Dihydrogen phosphate mediated supramolecular frameworks in 2- and 4-chloroanilinium dihydrogen phosphate salts

P. Balamurugan,^a R. Jagan^b and K. Sivakumar^{b*}

^aDepartment of Physics, Dhanalakshmi College of Engineering, Manimangalam, Chennai 601 301, India, and ^bDepartment of Physics, Anna University, Chennai 600 025, India

Correspondence e-mail: ksivakumar@annauniv.edu

Received 2 November 2009

Accepted 15 January 2010

Online 3 February 2010

The title compounds, 2-chloroanilinium dihydrogen phosphate (2CADHP) and 4-chloroanilinium dihydrogen phosphate (4CADHP), both $C_6H_7NCl^+ \cdot H_2PO_4^-$, form two-dimensional supramolecular organic–inorganic hybrid frameworks. In 2CADHP, the dihydrogen phosphate anions form a double-stranded anionic chain generated parallel to the [010] direction through $O-H \cdots O$ hydrogen bonds, whereas in 4CADHP they form a two-dimensional supramolecular net extending parallel to the crystallographic (001) plane into which the cations are linked through strong $N-H \cdots O$ hydrogen bonds.

Comment

The construction of organic–inorganic hybrid compounds has been of considerable interest and importance in recent years, not only because they are a powerful means of generating interesting supramolecular frameworks but also due to their potential for providing new materials with magnetic, semi-conducting, optical and electrolytic properties (Doyle *et al.*, 2002; Zaccaro & Ibanez, 2000; Chisholm & Haile, 2000). The supramolecular frameworks of these organic–inorganic compounds are generated by hydrogen-bond interactions between donor (*D*) and acceptor (*A*) moieties. Orthophosphoric acid (H_3PO_4), an inorganic oxy-acid, forms dihydrogen phosphate salts with organic amines, resulting in organic–inorganic hybrid systems with potentially powerful hydrogen-bonded *D/A* moieties. The dihydrogen phosphate anions ($H_2PO_4^-$) form substructures in these compounds, generating anionic networks *via* $O-H \cdots O$ hydrogen bonds which act as a template for the assembly of cations (Shylaja *et al.*, 2008). A considerable number of dihydrogen phosphate salts are recorded in the Cambridge Structural Database (CSD, Version 5.28; Allen, 2002). In the crystal structure of benzylammonium dihydrogen phosphate monohydrate (Elaoud *et*

al., 1998), the $H_2PO_4^-$ anions form a one-dimensional chain network, while in 3-amino-2-chloropyridinium dihydrogen phosphate (Hamed *et al.*, 2007) they form chains of fused $R_2^2(8)$ ring motifs [for graph-set analysis, see Bernstein *et al.* (1995)]. Two-dimensional nets of anionic substructures were also observed in dimethylammonium dihydrogen phosphate (Pietraszko *et al.*, 1999) and 2-methylpiperazinedium dihydrogen phosphate (Choudhury *et al.*, 2000). Interestingly, in the structure of imidazolinium dihydrogen phosphate (Blessing, 1986), the $H_2PO_4^-$ anions form a three-dimensional cage-type framework inside which the imidazolinium cations are trapped. We have prepared the dihydrogen phosphate salts 2-chloroanilinium dihydrogen phosphate (2CADHP) and 4-chloroanilinium dihydrogen phosphate (4CADHP), and have determined their structures and studied the supramolecular networks in these salts.

The salt 2CADHP crystallizes in the space group $P2_1/n$, whereas 4CADHP crystallizes in $Pbca$. The asymmetric units of both 2CADHP and 4CADHP contain a dihydrogen phosphate anion and a singly protonated 2- or 4-chloroanilinium cation, respectively. In the tetrahedral dihydrogen phosphate group of both 2CADHP and 4CADHP, the protonated P–O bond distances are $P1-O1 = 1.5696(11)$ Å and $P1-O2 = 1.5529(12)$ Å for 2CADHP, and $P1-O1 = 1.541(2)$ Å and $P1-O2 = 1.557(2)$ Å for 4CADHP. These values are as expected and are longer than the other two P–O bonds, *viz.* $P1-O3 = 1.5036(12)$ Å and $P1-O4 = 1.5031(12)$ Å for 2CADHP, and $P1-O3 = 1.5168(17)$ Å and $P1-O4 = 1.4957(19)$ Å for 4CADHP. The identical $P1-O3$ and $P1-O4$ bond distances observed in 2CADHP indicate delocalization of negative charge between them (Demir *et al.*, 2006). The geometries of the 2- and 4-chloroanilinium cations

Figure 1

The independent components of 2CADHP, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

Figure 2

The independent components of 4CADHP, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

show characteristic values compared with other reported structures (Muthamizhchelvan *et al.*, 2005; Glidewell *et al.*, 2005). The C–N distances of the 2- and 4-chloroanilinium cations [C1–N1 = 1.4545 (18) and 1.467 (3) Å, respectively] are longer than the neutral C–NH₂ value [1.386 (4) Å; Ploug-Sørensen & Andersen, 1985] and this lengthening is due to the transfer of an H atom to the N atom from the orthophosphoric acid.

The hydrogen-bonded organic–inorganic supramolecular frameworks of 2CADHP and 4CADHP are determined primarily by a combination of O–H...O and N–H...O hydrogen bonds (Tables 1 and 2).

In 2CADHP, the inversion-related H₂PO₄[−] anions are linked through an O1–H1D...O3ⁱⁱⁱ hydrogen bond [symmetry code: (iii) $-x + 1, -y + 1, -z + 1$], forming an O–H...O hydrogen-bonded dimer with a ring motif of $R_2^2(8)$, with its centroid occupying the inversion centre. These dimers are interlinked through an O2–H2D...O3ⁱⁱ hydrogen bond [symmetry code: (ii) $x, y + 1, z$] to form a ring motif of type $R_4^2(12)$. The alternately fused $R_2^2(8)$ and $R_4^2(12)$ supramolecular motifs in turn generate a double-stranded inorganic H₂PO₄[−] chain made of P–OH...O=P hydrogen bonds extending infinitely along the [010] direction (Fig. 3). The 2-chloroanilinium cations are linked to the anionic substructure through three N–H...O hydrogen bonds and a Cl...O short contact [Cl...O = 3.1705 (14) Å]. The N1–H1A...O4 and N1–H1C...O4ⁱⁱ hydrogen bonds [symmetry code: (ii) $x, y + 1, z$], along with O2–H2D...O3ⁱⁱ, form a chain of edge-fused $R_3^3(10)$ ring motifs extending along the [010] direction, as observed in the structure of 3-acetylanilinium dihydrogen phosphate (Cinčić & Kaitner, 2008). The Cl...O1 interaction, which acts as a pseudo-hydrogen bond (Bryant *et al.*, 1998; Kubicki & Wagner, 2007), with the Cl1 atom at (x, y, z) as donor and atom O1 at $(-x + \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{2})$ as acceptor, along with the N–H...O hydrogen bonds, forms a chain of fused $R_4^2(10)$ motifs extending along the [010] direction. The 2₁ screw-related chains of $R_3^3(10)$ and $R_4^2(10)$ motifs along $(\frac{1}{4}, y, \frac{1}{4})$ (Fig. 4) link the anionic substructure, resulting in the formation of an organic–inorganic sheet framework parallel to (10 $\bar{1}$) (Fig. 5).

In 4CADHP, the H₂PO₄[−] anions form dimers through an O2–H2D...O3^{iv} hydrogen bond [symmetry code: (iv) $-x + 1,$

Figure 3

Part of the crystal structure of 2CADHP, showing the formation of the double-stranded H₂PO₄[−] anionic chain built from $R_2^2(8)$ and $R_4^2(12)$ rings through O2–H2D...O3ⁱⁱ and O1–H1D...O3ⁱⁱⁱ hydrogen bonds extending infinitely along the [010] direction. [Symmetry codes: (ii) $x, y + 1, z$; (iii) $-x + 1, -y + 1, -z + 1$.]

Figure 4

Part of the crystal structure of 2CADHP, showing the 2₁ screw-related chains of fused $R_3^3(10)$ and $R_4^2(10)$ rings extending along the [010] direction and linked to the H₂PO₄[−] anions through N1–H1A...O4, N1–H1C...O4ⁱⁱ, N1–H1B...O1ⁱ and O2–H2D...O3ⁱⁱ hydrogen bonds. H atoms attached to C atoms have been omitted for clarity. [Symmetry codes: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, y + 1, z$.]

$-y, -z + 1$], with the characteristic ring motif of $R_2^2(8)$, in which the centroid of the dimer occupies the crystallographic inversion centre. The O1–H1D...O4ⁱⁱⁱ hydrogen bond [symmetry code: (iii) $-x + \frac{3}{2}, y - \frac{1}{2}, z$] generates a C₄ chain which connects the glide-related anionic dimers with the glide plane perpendicular to the [100] direction, the glide component of which is $[0, \frac{1}{2}, 0]$. This forms an infinite two-dimensional layer in the form of a net extending parallel to the (001) plane. This inorganic supramolecular net of H₂PO₄[−] anions is built from $R_2^2(8)$ and $R_6^6(24)$ ring motifs (Fig. 6). The 4-chloroanilinium cations are anchored to the H₂PO₄[−] anionic net

Figure 5

Part of the crystal structure of 2CADHP, showing the formation of the organic-inorganic supramolecular (101) sheet built from $R_2^2(8)$, $R_2^2(12)$, $R_3^3(10)$ and $R_4^4(10)$ rings via N—H...O and O—H...O hydrogen bonds and a Cl...O interaction extending along the [010] and [101] directions, respectively. H atoms attached to C atoms have been omitted for clarity.

Figure 6

Part of the crystal structure of 4CADHP, showing the formation of the two-dimensional (001) net of H_2PO_4^- anions built from $R_2^2(8)$ and $R_6^6(24)$ rings through O2—H2D...O3^{iv} and O1—H1D...O4ⁱⁱⁱ hydrogen bonds. [Symmetry codes: (iii) $-x + \frac{3}{2}, y - \frac{1}{2}, z$; (iv) $-x + 1, -y, -z + 1$.]

through N1—H1A...O4ⁱ [symmetry code: (i) $-x + 1, -y + 1, -z + 1$], N1—H1B...O3 and N1—H1C...O3ⁱⁱ [symmetry code: (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, z$] hydrogen bonds, forming fused-ring motifs of $R_5^5(14)$ and $R_5^5(12)$ types with O—H...O hydrogen bonds (Fig. 7). The 4-chloroanilinium cations are pendant on both faces of the anionic net, thus resulting in the formation of two-dimensional sheet of an organic-inorganic supramolecular framework (Fig. 8) extending infinitely parallel to the crystallographic (001) plane.

It is of interest to note that in both the title compounds, although they form different types of anionic substructures, the overall anionic-cationic supramolecular framework results in the formation of infinite two-dimensional sheets. In 2CADHP, the formation of an anionic double-stranded substructure and the linking of the cations to it is analogous with other reported structures. In the crystal structures of 2,4-dimethylanilinium dihydrogen phosphate (Fábry *et al.*, 2001), 2-(methoxycarbonyl)anilinium dihydrogen phosphate (Shafiq *et al.*, 2009) and 3,5-dimethoxyanilinium dihydrogen phosphate (Kaabi *et al.*, 2004) ($Z' = 2Z$), the respective cations bound to the anionic substructures form two-dimensional

Figure 7

Part of the crystal structure of 4CADHP, showing the cations linked to the anionic substructure forming $R_5^5(14)$ and $R_5^5(12)$ fused ring motifs through N1—H1A...O4ⁱ, N1—H1B...O3, N1—H1C...O3ⁱⁱ, O1—H1D...O4ⁱⁱⁱ and O2—H2D...O3^{iv} hydrogen bonds. H atoms bonded to C atoms, and the unit-cell outline, have been omitted for clarity. [Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, z$; (iii) $-x + \frac{3}{2}, y - \frac{1}{2}, z$; (iv) $-x + 1, -y, -z + 1$.]

Figure 8

A projection, down [001], of part of the crystal structure of 4CADHP, showing the (001) sheet of the organic-inorganic supramolecular framework built from $R_2^2(8)$, $R_6^6(24)$, $R_5^5(14)$ and $R_5^5(12)$ ring motifs extending parallel to the [100] and [010] directions. H atoms bonded to C atoms have been omitted for clarity.

sheets. In the last compound, the substructure was formed with different ring motifs than the other two structures. A three-dimensional hydrogen-bonded framework was observed for 1,3-propanediammonium bis(dihydrogen phosphate) (Marsh, 2004), in which the cation contains an additional three N—H bonds involved in hydrogen bonding. The crystal structure of 4CADHP is isomorphous with 4-bromoanilinium dihydrogen phosphate (CSD refcode UGISEI; Zhang *et al.*, 2001), but no H atoms are reported in CSD. In 4CADHP, the hydrogen-bonded anionic substructure formation and the linking of cations pendant from the supramolecular net are analogous to the structures of 4-methylanilinium dihydrogen phosphate (Smirani *et al.*, 2004) and 4-ethylanilinium dihy-

drogen phosphate (Kaabi *et al.*, 2003), but it has markedly different cell dimensions from 4-bromoanilinium dihydrogen phosphate, even though they belong to the same *Pbca* space group.

Experimental

Ethanol solutions containing equimolar quantities of 2-chloroaniline and orthophosphoric acid were mixed to produce a white precipitate, which was filtered off, dried for a few hours, dissolved in ethanol and allowed to recrystallize to afford colourless single crystals of 2CADHP after a period of about two weeks.

Colourless crystals of 4CADHP were obtained from a solution of 4-chloroaniline and orthophosphoric acid mixed at a 1:1 stoichiometric ratio in a mixed solvent of ethanol and water in equal proportions (50:50 v/v) upon gentle heating. The solution thus prepared was allowed to crystallize and crystals were obtained by slow evaporation of the solvent.

2CADHP

Crystal data

$\text{C}_6\text{H}_7\text{ClN}^+\cdot\text{H}_2\text{PO}_4^-$	$V = 891.17 (8) \text{ \AA}^3$
$M_r = 225.56$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 11.3143 (6) \text{ \AA}$	$\mu = 0.59 \text{ mm}^{-1}$
$b = 4.7466 (2) \text{ \AA}$	$T = 292 \text{ K}$
$c = 17.5024 (9) \text{ \AA}$	$0.25 \times 0.20 \times 0.15 \text{ mm}$
$\beta = 108.540 (3)^\circ$	

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer	9738 measured reflections
Absorption correction: multi-scan (SADABS; Bruker 1999)	2044 independent reflections
$T_{\min} = 0.867$, $T_{\max} = 0.917$	1902 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$	122 parameters
$wR(F^2) = 0.084$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
2044 reflections	$\Delta\rho_{\text{min}} = -0.47 \text{ e \AA}^{-3}$

4CADHP

Crystal data

$\text{C}_6\text{H}_7\text{ClN}^+\cdot\text{H}_2\text{PO}_4^-$	$V = 1927.9 (7) \text{ \AA}^3$
$M_r = 225.56$	$Z = 8$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 9.7371 (19) \text{ \AA}$	$\mu = 0.55 \text{ mm}^{-1}$
$b = 7.8756 (16) \text{ \AA}$	$T = 292 \text{ K}$
$c = 25.141 (5) \text{ \AA}$	$0.25 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer	8591 measured reflections
Absorption correction: multi-scan (SADABS; Bruker 1999)	1687 independent reflections
$T_{\min} = 0.876$, $T_{\max} = 0.899$	1483 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	123 parameters
$wR(F^2) = 0.127$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.50 \text{ e \AA}^{-3}$
1687 reflections	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$) for 2CADHP.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O4}$	0.89	1.95	2.8348 (19)	170
$\text{N1}-\text{H1B}\cdots\text{O1}^{\text{i}}$	0.89	2.17	3.0509 (18)	169
$\text{N1}-\text{H1C}\cdots\text{O4}^{\text{ii}}$	0.89	1.87	2.7515 (18)	172
$\text{O1}-\text{H1D}\cdots\text{O3}^{\text{iii}}$	0.82	1.78	2.6017 (17)	174
$\text{O2}-\text{H2D}\cdots\text{O3}^{\text{ii}}$	0.82	1.77	2.5411 (17)	157

Symmetry codes: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, y + 1, z$; (iii) $-x + 1, -y + 1, -z + 1$.

Table 2

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$) for 4CADHP.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O4}^{\text{i}}$	0.89	1.80	2.689 (3)	176
$\text{N1}-\text{H1B}\cdots\text{O3}$	0.89	1.97	2.862 (3)	175
$\text{N1}-\text{H1C}\cdots\text{O3}^{\text{ii}}$	0.89	2.09	2.951 (3)	162
$\text{O1}-\text{H1D}\cdots\text{O4}^{\text{iii}}$	0.82	1.79	2.540 (3)	152
$\text{O2}-\text{H2D}\cdots\text{O3}^{\text{iv}}$	0.82	1.82	2.585 (3)	155

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x + \frac{3}{2}, y + \frac{1}{2}, z$; (iii) $-x + \frac{3}{2}, y - \frac{1}{2}, z$; (iv) $-x + 1, -y, -z + 1$.

The positions of the H atoms bound to N and O atoms were identified from difference electron-density maps, but were subsequently geometrically optimized ($\text{O}-\text{H} = 0.82 \text{ \AA}$ and $\text{N}-\text{H} = 0.89 \text{ \AA}$) and allowed to ride at the best staggered positions, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O}, \text{N})$, except for O2 of 2CADHP whose $\text{O}-\text{H}$ vector was allowed to rotate around the $\text{P}-\text{O}$ bond. H atoms bound to C atoms were treated as riding atoms, with $\text{C}-\text{H} = 0.93 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

For both compounds, data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREF* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009).

The authors thank Dr Babu Varghese, Senior Scientific Officer, and SAIF at IITM for providing the data-collection facility.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3320). Services for accessing these data are described at the back of the journal.

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