

Non-profiled  $\omega$  scans  
 Absorption correction:  $R_{\text{int}} = 0.012$   
 $\theta_{\text{max}} = 27.47^\circ$   
 $\psi$  scan (North *et al.*,  
 1968)  $h = 0 \rightarrow 6$   
 $T_{\text{min}} = 0.936$ ,  $T_{\text{max}} = 0.969$   $k = 0 \rightarrow 13$   
 3739 measured reflections  $l = -36 \rightarrow 36$   
 3355 independent reflections 3 standard reflections  
 frequency: 60 min  
 intensity decay: none

### Refinement

Refinement on  $F^2$   
 $R(F) = 0.035$   $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $wR(F^2) = 0.104$   $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{\AA}^{-3}$   
 $S = 1.053$   $\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$   
 3355 reflections Extinction correction: none  
 259 parameters Scattering factors from  
 All H-atom parameters International Tables for  
 refined Crystallography (Vol. C)

A difference Fourier map indicated the possible locations of the H atoms. They were inserted as such and refined freely without constraint resulting in the final figures of merit for the compound. The C—H distances and  $U_{\text{iso}}$  values for the refined H atoms are in the ranges 0.91 (3)–1.01 (3) Å and 0.034 (4)–0.077 (7) Å<sup>2</sup>, respectively.

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994). Cell refinement: CAD-4 EXPRESS. Data reduction: XCAD4 (Harms & Wocadlo, 1995). Program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997). Molecular graphics: ORTEP-3 for Windows (Farrugia, 1997). Software used to prepare material for publication: WinGX (Farrugia, 1998).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1217). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 1874–1877

## Hydrogen bonding in two piperidinium derivatives

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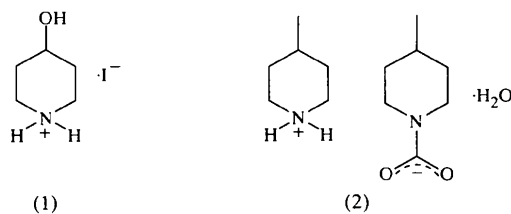
(Received 3 June 1999; accepted 12 July 1999)

### Abstract

4-Hydroxypiperidinium iodide, C<sub>5</sub>H<sub>12</sub>NO<sup>+</sup>·I<sup>−</sup>, displays normal bond lengths and angles, but its crystal packing is unusual in involving four-centre hydrogen bonds from one NH group to two OH groups and one iodide anion as acceptors. 4-Methylpiperidinium 4-methylpiperidine-*N*-carboxylate hydrate, C<sub>6</sub>H<sub>14</sub>N<sup>+</sup>·C<sub>7</sub>H<sub>12</sub>NO<sub>2</sub><sup>−</sup>·H<sub>2</sub>O, is formed when 4-methylpiperidine absorbs CO<sub>2</sub> from the air. The N atom of the anion is essentially planar, in contrast to that in the parent amine. Four classical hydrogen bonds, one from each donor, are involved in forming broad layers of residues parallel to the *xy* plane.

### Comment

We are interested in secondary interactions in various systems, *e.g.* in amine complexes of metals (Ahrens *et al.*, 1999) and in various amine derivatives (Jones & Ahrens, 1998; Freytag & Jones, 1999). We present here the structures of two piperidinium derivatives, namely 4-hydroxypiperidinium iodide, (1), and 4-methylpiperidinium 4-methylpiperidine-*N*-carboxylate hydrate, (2), one of which was obtained by chance.



4-Hydroxypiperidinium iodide, (1) (Fig. 1), displays the expected chair conformation of the cation, marginally flattened in the region of the N atom, with the hydroxy substituent equatorial. Bond lengths and angles may also be regarded as normal.

The crystal packing is largely determined by hydrogen bonding (Table 2); there are conventional, essentially linear, hydrogen bonds from N—H1' (axial) and the OH group as donors to iodide as acceptor, with H···A distances similar to those of 2.76 and 2.55 Å, respectively, quoted by Steiner (1998) for normalized

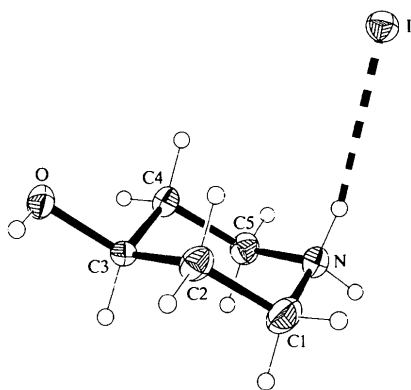


Fig. 1. The asymmetric unit of compound (1) in the crystal. Ellipsoids correspond to the 50% probability level.

H...I distances (N—H normalized to 1.01 Å and O—H to 0.98 Å; Mascall (1997) quotes 2.67 Å for N<sup>+</sup>—H...I and 2.72 Å for O—H...I, but it is not clear whether the D—H distances were normalized. A more unusual feature is a four-centre hydrogen bond from N—H2' (equatorial) to two OH groups and one iodide ion as acceptors. The H...A distances are rather long, as would be expected in such cases. [A referee points out that the H...A distances are not much less than the sum of the van der Waals radii, especially for the H...I contact of 3.31 (3) Å. This is true, but H2' would be expected to be a hydrogen-bond donor, and it has three neighbouring potential acceptors, making N—H...A angles of 122–137°. In the final analysis, it is impossible to prove whether any contact is genuinely a hydrogen bond, but we feel our interpretation is plausible.] The net effect of the hydrogen bonds is to connect the residues in a three-dimensional network (Fig. 2) with hydrophilic regions at  $y = 0, \frac{1}{2}, 1$  etc. There are also three C—H...I contacts that may be borderline cases of hydrogen bonds; these are given in Table 2, but are not shown in Fig. 2.

4-Methylpiperidinium 4-methylpiperidine-*N*-carboxylate hydrate, (2) (Fig. 3), furnishes an example of the principle that secondary amines can absorb carbon dioxide from the air to form *sec*-ammonium *N*-carboxylates

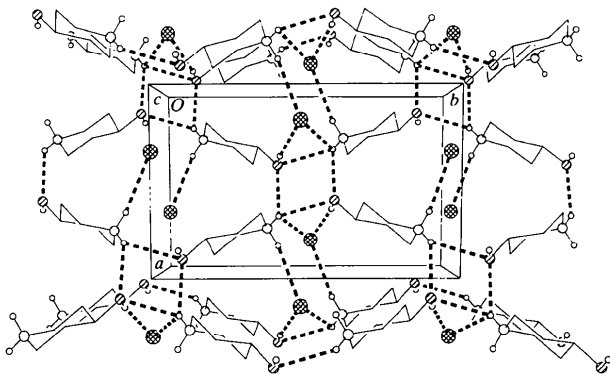


Fig. 2. Packing diagram of compound (1), viewed parallel to the  $z$  axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity. C atoms are drawn as points.

(carbamates). [It is conceivable that the formation of (2) is catalysed by the silver ions present during the crystallization (see *Experimental*). This was first noted by Knorr (1898) for morpholine, and the corresponding structure of morpholinium morpholine-4-carboxylate has been determined by Brown & Gray (1982), von Dreele *et al.* (1983) and Czugler *et al.* (1995). Other related adducts of secondary amines are known, but seem to be much less stable; their exact nature has not been established. The piperidine derivative was reported, but with few details (Schroth *et al.*, 1989).

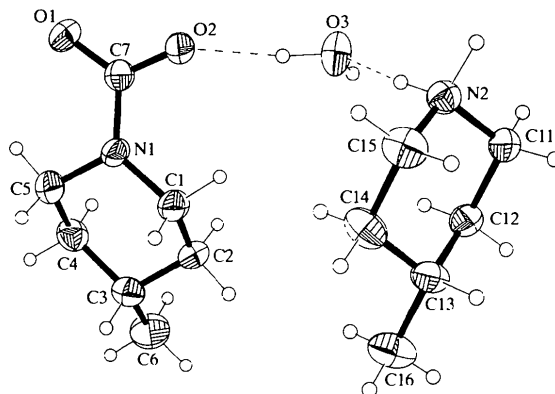


Fig. 3. The formula unit of compound (2) in the crystal. Ellipsoids correspond to the 50% probability level.

Both methyl groups in (2) are equatorial. The major effect on the anion ring structure is the planarization of the N atom; N1 lies only 0.052 (2) Å out of the plane of atoms C1, C5 and C7. The chair form is retained, however. The endocyclic C5—N1—C1 angle is still narrow at 114.40 (14)°, but the exocyclic angles are widened to 122–123°. The carboxylate group is essentially coplanar with the N1 centre [interplanar angle 2.32 (5)°]. The N1—C7 bond length of 1.373 (2) Å compares well with the value of 1.379 (2) Å observed by von Dreele *et al.* (1983) for the morpholine analogue.

The crystal packing involves four classical hydrogen bonds (Table 4). The water H atoms each act as donor

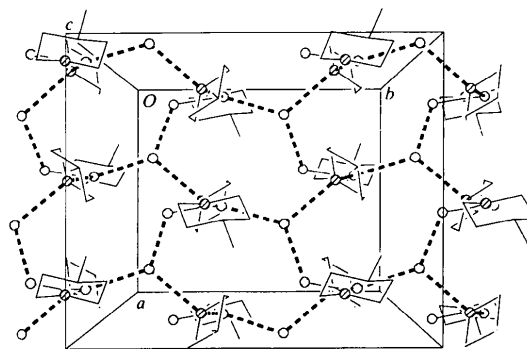


Fig. 4. Packing diagram of compound (2), viewed parallel to the  $z$  axis. H atoms have been omitted for clarity. Hydrogen bonds are drawn as thick dashed lines. C atoms are drawn as points.

to one carboxylate O atom, whereas the N—H groups donate to water and to the carboxylate O2 atom. The net effect is to form hydrophilic regions at  $z = \frac{1}{4}, \frac{3}{4}$  etc. (with hydrophobic regions at  $z = 0, \frac{1}{2}, 1$  etc.), wherein the residues associate to give two broad layers of ions per unit cell parallel to the  $xy$  plane. One of these is shown in Fig. 4. Additionally, there are two possible hydrogen bonds of the form C—H...O (Table 4).

## Experimental

For the preparation of (1), 4-hydroxypiperidine (Fluka) was treated with aqueous HI. After evaporation almost to dryness, the residue was crystallized from ethanol/petroleum ether. In an attempt to obtain a silver complex, silver trifluoromethanesulfonate (0.26 g) was dissolved in 4-methylpiperidine (5 ml). After one month, small quantities of crystals had formed, but these proved to be compound (2) rather than the desired complex.

### Compound (1)

#### Crystal data

C<sub>5</sub>H<sub>12</sub>NO<sup>+</sup>·I<sup>-</sup>M<sub>r</sub> = 229.06

Monoclinic

P2<sub>1</sub>/n

a = 7.7939 (8) Å

b = 12.3957 (14) Å

c = 7.978 (1) Å

β = 97.48 (1)°

V = 764.20 (15) Å<sup>3</sup>

Z = 4

D<sub>x</sub> = 1.991 Mg m<sup>-3</sup>D<sub>m</sub> not measured

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 62 reflections

θ = 3.0–12.5°

μ = 4.107 mm<sup>-1</sup>

T = 173 (2) K

Prism

0.4 × 0.3 × 0.2 mm

Colourless

#### Data collection

Siemens P4 diffractometer

ω scans

Absorption correction:

ψ scan (XEMP; Siemens, 1994a)

T<sub>min</sub> = 0.702, T<sub>max</sub> = 0.996

5555 measured reflections

1750 independent reflections

1595 reflections with

I &gt; 2σ(I)

R<sub>int</sub> = 0.023θ<sub>max</sub> = 27.5°

h = -4 → 10

k = -16 → 13

l = -10 → 10

3 standard reflections

every 247 reflections

intensity decay: none

#### Refinement

Refinement on F<sup>2</sup>R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.014wR(F<sup>2</sup>) = 0.035

S = 1.071

1750 reflections

86 parameters

H atoms treated by a

mixture of independent and constrained refinement

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0193P)<sup>2</sup> + 0.1398P]where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3(Δ/σ)<sub>max</sub> = 0.001Δρ<sub>max</sub> = 0.50 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.47 e Å<sup>-3</sup>

Extinction correction:

SHELXL97 (Sheldrick, 1997a)

Extinction coefficient:

0.0357 (8)

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °) for (1)

|             |              |             |             |
|-------------|--------------|-------------|-------------|
| N—C5        | 1.495 (2)    | O—C3        | 1.431 (2)   |
| N—C1        | 1.500 (2)    |             |             |
| C5—N—C1     | 113.32 (15)  |             |             |
| C5—N—C1—C2  | -54.8 (2)    | O—C3—C4—C5  | 179.78 (14) |
| N—C1—C2—C3  | 56.5 (2)     | C2—C3—C4—C5 | 58.45 (19)  |
| C1—C2—C3—O  | -177.95 (15) | C1—N—C5—C4  | 54.9 (2)    |
| C1—C2—C3—C4 | -59.0 (2)    | C3—C4—C5—N  | -56.2 (2)   |

Table 2. Hydrogen-bonding geometry (Å, °) for (1)

| D—H...A                               | D—H      | H...A    | D...A     | D—H...A |
|---------------------------------------|----------|----------|-----------|---------|
| N—H1 <sup>i</sup> ...O <sup>i</sup>   | 0.88 (2) | 2.70 (3) | 3.566 (2) | 169 (2) |
| N—H2 <sup>i</sup> ...O <sup>i</sup>   | 0.85 (3) | 2.30 (3) | 2.985 (2) | 137 (2) |
| N—H2 <sup>j</sup> ...O <sup>ii</sup>  | 0.85 (3) | 2.46 (3) | 3.106 (2) | 133 (2) |
| N—H2 <sup>k</sup> ...I <sup>iii</sup> | 0.85 (3) | 3.31 (3) | 3.835 (2) | 122 (2) |
| O—H3 <sup>i</sup> ...I <sup>iv</sup>  | 0.74 (2) | 2.76 (3) | 3.488 (1) | 169 (2) |
| C3—H3...I <sup>v</sup>                | 1.00     | 3.30     | 4.283 (2) | 167     |
| C5—H5A...I <sup>v</sup>               | 0.99     | 3.29     | 3.919 (2) | 123     |
| C5—H5B...I <sup>vi</sup>              | 0.99     | 3.39     | 4.047 (2) | 125     |

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

### Compound (2)

#### Crystal data

C<sub>6</sub>H<sub>14</sub>N<sup>+</sup>·C<sub>7</sub>H<sub>12</sub>NO<sub>2</sub><sup>-</sup>·H<sub>2</sub>OM<sub>r</sub> = 260.37

Orthorhombic

Pbca

a = 9.1378 (10) Å

b = 10.9924 (14) Å

c = 30.580 (3) Å

V = 3071.6 (6) Å<sup>3</sup>

Z = 8

D<sub>x</sub> = 1.126 Mg m<sup>-3</sup>D<sub>m</sub> not measured

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 45 reflections

θ = 4.8–11.9°

μ = 0.079 mm<sup>-1</sup>

T = 173 (2) K

Cone

0.4 × 0.3 × 0.3 mm

Colourless

#### Data collection

Siemens P4 diffractometer

ω scans

Absorption correction: none

3094 measured reflections

2694 independent reflections

1486 reflections with

I &gt; 2σ(I)

R<sub>int</sub> = 0.029θ<sub>max</sub> = 25°

h = -10 → 0

k = -13 → 2

l = -36 → 0

3 standard reflections

every 247 reflections

intensity decay: none

#### Refinement

Refinement on F<sup>2</sup>R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.038wR(F<sup>2</sup>) = 0.078

S = 0.797

2694 reflections

181 parameters

H atoms treated by a

mixture of independent and constrained refinement

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0346P)<sup>2</sup>]where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3(Δ/σ)<sub>max</sub> < 0.001Δρ<sub>max</sub> = 0.12 e Å<sup>-3</sup>Δρ<sub>min</sub> = -0.13 e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 3. Selected geometric parameters (Å, °) for (2)

|        |           |       |           |
|--------|-----------|-------|-----------|
| N2—C15 | 1.483 (2) | N1—C7 | 1.373 (2) |
| N2—C11 | 1.488 (2) | N1—C5 | 1.449 (2) |
| O1—C7  | 1.256 (2) | N1—C1 | 1.455 (2) |
| O2—C7  | 1.275 (2) |       |           |

|                 |              |          |             |
|-----------------|--------------|----------|-------------|
| C15—N2—C11      | 112.24 (15)  | O1—C7—O2 | 124.23 (17) |
| C7—N1—C5        | 122.29 (15)  | O1—C7—N1 | 118.19 (17) |
| C7—N1—C1        | 122.91 (14)  | O2—C7—N1 | 117.58 (16) |
| C5—N1—C1        | 114.40 (14)  |          |             |
| C15—N2—C11—C12  | 55.8 (2)     |          |             |
| N2—C11—C12—C13  | -55.9 (2)    |          |             |
| C11—C12—C13—C14 | 55.8 (2)     |          |             |
| C11—C12—C13—C16 | 179.68 (16)  |          |             |
| C12—C13—C14—C15 | -55.9 (2)    |          |             |
| C16—C13—C14—C15 | -179.73 (16) |          |             |
| C11—N2—C15—C14  | -55.9 (2)    |          |             |
| C13—C14—C15—N2  | 56.5 (2)     |          |             |
| C7—N1—C1—C2     | -131.71 (17) |          |             |
| C5—N1—C1—C2     | 55.4 (2)     |          |             |
| N1—C1—C2—C3     | -53.1 (2)    |          |             |
| C1—C2—C3—C4     | 52.1 (2)     |          |             |
| C1—C2—C3—C6     | 176.33 (15)  |          |             |
| C2—C3—C4—C5     | -52.5 (2)    |          |             |
| C6—C3—C4—C5     | -176.44 (16) |          |             |
| C7—N1—C5—C4     | 131.01 (17)  |          |             |
| C1—N1—C5—C4     | -56.0 (2)    |          |             |
| C3—C4—C5—N1     | 54.3 (2)     |          |             |
| C5—N1—C7—O1     | -4.3 (3)     |          |             |

Table 4. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ) for (2)

| D—H...A                      | D—H      | H...A    | D...A     | D—H...A |
|------------------------------|----------|----------|-----------|---------|
| O3—H1W...O2                  | 0.94 (2) | 1.79 (2) | 2.718 (2) | 172 (2) |
| O3—H2W...O1 <sup>i</sup>     | 0.92 (2) | 1.79 (2) | 2.712 (2) | 177 (2) |
| N2—H1...O2 <sup>ii</sup>     | 1.06 (2) | 1.63 (2) | 2.681 (2) | 167 (2) |
| N2—H2...O3                   | 0.97 (2) | 1.84 (2) | 2.796 (2) | 172 (2) |
| C15—H15A...O3 <sup>iii</sup> | 0.99     | 2.58     | 3.442 (2) | 146     |
| C12—H12A...O1 <sup>iv</sup>  | 0.99     | 2.58     | 3.303 (2) | 129     |

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

For (1), NH atoms were refined freely, the OH atom was treated as a rigid group (initial position from difference synthesis, allowed to rotate but not tip) and the remaining H atoms were treated as riding. The largest features of residual electron density ( $0.5 \text{ e \AA}^{-3}$ ) are  $0.8 \text{ \AA}$  from the I atom. A rigid-body libration correction (Schomaker & Trueblood, 1968) gave  $R_{\text{lib}} = 0.039$  and corrected bond lengths ( $\text{\AA}$ ) of N—C1 = 1.507, C1—C2 = 1.522, C2—C3 = 1.528, C3—C4 = 1.528, C4—C5 = 1.525, C5—N = 1.503 and C3—O = 1.435. For (2), the NH and OH atoms were refined freely, methyl group H atoms were refined as rigid groups (as for OH above) and the remaining H atoms were treated as riding. The freely refined N—H bonds are longer than expected (for fixed N—H of this type, the program system sets a distance of  $0.91 \text{ \AA}$ ), but attempts to idealize the N—H bond lengths with refinement restraints were unsuccessful (the program indicated that there were 'disagreeable restraints'). Clearly, if the N—H bonds are for any reason systematically too long, then the H...A hydrogen-bonding contacts will be too short. It is unlikely that X-ray methods alone can resolve this problem. A rigid-body libration correction (Schomaker & Trueblood, 1968) for the cation gave  $R_{\text{lib}} = 0.056$  and corrected bond lengths ( $\text{\AA}$ ) of N2—C11 = 1.499, C11—C12 = 1.519, C12—C13 = 1.529, C13—C14 = 1.531, C14—C15 = 1.530 and C15—N2 = 1.492. The anion was not amenable to correction, presumably because of rotation about the exocyclic N—C bond.

For both compounds, data collection: XSCANS (Fait, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structures: SHELXS97 (Sheldrick, 1997b); program(s) used to refine structures: SHELXL97 (Sheldrick, 1997a); molecular graphics: XP (Siemens, 1994b); software used to prepare material for publication: SHELXL97.

We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauff for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1355). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 1877–1881

## A rhodanine derivative and its cycloadduct with diphenyl nitrile imine

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## Abstract

The phenylmethylene substituent at the C5 position of 5-benzylidene-3-phenyl-2-thioxo-1,3-thiazolidin-4-one,  $\text{C}_{16}\text{H}_{11}\text{NOS}_2$ , (I), is virtually coplanar with the planar five-membered ring, whereas the phenyl group at N3 is almost orthogonal to this plane. The crystals of the