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## The Twinned Crystal Structure of 3,4-Dimethylpyridine Hydrobromide at 157 K

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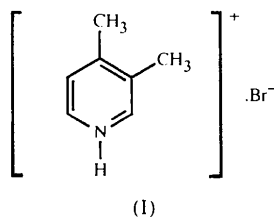
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### Abstract

Crystals of the title salt, 3,4-dimethylpyridinium bromide,  $C_7H_{10}N^+.Br^-$ , the structure of which has been determined by single-crystal X-ray diffraction at 157 K, appeared to be twinned. Transformation to a monoclinic *C*-centred cell was necessary in order to derive the twin law. But since the structure is in fact triclinic primitive, cell parameters, reflection indices, coordinates and the twin law have to be transformed back to carry out a proper refinement. The protonated N atom forms a hydrogen bond to the  $Br^-$  ion, and three further contacts shorter than 3 Å from the  $Br^-$  ion to aromatic H atoms can be found.

### Comment

The reaction of 3,4-dimethylpyridine with HBr, accidentally present in another reagent, led to the title compound, (I).



The geometry of the protonated 3,4-dimethylpyridine molecule is as expected. The  $Br^-$  ion is bound *via* a hydrogen bond to the protonated N atom [ $H1 \cdots Br1$  2.37 (9) Å and  $N1-H1 \cdots Br1$  163 (8)°] and three further contacts less than 3 Å from the  $Br^-$  ion to an aromatic H atom can be found [ $Br1 \cdots H2^i$  2.96 (2) Å and  $Br1 \cdots H2^i-C2^i$  123 (2)°;  $Br1 \cdots H5^{ii}$  2.83 (2) Å and  $Br1 \cdots H5^{ii}-C5^{ii}$  162 (2)°;  $Br1 \cdots H6^{iii}$  2.67 (2) Å and  $Br1 \cdots H6^{iii}-C6^{iii}$  163 (2)°; symmetry codes: (i)  $-x, -y+1, -z+2$ ; (ii)  $x, y, z+1$ ; (iii)  $-x, -y, -z+2$ ].

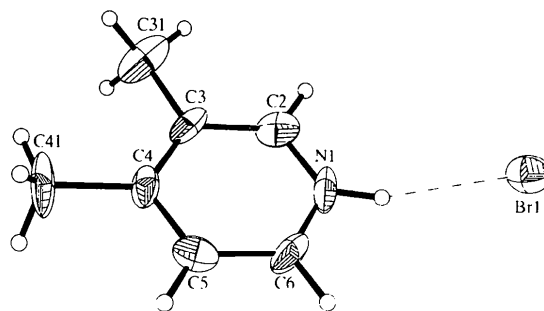


Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

### Experimental

Phenyldibromosilane was dissolved in acetonitrile under an atmosphere of dry argon. The phenyldibromosilane was prepared according to the procedure of Rühlmann & Mansfield (1964) and appears to have been contaminated with HBr. 3,4-Dimethylpyridine was added dropwise at room temperature with stirring. Single crystals of the title compound were obtained after slow evaporation.

#### Crystal data

$C_7H_{10}N^+.Br^-$   
 $M_r = 188.07$   
 Triclinic  
 $P\bar{1}$   
 $a = 7.3367$  (7) Å  
 $b = 7.7985$  (8) Å  
 $c = 8.3960$  (9) Å  
 $\alpha = 69.989$  (1)°  
 $\beta = 64.152$  (1)°  
 $\gamma = 89.997$  (2)°  
 $V = 399.85$  (7) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.562$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 2247 reflections  
 $\theta = 1-25^\circ$   
 $\mu = 5.055$  mm<sup>-1</sup>  
 $T = 157$  K  
 Plate  
 $0.25 \times 0.20 \times 0.05$  mm  
 Colourless

#### Data collection

Siemens CCD three-circle diffractometer

1243 reflections with  $I > 2\sigma(I)$

$\omega$ scans	$R_{\text{int}} = 0.046$
Absorption correction:	$\theta_{\text{max}} = 26.02^\circ$
empirical (SADABS;	$h = -8 \rightarrow 9$
Sheldrick, 1996)	$k = -9 \rightarrow 9$
$T_{\text{min}} = 0.534, T_{\text{max}} = 0.777$	$l = -10 \rightarrow 10$
3587 measured reflections	76 standard reflections
1386 independent reflections	intensity decay: none

### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.062$	$\Delta\rho_{\text{max}} = 1.415 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.132$	(0.93 $\text{\AA}$ from Br1)
$S = 1.080$	$\Delta\rho_{\text{min}} = -1.111 \text{ e } \text{\AA}^{-3}$
1386 reflections	(0.80 $\text{\AA}$ from C41)
87 parameters	Extinction correction: none
H atoms: see below	Scattering factors from
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2$	<i>International Tables for</i>
$+ 2.3936P]$	<i>Crystallography</i> (Vol. C)
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a_i^* a_j^*$			
	x	y	z	$U_{\text{eq}}$
Br1	0.0527 (2)	0.18021 (17)	1.12747 (18)	0.0430 (3)
N1	0.1897 (13)	0.3823 (12)	0.6779 (12)	0.037 (2)
C2	0.2587 (13)	0.5649 (14)	0.6080 (15)	0.035 (2)
C3	0.3202 (14)	0.6750 (12)	0.4160 (14)	0.032 (2)
C31	0.3942 (18)	0.8784 (15)	0.3438 (19)	0.059 (4)
C4	0.3021 (14)	0.5852 (13)	0.3043 (13)	0.031 (2)
C41	0.3515 (18)	0.6936 (18)	0.0992 (12)	0.059 (3)
C5	0.2296 (14)	0.3954 (13)	0.3833 (14)	0.036 (2)
C6	0.1763 (15)	0.2966 (13)	0.5721 (15)	0.036 (2)

The data collection nominally covered over a sphere of reciprocal space, by a combination of five sets of exposures; each set had a different  $\varphi$  angle for the crystal and each exposure covered  $0.3^\circ$  in  $\omega$ . The crystal-to-detector distance was 5.95 cm. Coverage of the unique set is over 88% complete to at least  $25^\circ$  in  $\theta$ . Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections. Cell reduction revealed that the cell could be transformed to a monoclinic  $C$ -centred one, but  $R_{\text{int}}$  and  $R_\sigma$  of 0.046 and 0.066, respectively, were significantly better for the triclinic case than for a monoclinic lattice (0.146 and 0.079). Furthermore, the data showed a suspicious value of 0.488 for  $\langle E^2 - 1 \rangle$ , which is a warning sign for twinning. The structure could be solved in the triclinic space group  $P\bar{1}$  with many difficulties. During refinement, large positional shifts led to unreasonable molecular geometry, and additional peaks appeared in the difference map. This behaviour is typical of merohedral twins, which are formed when the metric symmetry of the unit cell is higher than that of its contents. Under these conditions, a distinct symmetry element of the higher symmetry cell can be used as a twin law. Here the mirror plane perpendicular to the  $b$  axis of the monoclinic cell was selected and then expressed with respect to the triclinic axes by the matrix  $(\bar{1}00/010/\bar{1}01)$ . After allowing for twinning, the refinement proceeded normally; the twin scale factor was 0.647 (3). H atoms H1, H2, H31A, H41B and H5 were located by difference Fourier synthesis; the others were placed at calculated positions. While H1 was refined isotropically, the others were refined with fixed individual

displacement parameters [ $U(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$  or  $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ] using a riding model with  $\text{C}-\text{H}_{\text{methyl}} = 0.98$  or  $\text{C}-\text{H}_{\text{aromatic}} = 0.95 \text{ \AA}$ .

Data collection: SMART (Siemens, 1995). Cell refinement: SMART. Data reduction: SAINT (Siemens, 1995). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997). Molecular graphics: XP in SHELXTL-Plus (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1209). Services for accessing these data are described at the back of the journal.

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## 6-Chloro-4-(*o*-chlorobenzyl)-3-methylpyridazine at 173 K

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### Abstract

The title compound, C<sub>12</sub>H<sub>10</sub>Cl<sub>2</sub>N<sub>2</sub>, crystallizes with two independent molecules in the asymmetric unit, which differ significantly only in the two central torsion angles. The intermolecular packing is stabilized by short C—H...N contacts.