

Tetramethylphosphonium hydrogen carbonate

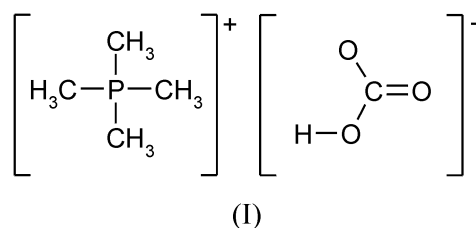
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Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{O}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.040
wR factor = 0.087
Data-to-parameter ratio = 25.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.In $\text{P}(\text{CH}_3)_4(\text{HCO}_3^-)$ the hydrogen carbonate ions form hydrogen-bonded head-to-tail dimers, as in the corresponding compounds of the heavier alkali metals.Received 1 July 2002
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Comment

The hydrogen carbonates of the alkali metals sodium (Sharma, 1965), potassium (Thomas *et al.*, 1974) and caesium (Kaduk, 1993) have been characterized by single-crystal structure determination. Whereas the hydrogen carbonate ions form hydrogen-bonded infinite chains in the sodium compound, the larger separation of the anions in the compounds of the heavier alkali metals leads to the formation of dimers.The tetramethylphosphonium ion can be considered to behave like a large alkali metal; in fact, the hydrogen carbonate ions do also form dimers in the title compound. (Fig. 1). The geometry of the tetramethylphosphonium cation is as expected (Margarf *et al.*, 2002). In the hydrogen carbonate moiety, the C5–O3 bond involving the hydrogen-bond acceptor is slightly longer than the C5–O2 bond that does not take part in the hydrogen bonding. The packing resembles that of the antiferrotype. The hydrogen carbonate dimers are surrounded by eight tetramethylphosphonium ions, with the P atoms forming a strongly distorted parallelepiped. The centers of half of these parallelepipeds are occupied by hydrogen carbonate dimers (Fig. 2).

Experimental

The title compound was obtained, by accident, by exposure of a solution of tetramethylphosphonium superoxide in liquid ammonia to air. Some of the mother liquor containing crystals was taken and immersed in cooled perfluorinated polyether oil. A suitable crystal was chosen under a microscope and picked up with a nylon loop attached to a prealigned goniometer head, which was transferred in liquid nitrogen to the diffractometer and mounted in a cooling stream.

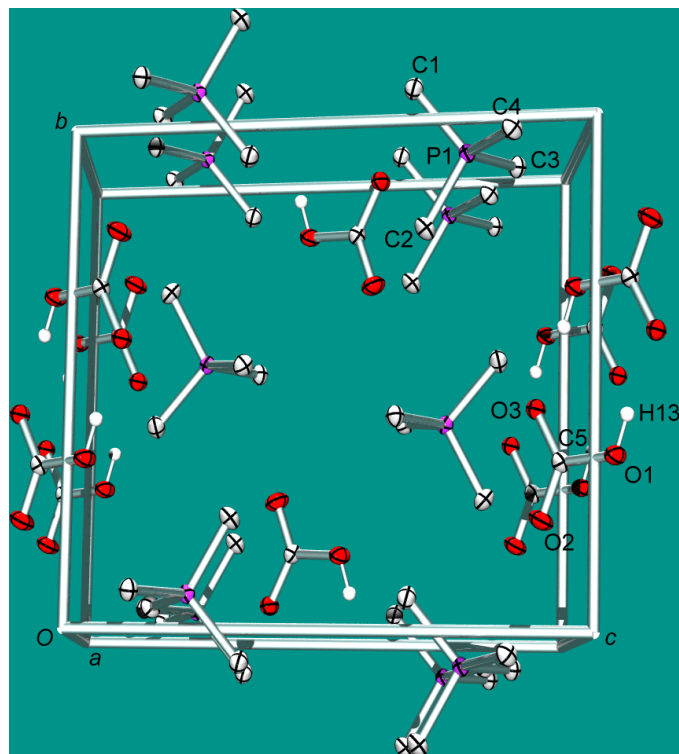


Figure 1
Packing diagram of the title compound, with the atom-numbering scheme shown. Displacement ellipsoids are drawn at the 50% probability level.

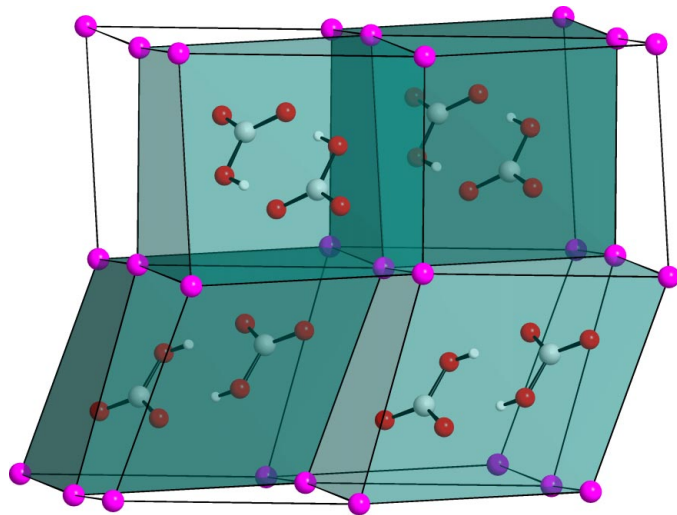


Figure 2
View of the polyhedra surrounding the hydrogen carbonate dimers. The C atoms of the phosphonium ions have been omitted for clarity.

Crystal data

$\text{C}_4\text{H}_{12}\text{P}^+\cdot\text{CHO}_3^-$
 $M_r = 152.12$
 Monoclinic, $P2_1/n$
 $a = 6.862(2) \text{ \AA}$
 $b = 10.293(3) \text{ \AA}$
 $c = 11.540(3) \text{ \AA}$
 $\beta = 104.296(6)^\circ$
 $V = 789.9(4) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.279 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 2752 reflections
 $\theta = 2.7\text{--}33.1^\circ$
 $\mu = 0.29 \text{ mm}^{-1}$
 $T = 100(1) \text{ K}$
 Plate, colorless
 $0.02 \times 0.01 \times 0.01 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer
 ω scans
 Absorption correction: none
 12209 measured reflections
 3349 independent reflections

2211 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.073$
 $\theta_{\text{max}} = 35.0^\circ$
 $h = -10 \rightarrow 11$
 $k = -16 \rightarrow 14$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.087$
 $S = 0.89$
 3349 reflections
 134 parameters
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0024P)^2 + 0.2026P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.68 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$

Table 1

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

P1—C1	1.7762 (13)	O1—C5	1.3623 (14)
P1—C4	1.7767 (12)	O2—C5	1.2287 (15)
P1—C3	1.7777 (12)	O3—C5	1.2630 (14)
P1—C2	1.7787 (14)		
C1—P1—C4	108.76 (6)	C3—P1—C2	108.47 (7)
C1—P1—C3	109.62 (7)	O2—C5—O3	126.63 (11)
C4—P1—C3	110.13 (6)	O2—C5—O1	116.27 (11)
C1—P1—C2	109.83 (7)	O3—C5—O1	117.11 (11)
C4—P1—C2	110.02 (7)		

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H13 \cdots O3 ⁱ	0.840 (19)	1.75 (2)	2.5817 (14)	173.3 (18)

Symmetry code: (i) $-x, 1 - y, 2 - z$.

Data collection: SMART32 (Bruker, 2000); cell refinement: SAINT32 (Bruker, 2000); data reduction: SAINT32; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg, 2000) and ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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