |
| C2—C3—Cl1 | 118.46 (13) | C7—C11—H11B | 109.2 |
| C3—C4—C5 | 118.82 (15) | H11A—C11—H11B | 107.9 |
| C3—C4—H4A | 120.6 | O2—C12—O3 | 124.52 (14) |
| C5—C4—H4A | 120.6 | O2—C12—C13 | 118.42 (13) |
| C6—C5—C4 | 121.26 (15) | O3—C12—C13 | 117.06 (13) |
| C6—C5—H5A | 119.4 | C14—C13—C18 | 118.72 (13) |
| C4—C5—H5A | 119.4 | C14—C13—C12 | 120.11 (13) |
| C5—C6—C1 | 118.06 (13) | C18—C13—C12 | 121.12 (13) |
| C5—C6—C7 | 122.92 (13) | C15—C14—C13 | 120.57 (14) |

| | | | |
|---|---|---|---|
| C1—C6—C7 | 119.01 (12) | C15—C14—H14A | 119.7 |
| O1—C7—C6 | 110.41 (11) | C13—C14—H14A | 119.7 |
| O1—C7—C8 | 108.40 (12) | C16—C15—C14 | 120.02 (16) |
| C6—C7—C8 | 112.93 (11) | C16—C15—H15A | 120.0 |
| O1—C7—C11 | 106.31 (12) | C14—C15—H15A | 120.0 |
| C6—C7—C11 | 110.12 (12) | C17—C16—C15 | 120.19 (15) |
| C8—C7—C11 | 108.43 (11) | C17—C16—H16A | 119.9 |
| C9—C8—C7 | 112.22 (12) | C15—C16—H16A | 119.9 |
| C9—C8—H8A | 109.2 | C16—C17—C18 | 119.94 (14) |
| C7—C8—H8A | 109.2 | C16—C17—H17A | 120.0 |
| C9—C8—H8B | 109.2 | C18—C17—H17A | 120.0 |
| C7—C8—H8B | 109.2 | C17—C18—C13 | 120.54 (15) |
| H8A—C8—H8B | 107.9 | C17—C18—H18A | 119.7 |
| N1—C9—C8 | 111.13 (12) | C13—C18—H18A | 119.7 |
| C6—C1—C2—C3 | −0.1 (2) | C10—N1—C9—C8 | −56.75 (16) |
| C1—C2—C3—C4 | −0.1 (2) | C7—C8—C9—N1 | 55.69 (16) |
| C1—C2—C3—Cl1 | −179.23 (12) | C9—N1—C10—C11 | 57.50 (16) |
| C2—C3—C4—C5 | 0.0 (3) | N1—C10—C11—C7 | −57.35 (17) |
| Cl1—C3—C4—C5 | 179.15 (13) | O1—C7—C11—C10 | −61.50 (15) |
| C3—C4—C5—C6 | 0.3 (3) | C6—C7—C11—C10 | 178.89 (12) |
| C4—C5—C6—C1 | −0.5 (2) | C8—C7—C11—C10 | 54.87 (16) |
| C4—C5—C6—C7 | −179.69 (14) | O2—C12—C13—C14 | −173.36 (15) |
| C2—C1—C6—C5 | 0.4 (2) | O3—C12—C13—C14 | 6.1 (2) |
| C2—C1—C6—C7 | 179.65 (13) | O2—C12—C13—C18 | 3.9 (2) |
| C5—C6—C7—O1 | 114.43 (16) | O3—C12—C13—C18 | −176.65 (15) |
| C1—C6—C7—O1 | −64.80 (17) | C18—C13—C14—C15 | −1.0 (2) |
| C5—C6—C7—C8 | −7.11 (19) | C12—C13—C14—C15 | 176.34 (15) |
| C1—C6—C7—C8 | 173.66 (13) | C13—C14—C15—C16 | 0.7 (3) |
| C5—C6—C7—C11 | −128.48 (15) | C14—C15—C16—C17 | 0.6 (3) |
| C1—C6—C7—C11 | 52.29 (16) | C15—C16—C17—C18 | −1.5 (3) |
| O1—C7—C8—C9 | 61.09 (15) | C16—C17—C18—C13 | 1.1 (3) |
| C6—C7—C8—C9 | −176.24 (11) | C14—C13—C18—C17 | 0.1 (2) |
| C11—C7—C8—C9 | −53.92 (15) | C12—C13—C18—C17 | −177.21 (14) |

*Hydrogen-bond geometry (Å, °)*

Cg3 is the centroid of the C13–C18 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1NA···O3$^{i}$ | 0.87 (1) | 1.84 (1) | 2.6964 (17) | 166.(2) |
| O1—H1O···O3 | 0.82 (2) | 2.05 (2) | 2.7780 (16) | 147.(2) |
| N1—H1NB···O2$^{ii}$ | 0.86 (1) | 1.92 (1) | 2.7609 (18) | 166.(2) |
| C16—H16A···Cl1$^{iii}$ | 0.95 | 2.78 | 3.5268 (17) | 136 |
| C9—H9B···O1$^{i}$ | 0.99 | 2.46 | 3.3008 (19) | 143 |
| C1—H1A···Cg3$^{iv}$ | 0.95 | 2.70 | 3.554 (2) | 150 |

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

**Fig. 1**

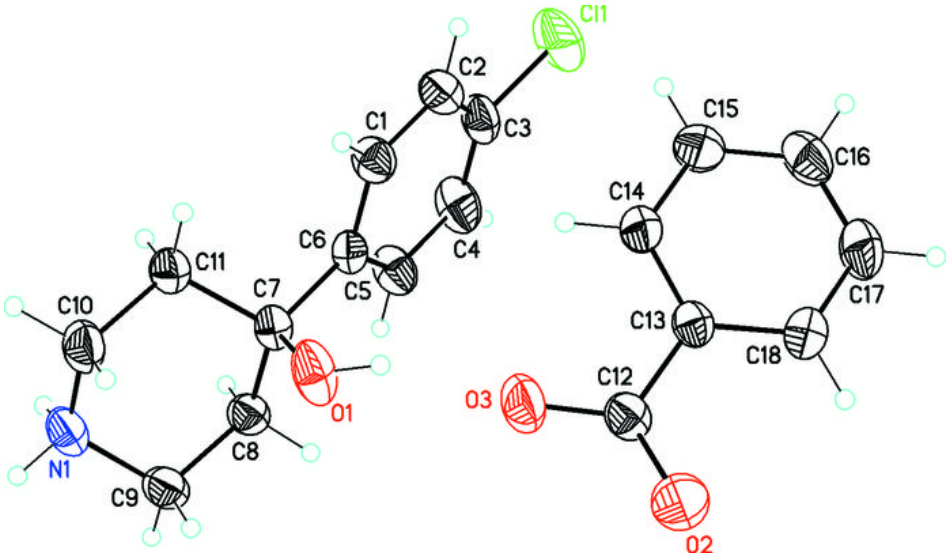
Cl1
C2
C1
C3
C15
C16
C14
C6
C4
C11
C7
C5
C10
C13
C17
C18
C12
O3
O1
C8
N1
C9
O2

**Fig. 2**