

to thank FAPESP (proc. 91/2890-2) for a scholarship, the Weizemann Institute of Science for providing X-ray facilities and Dr F. Frolow for the data collection.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55753 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1029]

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*Acta Cryst.* (1993). **C49**, 1017–1018

## Triphenylphosphonium Bromide

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(Received 28 July 1992; accepted 10 December 1992)

### Abstract

The structure consists of unique  $[\text{C}_{18}\text{H}_{15}\text{PH}]^+\text{Br}^-$  ion pairs, which are bisected by a crystallographic mirror plane. The proton is bonded to the P atom [P—H 1.61 (9) Å] and is directed towards a Br anion [P $\cdots$ Br 2.20 (9) Å]. Although the structure is ionic, comparison with phosphonium bromide  $[\text{PH}_4]^+\text{Br}^-$ , P $\cdots$ Br 4.029 (4) Å; Schröder & Rush (1971). *J. Chem. Phys.*

**54**, 1968–1973] indicates a much greater cation–anion interaction, analogous to molecular  $\text{Ph}_3\text{PBr}_2$  [Bricklebank, Godfrey, McAuliffe, Mackie & Pritchard (1992). *J. Chem. Soc. Chem. Commun.* pp. 355–356], which has a novel four-coordinate spoke structure with significant covalent Br–Br interaction.

### Comment

The phosphonium salt reported here is an example of the type of product which can be expected if the anhydrous anaerobic conditions required for reacting metal powders with dihalophosphoranes are not rigorously maintained. The formation of triphenylphosphonium halides from triphenyldihalophosphanes in moist solvents has been reported previously (Beveridge, Harris & Payne, 1966). The title compound was isolated from the filtrate of the reaction of  $\text{Ph}_3\text{PBr}_2$  with Cr-metal powder, which had been penetrated by moisture (Godfrey, Kelly, McAuliffe, Mackie, Matear & Pritchard, 1991). Despite their ubiquity, the only triphenylphosphonium halide structure reported to date is that of (2-hydroxyphenyl)diphenylphosphonium bromide (Schmutzler, Schomburg, Bartsch & Stelzer, 1984). The presence of the hydroxy substituent in this compound involves the P—H group in hydrogen bonding, which is impossible in the title compound.

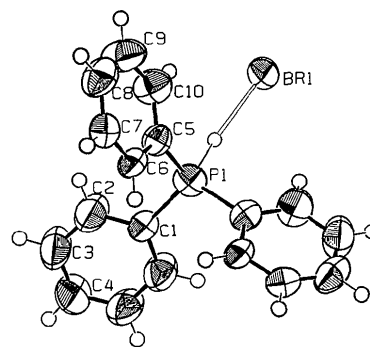


Fig. 1. The title molecule, including atomic numbering scheme, drawn using *ORTEPII* (Johnson, 1976).

### Experimental

#### Crystal data

$\text{C}_{18}\text{H}_{16}\text{P}^+\text{Br}^-$   
 $M_r = 343.2$   
Orthorhombic  
*Pnma*  
 $a = 10.951$  (5) Å  
 $b = 12.269$  (3) Å  
 $c = 11.754$  (4) Å  
 $V = 1579$  (1) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.443$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107$  Å  
Cell parameters from 25 reflections  
 $\theta = 10.42$ – $24.52^\circ$   
 $\mu = 2.66$  mm<sup>-1</sup>  
 $T = 296$  K  
Trapezoid  
 $0.30 \times 0.20 \times 0.20$  mm  
Colourless

**Data collection**

CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 refined from  $\Delta F$  (DI-  
 FABS; Walker & Stuart,  
 1983)  
 $T_{\min} = 0.60$ ,  $T_{\max} = 1.19$   
 1477 measured reflections  
 1456 independent reflections  
 676 observed reflections  
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.024$   
 $\theta_{\text{max}} = 24.0^\circ$   
 $h = 0 \rightarrow 13$   
 $k = 0 \rightarrow 14$   
 $l = 0 \rightarrow 12$   
 3 standard reflections  
 monitored every 150  
 reflections  
 intensity variation:  
 none

**Refinement**

Refinement on  $F$   
 Final  $R = 0.0520$   
 $wR = 0.0562$   
 $S = 1.278$   
 676 reflections  
 100 parameters  
 H atoms in calculated po-  
 sitions except for H at-  
 tached to P which was lo-  
 cated in a difference map  
 and refined isotropically  
 $w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{\text{max}} = 0.0109$   
 $\Delta\rho_{\text{max}} = 0.48 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.38 \text{ e } \text{Å}^{-3}$   
 Extinction correction:  
 Zachariasen type 2 Gaus-  
 sian isotropic  
 Extinction coefficient:  
 $1.88209 \times 10^{-7}$   
 Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallography*  
 (1974, Vol. IV)

Data collection: Enraf-Nonius CAD-4AJ. Cell refinement:  
 Enraf-Nonius CAD-4AJ. Data reduction: *TEXSAN PROCESS*  
 (Molecular Structure Corporation, 1985). Program(s) used to  
 solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used  
 to refine structure: *TEXSAN LS* (Molecular Structure Corpora-  
 tion, 1985). Software used to prepare material for publication:  
*TEXSAN FINISH* (Molecular Structure Corporation, 1985).

Lists of structure factors, anisotropic thermal parameters, H-atom coor-  
 dinates and complete geometry have been deposited with the British Li-  
 brary Document Supply Centre as Supplementary Publication No. SUP  
 55968 (16 pp.). Copies may be obtained through The Technical Editor,  
 International Union of Crystallography, 5 Abbey Square, Chester CH1  
 2HU, England. [CIF reference: HA1023]

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*Acta Cryst.* (1993). **C49**, 1018–1022

## Structures of Two Ribonucleotide Reductase Inhibitors: 1-Hydroxy-1-methylurea and 1-Hydroxy-3-methylurea

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(Received 15 September 1992; accepted 27 November 1992)

**Abstract**

The conformation of O—N—C=O in both 1-hydroxy-1-methylurea and 1-hydroxy-3-methylurea is antiperiplanar and is stabilized by intramolecular NH···O hydrogen bonding. Pyramidalization of the N atom carrying the hydroxy group is observed in both compounds and the N—O bonds are twisted by about 17° out of the N—(C=O)—N urea planes. The methyl C atom of the N—(C=O)—N urea planes. The methyl C atom of the 1-hydroxy-1-methylurea is not situated in the urea plane but the corresponding atom in 1-hydroxy-3-methylurea is included in the plane. 1-Hydroxy-3-methylurea is consequently the more planar of the two compounds.

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

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}}$
Br1	0.5091 (1)	0.2500	−0.04161 (10)	0.0655
P1	0.3820 (3)	0.2500	0.2597 (3)	0.0640
C1	0.4723 (9)	0.2500	0.3880 (11)	0.0550
C2	0.5106 (9)	0.1552 (8)	0.4334 (9)	0.0957
C3	0.5831 (11)	0.1551 (9)	0.5297 (11)	0.1107
C4	0.6163 (13)	0.2500	0.5764 (11)	0.0984
C5	0.2946 (7)	0.1274 (7)	0.2525 (8)	0.0568
C6	0.2107 (8)	0.1033 (8)	0.3387 (9)	0.0634
C7	0.1508 (8)	0.0044 (9)	0.3330 (8)	0.0674
C8	0.1696 (10)	−0.0691 (8)	0.2484 (10)	0.0866
C9	0.2551 (11)	−0.0446 (11)	0.1651 (11)	0.0989
C10	0.3167 (9)	0.0541 (11)	0.1669 (9)	0.0825

Table 2. Geometric parameters ( $\text{Å}$ ,  $^\circ$ )

P1—C1	1.80 (1)	C5—C10	1.37 (1)
P1—C5	1.785 (8)	C6—C7	1.38 (1)
C1—C2	1.35 (1)	C7—C8	1.36 (1)
C2—C3	1.38 (1)	C8—C9	1.39 (2)
C3—C4	1.34 (1)	C9—C10	1.39 (2)
C5—C6	1.40 (1)		
C1—P1—C5	109.5 (4)	P1—C5—C6	119.8 (7)
C5—P1—C5 <sup>i</sup>	114.8 (5)	P1—C5—C10	119.5 (7)
P1—C1—C2	120.1 (6)		

Symmetry code: (i)  $x, \frac{1}{2} - y, z$ .