O1—C2—N3	108.5 (2)	C1″—C2′—C3′	117.9 (2)
C1′—N3—C2	126.4 (2)	C1″—C2′—C1′	124.3 (2)
C1′—N3—C4	122.47 (15)	C3′—C2′—C1′	117.4 (2)
C2—N3—C4	111.09 (14)	O4—C3′—O5	124.3 (2)
N3—C4—C5	99.70 (14)	O4—C3′—C2′	125.2 (2)
N3—C4—C41	112.26 (14)	O5—C3′—C2′	110.5 (2)
C5—C4—C41	114.8 (2)	C3′—O5—C4′	116.2 (2)
O1—C5—C4	104.94 (14)		

Intensities were measured with a 'learnt profile' method (Clegg, 1981). The space group was determined from the Laue symmetry, the systematically absent reflections and the average value of $|E^2 - 1|$ using program *XPREP* (Sheldrick, 1994). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). All non-H atoms were refined anisotropically using *SHELXL*93 (Sheldrick, 1993). H atoms were refined using a riding model. The isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameter of the atom they are attached to. The absolute structure could not be determined as no significant anomalous scatterer was present. The known absolute structure of the educt was thus assumed (Tietze & Schünke, 1995). The floating origin restraint was applied (Flack & Schwarzenbach, 1988).

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1277). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-Amino-3-methylpyridinium Bromide Monohydrate

Tom Coffey, Ward T. Robinson and Mark M. Turnbull†

Department of Chemistry, University of Canterbury, Private Bag 4800, Christchurch, New Zealand

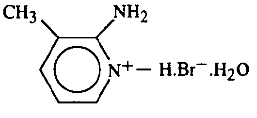
(*Received 4 April 1995; accepted 10 August 1995*)

Abstract

The title compound, $C_6H_9N_2^+.Br^-.H_2O$, was crystallized from water by slow evaporation. The 2-amino-3-methylpyridinium cation shows bond lengths and angles comparable with those observed in more complex counterions.

Comment

As a result of our interest in low-dimensional magnetic lattices, we have been studying complexes of the family $(LH)_2MX_4$, where M is a 2+ first row transition metal ion, X is Cl or Br, and L is an organic base. These compounds are well known to pack in the crystal lattice such that low-dimensional magnetic lattices, arising from van der Waals contacts between the MX_4 ions, are frequently obtained (for examples see Place & Willett, 1987*a*,*b*). The nature of these contacts is determined by the organic base L. Several complexes, where L is 2-amino-3-methylpyridine, have been prepared and we were interested in seeing whether the pronounced effect of L on the packing of the anions was reciprocated by the anions altering the structure of L in the crystal lattice. The crystal structure determination of 2-amino-3-methylpyridinium bromide monohydrate, (I), was thus undertaken.



(I)

The general structure of the title material is unremarkable, with bond lengths and angles comparable with other structures containing the protonated moiety, where the anions are copper or palladium-based complex ions (Place & Willett, 1987*b*; Grigereit *et al.*, 1987; Roman, Beitia & Luque, 1993). The bonds were slightly longer than those of the neutral compound (Espenbetov, Struchkov, Poplavskaya & Kurman-

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† Permanent address: Department of Chemistry, Clark University, 950 Main St., Worcester, MA 01610, USA.

galieva, 1985), except for the C11—N12 bond, which was slightly shorter (0.05 Å). The pyridinium proton was located in the difference Fourier map and refined using a riding model. The bromide ion is held in the lattice *via* hydrogen bonds to the cation (Br···H—N11 3.44 and Br···H—N12 3.36 Å) and the water molecule (Br···H—O1 3.34 Å). The water molecule is similarly linked to the cation (O1···H—N12 2.86 Å).

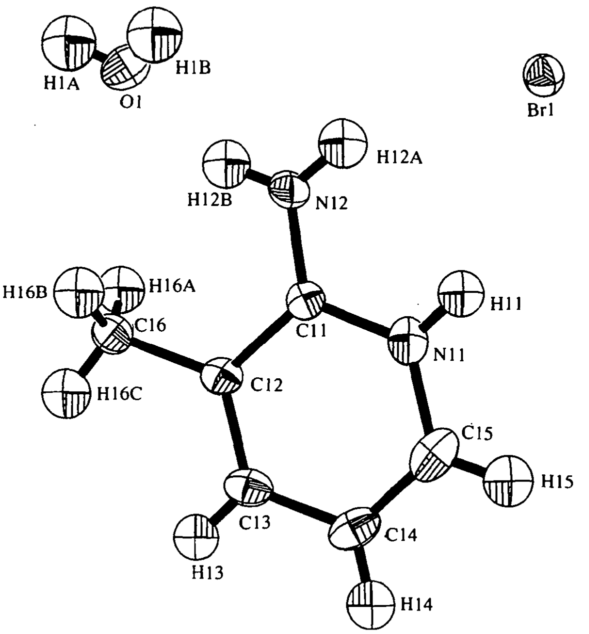

Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids.

Experimental

Crystals of (I) were grown by slow evaporation of an aqueous solution containing a 1:1 molar ratio of 2-amino-3-methylpyridine and hydrogen bromide.

Crystal data

$C_6H_9N_2^+.Br^-.H_2O$
$M_r = 207.08$
Triclinic
$P\bar{1}$
$a = 6.9820\,(10)$ Å
$b = 8.106\,(6)$ Å
$c = 8.3890\,(10)$ Å
$\alpha = 106.75\,(4)°$
$\beta = 108.51\,(4)°$
$\gamma = 97.30\,(3)°$
$V = 418.4\,(3)$ Å^3
$Z = 2$
$D_x = 1.644$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 17 reflections
$\theta = 4.49–12.46°$
$\mu = 4.850$ mm^{-1}
$T = 133\,(2)$ K
Plate
0.70 × 0.35 × 0.05 mm
Transparent

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: empirical *via* ψ scans (Siemens, 1990)
$T_{min} = 0.28$, $T_{max} = 0.70$
1828 measured reflections
1398 independent reflections
1209 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.0256$
$\theta_{max} = 24.99°$
$h = -8 \rightarrow 2$
$k = -9 \rightarrow 9$
$l = -9 \rightarrow 9$
3 standard reflections monitored every 97 reflections
intensity decay: 7.52%

Refinement

Refinement on F^2
$R(F) = 0.0428$
$wR(F^2) = 0.1279$
$S = 0.998$
1397 reflections
100 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2 + 0.9364P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.068$
$\Delta\rho_{max} = 0.926$ e Å^{-3}
$\Delta\rho_{min} = -0.745$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Br1	−0.17453 (8)	0.40056 (7)	0.10804 (7)	0.0256 (2)
O1	−0.3360 (7)	−0.2963 (6)	−0.0846 (6)	0.0345 (10)
N11	−0.1654 (7)	0.1866 (6)	−0.3293 (6)	0.0256 (10)
C11	−0.2109 (8)	0.0208 (7)	−0.3248 (6)	0.0201 (10)
N12	−0.1857 (7)	0.0016 (6)	−0.1675 (5)	0.0268 (10)
C12	−0.2766 (8)	−0.1249 (7)	−0.4882 (6)	0.0221 (11)
C13	−0.2983 (8)	−0.0879 (8)	−0.6423 (7)	0.0264 (12)
C14	−0.2550 (9)	0.0867 (8)	−0.6404 (8)	0.0289 (12)
C15	−0.1863 (8)	0.2217 (8)	−0.4828 (8)	0.0295 (12)
C16	−0.3127 (9)	−0.3088 (7)	−0.4842 (7)	0.0256 (11)

Table 2. *Selected geometric parameters (Å, °)*

N11—C11	1.355 (7)	C12—C13	1.376 (7)
N11—C15	1.365 (7)	C12—C16	1.492 (8)
C11—N12	1.333 (7)	C13—C14	1.403 (8)
C11—C12	1.419 (7)	C14—C15	1.344 (8)
C11—N11—C15	123.8 (5)	C13—C12—C16	123.5 (5)
N12—C11—N11	118.9 (5)	C11—C12—C16	118.9 (4)
N12—C11—C12	123.0 (5)	C12—C13—C14	122.0 (5)
N11—C11—C12	118.1 (5)	C15—C14—C13	119.0 (5)
C13—C12—C11	117.6 (5)	C14—C15—N11	119.5 (5)

Data collection: *XSCANS* (Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *SHELXTL/PC* (Siemens, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, together with a stereopacking diagram, have been deposited with the IUCr (Reference: TA1032). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,8-Dithia-1-phospha-5-arsabicyclo[3.3.0]-octane 1-Oxide

KLAUS JURKSCHAT[a] AND DIETER SCHOLLMEYER[b]

[a]*Lehrstuhl für Anorganische Chemie II, Universität Dortmund, Otto-Hahn-Strasse 6, 44227 Dortmund, Germany, and* [b]*Institut für Organische Chemie der Universität Mainz, Saarstrasse 21, 55099 Mainz, Germany*

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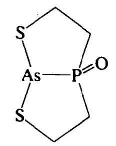
Abstract

The title compound, $C_4H_8AsOPS_2$, obtained by oxidation of $As(SCH_2CH_2)_2P$, shows a bicyclooctane structure with an As—P bond distance of 2.345 (1) Å. The compound shows weak intermolecular As···O interactions of 2.882 (2) Å.

Comment

In 1982, we reported a number of 2,8-dithia-1-phospha-5-arsabicyclo[3.3.0]octanes (Jurkschat, Mügge, Tzschach, Uhlig & Zschunke, 1982). A detailed conformational study of these compounds in solution, followed later by NMR spectroscopic analysis (Zschunke, Mügge, Meyer, Tzschach & Jurkschat, 1983), suggested that they exist in a preferred conformer with strong puckering of the fused five-membered rings. No single crystals of any of these compounds has been obtained thus far. However, oxide $As(SCH_2CH_2)_2PO$, hereafter (1), was obtained as a minor product by recrystallization of $As(SCH_2CH_2)_2P$ from toluene in the presence of air. Its ^{31}P NMR spectrum shows a singlet at 128.8 p.p.m., which is consistent with the presence of pentavalent phosphorus.



(1)

The twist conformation of the two fused five-membered rings, As—S1—C1—C2—P1 [$\varphi(2) = -118.9 (4)°$] and As—S2—C4—C3—P1 [$\varphi(2) = 94.6 (3)°$], of (1) in the solid state resembles that predicted for $As(SCH_2CH_2)_2P$ in solution (Zschunke *et al.*, 1983). The As—P distance of 2.345 (1) Å may be regarded as normal; it resembles those reported for other As—P compounds (Baudler, Aktalay, Heinlein & Tebbe, 1982; Weber, Bungardt & Boese, 1989; Jutzi, Meyer, Opiela, Olmstead & Power, 1990; Märkl, Dietl, Ziegl & Nuber, 1988; Sheldrick, 1975). The same holds for the As—S bond distances (Dräger, 1975). An interesting feature of the structure of (1) is the weak intermolecular As···O interaction of 2.882 (2) Å resulting in head-to-tail dimers. This distance is at the upper limit of such contacts (Dräger, 1975) and reflects the ambivalent character of (1), with weak Lewis acidity at the As atom and Lewis basicity at the P═O function. The P═O bond length of 1.490 (2) Å is not affected by the weak coordination and resembles those found in functional substituted organotin compounds (Weichmann, Rensch, Dargatz & Meunier-Piret, 1989; Weichmann & Meunier-Piret, 1993).

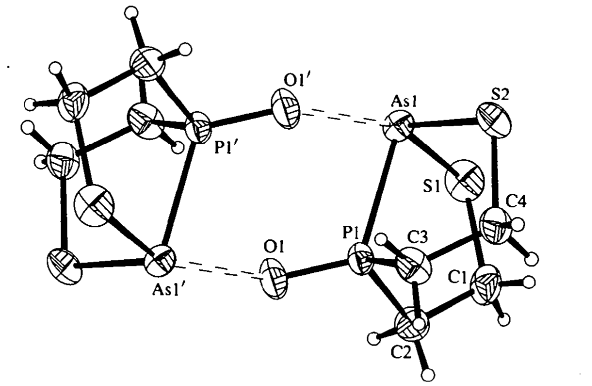


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title compound showing the atom-numbering scheme with 50% probability ellipsoids.

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