

**(1,3-Dioxan-2-ylmethyl)triphenylphosphonium  
bromide monohydrate****Yong-Zhong Wu,<sup>a\*</sup> Hong-Yun Li<sup>b</sup>  
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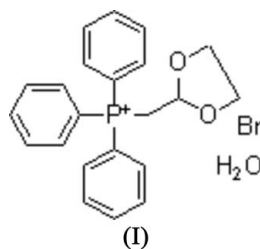
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In the crystal structure of the title phosphonium salt,  $C_{22}H_{22}O_2P^+ \cdot Br^- \cdot H_2O$ , the triphenylphosphonium group has three P—C bonds to phenyl rings which are equal within experimental error [mean 1.782 (3) Å]. The P atom is also attached directly to the C atom of a 1,3-dioxan-2-ylmethyl group with a longer P—C bond of 1.800 (3) Å. The C—C bond of the dioxane ring [1.418 (5) Å] is shorter than the normal bond distance. The asymmetric unit contains two bromide anions on twofold rotation axes to balance the charge of the cation.

**Comment**

As an inhibitor against the acid corrosion of iron, steel, zinc, and aluminium and its alloys with high efficiency, (1,3-dioxan-2-ylmethyl)triphenylphosphonium bromide has a broad range of applications and has been studied widely. The alkaline hydrolysis of the title phosphonium salt, (I), yielded triphenylphosphine oxide, cyclopentanecarboxylic acid and ethyl cyclopentanecarboxylate (Araya-Maturana & Castaneda, 1993). We report here the crystal structure of (1,3-dioxan-2-ylmethyl)triphenylphosphonium bromide monohydrate, (I) (Fig. 1).



The cation of (I) exhibits the usual tetrahedral coordination at P. The P—C bond lengths between the phenyl rings and P compare well with those reported previously for other triphenylphosphonium salts (Ferguson *et al.*, 1988; Boys *et al.*, 1995). The P atom is attached directly to the 1,3-dioxan-2-ylmethyl group, with a longer P—C bond of 1.800 (3) Å. The tetrahedral C—P—C angles range from 105.46 (13) to 112.84 (13)°. Atom C20 in the dioxolane ring has a large deviation [0.187 (3) Å] from the least-squares plane through O1/C21/C22/O2. The C—O bonds in the dioxolane ring have lengths in the range 1.395 (4)–1.427 (4) Å. The C—C bond of the dioxolane ring has a length of 1.418 (5) Å, shorter than the normal bond length of 1.49 Å. Molecules are linked to each other by van der Waals forces, forming a three-dimensional network. Bromide anions lie on twofold rotation axes, while the cations and water molecules are in general positions.

Experimental

The title compound, (I), was prepared as described by Cresp *et al.* (1974). 2-Bromomethyl-1,3-dioxolane and triphenylphosphine were heated in a steam bath. The cooled product was separated by filtration, washed well with dry diethyl ether and dried under vacuum to afford the triphenylphosphonium salt. A sample crystallized from dichloromethane–dry diethyl ether (3:1 *v/v*) formed prisms.

Crystal data

$C_{22}H_{22}O_2P^+ \cdot Br^- \cdot H_2O$   $D_x = 1.445 \text{ Mg m}^{-3}$   
 $M_r = 447.29$  Mo  $K\alpha$  radiation  
 Monoclinic,  $C2/c$  Cell parameters from 2671 reflections  
 $a = 17.322(4) \text{ \AA}$   $\theta = 2.5\text{--}24.1^\circ$   
 $b = 14.451(3) \text{ \AA}$   $\mu = 2.10 \text{ mm}^{-1}$   
 $c = 17.978(4) \text{ \AA}$   $T = 298(2) \text{ K}$   
 $\beta = 114.003(3)^\circ$  Block, colourless  
 $V = 4111.0(16) \text{ \AA}^3$   $0.45 \times 0.18 \times 0.16 \text{ mm}$   
 $Z = 8$

Data collection

Bruker SMART CCD area-detector 3636 independent reflections  
 diffractometer 2808 reflections with  $I > 2\sigma(I)$   
 $\varphi$  and  $\omega$  scans  $R_{int} = 0.038$   
 Absorption correction: multi-scan  $\theta_{max} = 25.0^\circ$   
 (SADABS; Sheldrick, 1997)  $h = -20 \rightarrow 18$   
 $T_{min} = 0.443, T_{max} = 0.716$   $k = -13 \rightarrow 17$   
 10117 measured reflections  $l = -17 \rightarrow 21$

Refinement

Refinement on  $F^2$  H-atom parameters constrained  
 $R[F^2 > 2\sigma(F^2)] = 0.044$   $w = 1/[\sigma^2(F_o^2) + (0.0678P)^2]$   
 $wR(F^2) = 0.119$  where  $P = (F_o^2 + 2F_c^2)/3$   
 $S = 1.04$   $(\Delta\sigma)_{max} = 0.001$   
 3636 reflections  $\Delta\rho_{max} = 0.46 \text{ e \AA}^{-3}$   
 245 parameters  $\Delta\rho_{min} = -0.25 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

|            |           |            |             |
|------------|-----------|------------|-------------|
| C1–P1      | 1.792 (3) | C20–O2     | 1.395 (4)   |
| C7–P1      | 1.782 (3) | C20–O1     | 1.408 (4)   |
| C13–P1     | 1.784 (3) | C21–C22    | 1.418 (5)   |
| C19–C20    | 1.515 (4) | C21–O1     | 1.427 (4)   |
| C19–P1     | 1.800 (3) | C22–O2     | 1.423 (4)   |
| C20–C19–P1 | 116.6 (2) | C20–O2–C22 | 105.6 (3)   |
| O2–C20–O1  | 105.7 (3) | C7–P1–C13  | 112.84 (13) |
| O2–C20–C19 | 110.0 (3) | C7–P1–C1   | 110.43 (14) |
| O1–C20–C19 | 112.4 (2) | C13–P1–C1  | 107.47 (13) |
| C22–C21–O1 | 106.6 (3) | C7–P1–C19  | 109.53 (14) |
| C21–C22–O2 | 106.7 (3) | C13–P1–C19 | 110.84 (14) |
| C20–O1–C21 | 105.0 (2) | C1–P1–C19  | 105.46 (14) |

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with  $C-H = 0.96 \text{ \AA}$  and  $U_{iso}(H) = 1.5U_{eq}(C)$ .

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve

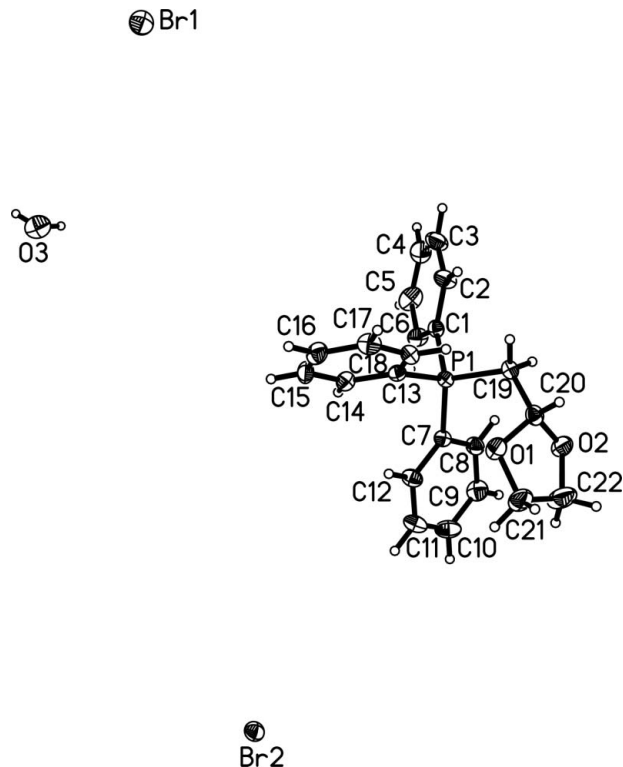


Figure 1

The asymmetric unit of compound (I), with the labelling of the non-H atoms. Displacement ellipsoids are drawn at the 30% probability level.

structure: SHELXTL (Bruker, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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