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1,4-Dimethylpyridinium triiodide

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Key indicators

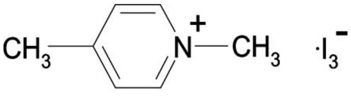
Single-crystal X-ray study
T = 288 K
Mean σ(C–C) = 0.013 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.105
Data-to-parameter ratio = 25.4

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

In an attempt to synthesize 1,4-dimethylpyridinium dodecatungstophosphate and 1,4-dimethylpyridinium dodecamolybdophosphate, the title compound, $C_7H_{10}N^+\cdot I_3^-$, was obtained as a by-product. The crystal structure contains a disordered 1,4-dimethylpyridinium cation and a triiodide anion.

Comment

The reaction of 1,4-dimethylpyridinium iodide (DMPI) and 12-tungstophosphate ($H_3PW_{12}O_{40}$) (or 12-molybdophosphate, $H_3PMo_{12}O_{40}$) in ethanol accidentally gave the title compound, (I). A search of the Cambridge Structural Database (Version 5.26 of November 2004; Allen, 2002) with respect to the cell parameters showed that the compound had not been reported previously. Thus, the structure of (I) has been fully characterized and is presented here.



(I)

The crystal structure of (I) is built from disordered cations and centrosymmetric anions (Fig. 1). The former are disordered, because there is an inversion centre at the centre of the pyridine ring. The sites labelled C1 and N1 in Fig.1 contain C or N atoms. Site C1 contains 68.11 (10)% C and 31.89 (10)% N, while site N1 contains 68.11 (10)% N and 31.89 (10)% C. This unequal disorder is different from the equal disorder found in related structures (Khan *et al.*, 1998; Fun *et al.*, 1996;

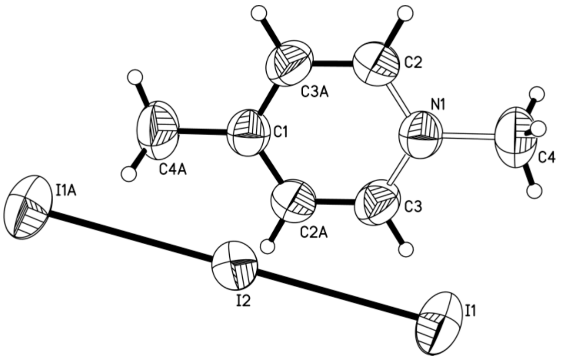


Figure 1
A view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitary radii. The suffix A denotes symmetry operation $-x$, $-y$, $-z$ for I1A and $1-x$, $1-y$, $-z$ for other atoms.

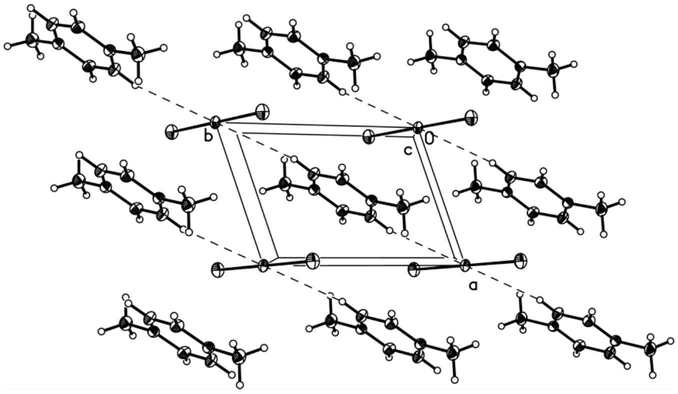


Figure 2
One layer of cations and anions viewed along the *c* axis; displacement ellipsoids are drawn at the 10% probability level. I···H contacts are shown as dashed lines.

Houghton & Wallwork, 1999; McKinney *et al.*, 1997), in which disordered *para*-C and *para*-N atoms could be treated as each having occupancies of 50%. It is also different from the situation in another dimethylpyridine salt (Tan *et al.*, 2004), where the cation is disordered over two different orientations related to each other by a small rotation.

The I_3^- anion of (I) is exactly linear since atom I2 lies on a centre of symmetry.

The crystal structure of (I) consists of alternate layers of cations and anions. One such layer viewed along the *c* axis is shown in Fig. 2. The $I\cdots I^{i}$ contact between layers is 3.9921 (16) Å and the $I\cdots H^{ii,iii}$ contact within the layer is 3.156 (8) Å [symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $x, y-1, z$; (iii) $-x, 1-y, -z$].

Experimental

All organic solvents and common materials used for the synthesis were of reagent grade and used without further purification. $H_3PMo_{12}O_{40}$ and $H_3PW_{12}O_{40}$ were prepared according to literature methods (Rocchiccioli-Deltcheff *et al.*, 1983; Keggin, 1934). 1,4-Dimethylpyridinium iodide (DMPI) was prepared following the method of Ren *et al.* (2000). Compound (I) was prepared by reacting a 0.01 *M* solution of DMPI in ethanol with a 0.01 *M* solution of $H_3PMo_{12}O_{40}$ in ethanol (or a 0.01 *M* solution of $H_3PW_{12}O_{40}\cdot 24H_2O$ in ethanol) in the molar ratio 3:1. The precipitate, a charge-transfer salt between DMPI and $H_3PMo_{12}O_{40}$ (or $H_3PW_{12}O_{40}$), was filtered off. On evaporation of the filtrate, brilliant brown crystals were obtained, which could be purified by recrystallization. Elemental analysis, calculated: C 17.2, H 2.1, N 2.9%; found: C 17.7, H 2.0, N 3.1%.

Crystal data

$C_7H_{10}N^+\cdot I_3^-$
$M_r = 488.86$
Triclinic, $P\overline{1}$
$a = 5.469$ (2) Å
$b = 7.786$ (3) Å
$c = 8.463$ (3) Å
$\alpha = 108.604$ (5)°
$\beta = 92.357$ (6)°
$\gamma = 107.546$ (5)°
$V = 321.9$ (2) Å^3
$Z = 1$
$D_x = 2.525$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 380 reflections
$\theta = 7.5$–27.0°
$\mu = 7.24$ mm^{-1}
$T = 288$ (2) K
Thick plate, brown
0.36 × 0.18 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.225$, $T_{\max} = 0.557$
1956 measured reflections
1346 independent reflections
925 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.0°$
$h = -4 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.105$
$S = 0.93$
1346 reflections
53 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.75$ e Å^{-3}
$\Delta\rho_{\min} = -0.84$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C3	1.349 (10)	C2—C3^{iv}	1.368 (12)
N1—C2	1.351 (10)	I1—I2	2.916 (1)
N1—C4	1.488 (11)		
C3—N1—C2	117.8 (7)	N1—C2—C3^{iv}	120.9 (7)
C3—N1—C4	120.5 (8)	N1—C3—C2^{iv}	121.4 (8)
C2—N1—C4	121.7 (7)		

Symmetry code: (iv) $-x+1, -y+1, -z$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.96 Å, and with $U_{\rm iso}$(H) = 1.5 $U_{\rm eq}$(C) for methyl H atoms and 1.2 $U_{\rm eq}$(C) for other H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97*.

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