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# Phase transition of 3-dimethylammonio-1-phenylpropan-1-one chloride monohydrate

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**Key indicators**

Single-crystal X-ray study
$T$ = 173 K
Mean $\sigma$(C–C) = 0.002 Å
$R$ factor = 0.026
$wR$ factor = 0.066
Data-to-parameter ratio = 18.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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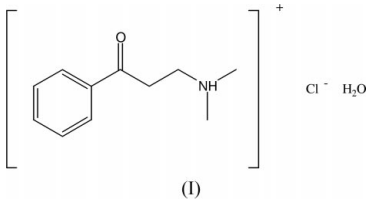
The title compound, $C_{11}H_{16}NO^{+}\cdot Cl^{-}\cdot H_2O$, undergoes a reversible phase transition between room temperature and 173 K. The structure at room temperature crystallizes in space group *Pbca*, with $Z' = 1$, whereas the inversion centre is lost upon cooling and the resulting space group is $P2_12_12_1$, with $Z' = 2$. The low-temperature structure is reported here.

## Comment

The structure of the title compound, (I), has already been determined at room temperature (Schlemper *et al.*, 1982), in space group *Pbca*, with $Z' = 1$. We collected a data set at 173 K and discovered that the reflections that should be absent for the different glide planes in *Pbca* (0*kl* for $k = 2n + 1$, *h*0*l* for $l = 2n + 1$ and *hk*0 for $h = 2n + 1$) were observed. The only extinction conditions that could be found were *h*00 for $h = 2n + 1$, 0*k*0 for $k = 2n + 1$ and 00*l* for $l = 2n + 1$, indicating three twofold screw axes. Therefore, the space group at 173 K must be $P2_12_12_1$, with $Z' = 2$. Refinement of the structure in $P2_12_12_1$ proceeded normally and did not show any signs of missed symmetry; for example, no correlation matrix element was larger that 0.5. A view of the two molecules in the asymmetric unit is shown in Fig. 1.




(I)

In order to check whether the phase transition is reversible or not, we collected a data set at room temperature for the previously cooled crystal. The space group was again *Pbca*, showing that the phase transition is reversible. The difference between the two phases is that the inversion centre, which is present at room temperature, is lost upon cooling. However,

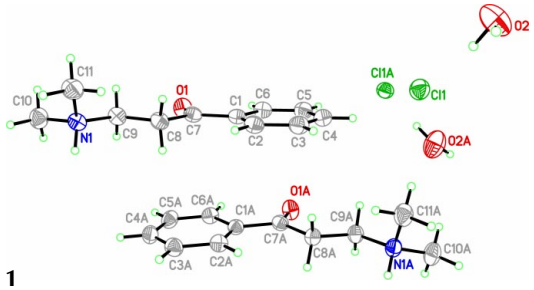



**Figure 1**
The asymmetric unit of the title compound, with the atom-numbering scheme; displacement ellipsoids are shown at the 50% probability level.

the two molecules in the asymmetric unit are still nearly centrosymmetrically related, as demonstrated by a least-squares fit (Fig. 2). When the phenyl rings are fitted, the side chain of molecule 1 is bent slightly downwards, whereas the side chain of molecule 2 is bent slightly upwards. The structure at room temperature represents an average of the two molecules in the asymmetric unit of (I) (Fig. 3). The Flack (1983) parameter [0.51 (3)] indicates possible inversion twinning.

## Experimental

The title compound was synthesized according to the method of Hünig *et al.* (1979).

*Crystal data*

$C_{11}H_{16}NO^+\cdot Cl^-\cdot H_2O$
$M_r$ = 231.71
Orthorhombic, $P2_12_12_1$
$a$ = 7.3010 (4) Å
$b$ = 11.0134 (7) Å
$c$ = 29.823 (2) Å
$V$ = 2398.0 (3) Å$^3$
$Z$ = 8
$D_x$ = 1.284 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 30714 reflections
$\theta$ = 2.1–27.5°
$\mu$ = 0.30 mm$^{-1}$
$T$ = 173 (2) K
Plate, colourless
0.48 × 0.42 × 0.24 mm

*Data collection*

Stoe IPDS-II two-circle diffractometer
$\omega$ scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
$T_{min}$ = 0.869, $T_{max}$ = 0.931
33 390 measured reflections
5557 independent reflections
4782 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.061
$\theta_{max}$ = 27.6°
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -38 \rightarrow 38$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.066
$S$ = 0.91
5557 reflections
296 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.11 e Å$^{-3}$
Absolute structure: Flack (1983), 2349 Friedel pairs
Flack parameter = 0.51 (3)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···Cl1$A^{i}$ | 0.931 (17) | 2.189 (17) | 3.0778 (13) | 159.3 (13) |
| O2—H2$B$···Cl1$A^{ii}$ | 0.84 (3) | 2.35 (3) | 3.1814 (16) | 168 (2) |
| N1$A$—H1$A$···Cl1$^{iii}$ | 0.92 (2) | 2.16 (2) | 3.0814 (14) | 175.2 (17) |
| O2$A$—H2$D$···O2$^{iv}$ | 0.90 (3) | 1.94 (3) | 2.837 (2) | 175 (2) |
| O2—H2$A$···Cl1 | 0.90 (3) | 2.23 (3) | 3.1138 (16) | 168 (3) |
| O2$A$—H2$C$···Cl1$A$ | 0.94 (3) | 2.32 (3) | 3.2353 (16) | 163 (2) |

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, 1+y, z$; (iii) $1+x, y, z$; (iv) $\frac{1}{2}+x, \frac{3}{2}-y, -z$.

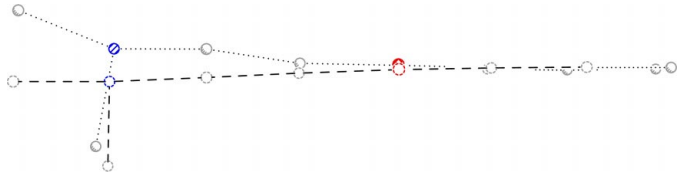


**Figure 2**
Least-squares fit of the two molecules in the asymmetric unit of (I); only the six C atoms of the phenyl ring were fitted. Cation 1 was inverted prior to fitting. Dashed lines: cation 1; dotted lines: cation 2.

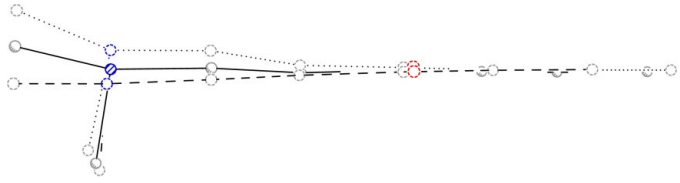


**Figure 3**
Least-squares fit of the two cations in the asymmetric unit of (I) and the room-temperature structure; only the six C atoms of the phenyl ring were fitted. Cation 1 was inverted prior to fitting. Dashed lines: cation 1; dotted lines: cation 2; solid lines: structure at room temperature.

H atoms bonded to C atoms were included with fixed individual displacement parameters [U(H) = 1.2 $U_{eq}$(C)] using a riding model, with $C_{aromatic}$—H = 0.95 Å, $C_{methyene}$—H = 0.99 Å and $C_{tertiary}$—H = 0.98 Å. H atoms bonded to N and O atoms were refined freely [N—H = 0.931 (17) and 0.92 (2) Å, and O—H = 0.84 (3)–0.94 (3) Å].

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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