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Anion–anion one-dimensional chain formation within cationic channels in tetrabutylammonium dihydrogenphosphate dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.013 Å
R factor = 0.156
wR factor = 0.393
Data-to-parameter ratio = 15.7

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

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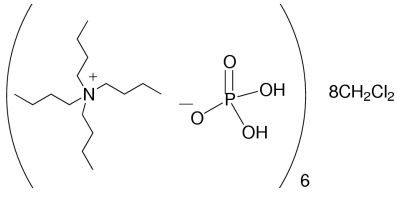
The title compound, $C_{16}H_{36}N^{+}.H_2PO_4^{-}.1.33CH_2Cl_2$, exists as infinite one-dimensional dihydrogen phosphate chains within cationic tetrabutylammonium channels.

Comment

The development of receptors and sensors for anions is a rapidly developing area in supramolecular chemistry (Atwood *et al.*, 1996; Beer & Gale, 2001; Beer & Smith, 1997; Bianchi *et al.*, 1997; Davis *et al.*, 1996; Kavallieratos *et al.*, 1997; Schmidtchen & Berger, 1997; Sessler & Allen, 1999). In addition, anions have recently been exploited as templates for the formation of self-assembled supramolecular architectures in both organic and inorganic systems (Gale, 2000, 2001). It is possible to imagine how dihydrogenphosphate would oligomerize to form a coordination polymer. Tetrabutylammonium anion salts are frequently used as sources of anions, but the crystal structure of tetrabutylammonium dihydrogenphosphate has not, to the best of our knowledge, previously been elucidated. Anion chain assembly has been observed in other crystal structures (*e.g.* Karle & Karle, 1988), including the hydrated tetramethylammonium dihydrogenphosphate (Ohama *et al.*, 1987) but not in the tetrabutylammonium salts that are commonly used in anion complexation studies.



(I)

The title compound, (I), crystallizes in a large triclinic cell with six tetrabutylammonium ions, six dihydrogenphosphate ions and eight dichloromethane molecules in the asymmetric unit. The dihydrogenphosphate ions donate and accept two hydrogen bonds to form an infinite chain and, although there are six independent ions in the asymmetric unit, the repeat length of the chain is 12 (Fig. 2). The tetrabutylammonium ions can be seen to form cationic channels within which reside the infinite dihydrogenphosphate chains (Fig. 3).

Experimental

The title compound was obtained from the Aldrich Chemical Company and recrystallized in a dry atmosphere *via* slow evaporation from dichloromethane.

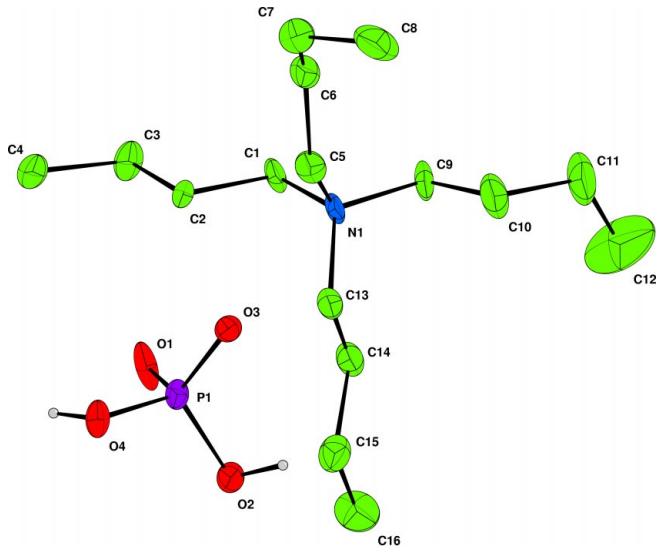


Figure 1
Displacement ellipsoid plot of one anion–cation pair. The ellipsoids are drawn at the 30% probablity level and the H atoms have been omitted for clarity. The remaining ions in the asymmetric unit are labelled in a similar fashion.

Crystal data

$C_{17.33}H_{40.67}Cl_{2.67}NO_4P$
M_r = 452.68
Triclinic, $P\overline{1}$
a = 16.1733 (3) Å
b = 17.8443 (3) Å
c = 26.7000 (5) Å
α = 77.520 (3)°
β = 77.237 (3)°
γ = 83.124 (3)°
V = 7316.7 (2) Å^3
Z = 12
D_x = 1.233 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 58198 reflections
θ = 2.9–24.1°
μ = 0.43 mm^{-1}
T = 120 (2) K
Block, colourless
0.50 × 0.15 × 0.10 mm

Data collection

Nonius Kappa CCD area-detector diffractometer
φ and ω scans to fill the Ewald sphere
Absorption correction: multi-scans (*SORTAV*; Blessing, 1997)
T_{min} = 0.816, T_{max} = 0.959
58198 measured reflections
22291 independent reflections
10322 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 24.1°
$h = -18 \rightarrow 18$
$k = -20 \rightarrow 20$
$l = -30 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.156
$wR(F^2)$ = 0.393
S = 1.93
22291 reflections
1417 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 2.834
$\Delta\rho_{max}$ = 2.66 e Å^{-3}
$\Delta\rho_{min}$ = −1.47 e Å^{-3}

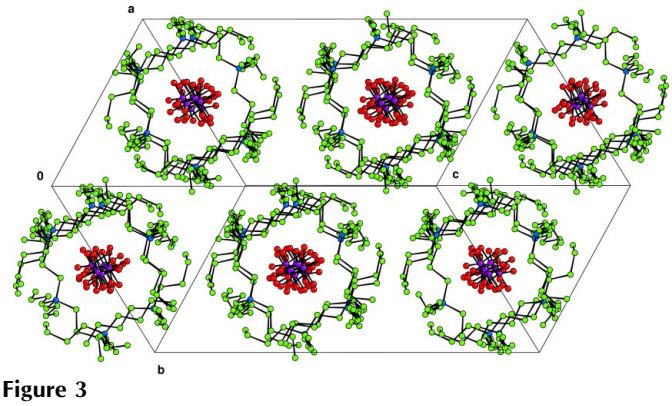


Figure 3
Packing diagram showing the sheath-like nature of the tetrabutylammonium ions around the dihydrogenphosphate chains.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H02···O5	0.84	1.70	2.505 (7)	160
O4—H04···O1^{i}	0.84	1.66	2.478 (7)	165
O7—H07···O24	0.84	1.71	2.521 (7)	163
O8—H08···O3	0.84	1.71	2.527 (6)	164
O9—H09···O13	0.84	1.73	2.553 (6)	167
O11—H011···O10^{ii}	0.84	1.70	2.518 (7)	165
O14—H014···O12	0.84	1.73	2.551 (6)	167
O16—H016···O18	0.84	1.77	2.529 (8)	150
O17—H017···O15	0.84	1.71	2.541 (6)	169
O20—H020···O21^{iii}	0.84	1.71	2.443 (9)	144
O22—H022···O19^{iv}	0.84	2.04	2.625 (11)	126
O23—H023···O6	0.84	1.70	2.448 (8)	148

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $2-x, 1-y, -z$; (iii) $x, y-1, z$; (iv) $x, 1+y, z$.

The crystals were extremely hygroscopic and it was found necessary to mount and place them on the diffractometer under a constant stream of cold nitrogen gas. The resulting data set was of low quality and it was necessary to restrain the geometries of the tetrabutylammonium ions to be similar. The structure contains a non-space-group translation.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *SCALEPACK* (Otwinoski & Minor, 1997), *COLLECT*; data reduction: *DENZO* (Otwinoski & Minor, 1997), *COLLECT*, *MAXUS* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS* 97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL* 97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1998).

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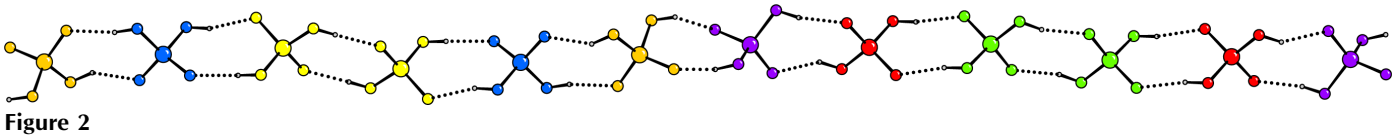

Figure 2
One repeating unit of the infinite hydrogen-bonded dihydrogenphosphate chain; P5 = orange, P4 = blue, P3 = yellow, P6 = purple, P2 = red, P1 = green.

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