References

Allen, F. H., Bellard, S., Brice, M. D., Cartwright, B. A., Doubleday, A., Higgs, H., Hummelink, T., Hummelink-Peters, B. G., Kennard, O., Motherwell, W. D. S., Rodgers, J. R. & Watson, D. G. (1979). *Acta Cryst.* B**35**, 2331-2339.

Atkin, C. L., Thelander, L., Reichard, P. & Lang, G. (1973). *J. Biol. Chem.* **248**, 7464-7472.

B. A. Frenz & Associates, Inc. (1982). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.

Due, L., Rasmussen, H. & Larsen, I. K. (1987). *Acta Cryst.* C**43**, 582-585.

Elford, H. L., van't Riet, B., Wampler, G. L., Lin, A. L. & Elford, R. M. (1981). *Adv. Enzyme Regul.* **19**, 151-168.

Elford, H. L., Wampler, G. L. & van't Riet, B. (1979). *Cancer Res.* **39**, 844-851.

Gräslund, A., Ehrenberg, A. & Thelander, L. (1982). *J. Biol. Chem.* **257**, 5711-5715.

Howell, M. L., Sanders-Loehr, J., Loehr, T. M., Roseman, N. A., Mathews, M. K. & Slabaugh, M. B. (1992). *J. Biol. Chem.* **267**, 1705-1711.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kjøller Larsen, I., Sjöberg, B.-M. & Thelander, L. (1982). *Eur. J. Biochem.* **125**, 75-81.

Larsen, I. K. (1978). *Acta Cryst.* B**34**, 962-964.

Larsen, I. K. (1980). *Acta Chem. Scand. Ser. B*, **34**, 209-212.

Larsen, I. K. (1988). *Acta Cryst.* B**44**, 527-533.

Nordlund, P., Sjöberg, B.-M. & Eklund, H. (1990). *Nature* (*London*), **345**, 593-598.

Riet, B. van't, Wampler, G. L. & Elford, H. L. (1979). *J. Med. Chem.* **22**, 589-592.

Rubens, R. D., Kaye, S. B., Soukop, M., Williams, C. J., Brampton, M. H. & Harris, A. L. (1991). *Br. J. Cancer*, **64**, 1187-1188.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467-473.

Thelander, M., Gräslund, A. & Thelander, L. (1985). *J. Biol. Chem.* **260**, 2737-2741.

Veale, D., Carmichael, J., Cantwell, B. M. J, Elford, H. L., Blackie, R., Kerr, D. J., Kaye, S. B. & Harris, A. L. (1988). *Br. J. Cancer*, **58**, 70-72.

Young, C. W., Schochetman, G., Hodas, S. & Balis, E. M. (1967). *Cancer Res.* **27**, 535-540.

Acta Cryst. (1993). C**49**, 813–815

Isopropylammonium Dihydrogenmonophosphate and Isopropylammonium Monohydrogenphosphite

Marie-Thérèse Averbuch-Pouchot

Laboratoire de Cristallographie associé à l'Université Joseph Fourier, CNRS, BP 166, 38042 Grenoble CEDEX, France

(*Received* 19 *June* 1992; *accepted* 13 *October* 1992)

Abstract

The atomic arrangements in isopropylammonium dihydrogenmonophosphate and isopropylammonium monohydrogenphosphite are described. In the phosphate, the distances between the H_2PO_4 groups are rather short (P—P = 4.238 and 4.218 Å) so that these entities form (through strong hydrogen bonds) infinite $(H_2PO_4)_n$ chains parallel to the **a** direction. In spite of the significantly longer (4.769 Å) P—P distance, the $(HPO_3H)_n$ groups in the phosphite compound also form infinite chains running parallel to the **a** direction. In the phosphate derivative, linear arrays of the isopropylammonium groups spread parallel to the **c** direction, *i.e.* perpendicular to the phosphoric chain, while in the phosphite compound the arrays of the organic component and the phosphoric chains are parallel.

Comment

Both arrangements are characterized principally by the existence of infinite phosphoric chains, $(H_2PO_4)_n$ for the phosphate and $(HPO_3H)_n$ in the case of the phosphite. In both structures, isopropylammonium groups are arranged in linear arrays, but the organization of these arrays relative to the phosphoric chains is fundamentally different in the two derivatives. In the phosphate, the phosphoric chains spread along the **a** direction, while the organic component arrays are parallel to the **c** direction. Fig. 1 shows the general organization of this arrangement projected along **c**. In contrast, both the $(HPO_3H)_n$ chains and the arrays of isopropylammonium groups in the phosphite compound are parallel to the **a** direction. In the phosphate, each H_2PO_4 group is connected to its two adjacent neighbours by relatively strong hydrogen bonds (2.567 and 2.631 Å) corresponding to O—O distances slightly longer than those observed inside the PO_4 tetrahedron. The short P—P distances observed in the chain (4.238 and 4.218 Å) are easily explained by the geometry of these hydrogen bonds. In the phosphite, the P—P distance is significantly longer (4.769 Å) because the infinite $(HPO_3H)_n$ chain is more stretched, each PO_3H tetrahedron being bonded to its two neighbours by only one hydrogen bond. Fig. 2 depicts the general organization of the phosphite derivative. Tables 2 and 4 give the main interatomic distances and bond angles in these two arrangements, includ-

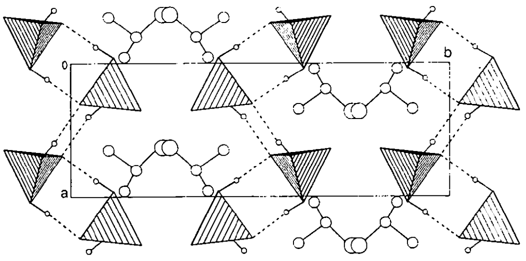

Fig. 1. Projection along the **c** direction of the atomic arrangement of the phosphate compound. The hatched tetrahedra denote the PO_4 groups. The open circles represent, in decreasing order of size, N, C and H atoms. Inside the phosphoric chain, hydrogen bonds are represented by solid and dashed lines. The H atoms of the organic components have been omitted.

0108-2701/93/040813-03$06.00

ing the hydrogen-bond schemes. In both structures, the organic entities are connected by strong N—H···O hydrogen bonds to the phosphoric anions. The organic entities (whose geometrical features are also reported in Tables 2 and 4) are quite regular and can be compared to those previously observed in a similar derivative, isopropylammonium *cyclo*-triphosphate, $[NH_3(C_3H_7)]_3P_3O_9$ (Averbuch-Pouchot, Durif & Guitel, 1988).

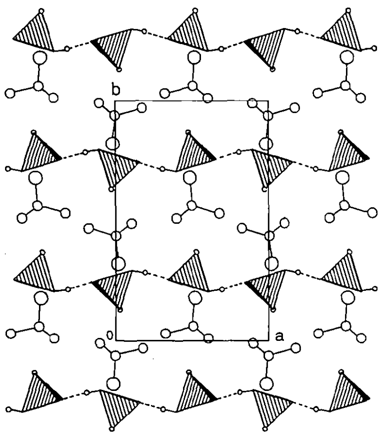

Fig. 2. Projection along the **c** direction of isopropylammonium phosphite. In the polyhedral representation of the PO_3H tetrahedron, the corner corresponding to the H atom is denoted by a small open circle. The H atoms of the organic components have been omitted.

Experimental

Phosphate derivative

Crystal data

$C_3H_{10}N^+.H_2O_4P^-$
$M_r = 157.11$
Monoclinic
$P2_1/c$
$a = 5.769$ (2) Å
$b = 15.39$ (1) Å
$c = 8.504$ (3) Å
$\beta = 109.43$ (3)°
$V = 712$ (1) Å^3
$Z = 4$
$D_x = 1.465$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 21 reflections
$\theta = 11.0$–12.0°
$\mu = 0.344$ mm^{-1}
$T = 295$ K
Stout prism
0.28 × 0.24 × 0.21 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
θ scans
2252 measured reflections
2055 independent reflections
2252 observed reflections
$R_{int} = 0.02$
$\theta_{max} = 30$°

$h = 0 \rightarrow 7$
$k = 0 \rightarrow 21$
$l = -11 \rightarrow 11$
2 standard reflections
frequency: 120 min
intensity variation: none

Refinement

Refinement on F
Final $R = 0.025$
$wR = 0.027$
$S = 0.390$
1487 reflections
[$I > 3.0\sigma(I)$]
131 parameters
All H-atom parameters refined
$w = 1$

$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.190$ e Å^{-3}
$\Delta\rho_{min} = 0.177$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for the phosphate derivative*

$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}/B_{iso}
P	0.75530 (6)	0.89967 (2)	0.00831 (4)	1.861 (4)
O1	0.3086 (2)	0.02335 (6)	0.1075 (1)	2.25 (2)
O2	0.0412 (2)	0.61327 (6)	0.5741 (1)	2.62 (2)
O3	0.3591 (2)	0.31539 (7)	0.5624 (1)	2.68 (2)
O4	0.3072 (2)	0.07969 (7)	0.8300 (1)	3.02 (2)
C1	0.1967 (3)	0.6709 (1)	0.0259 (2)	2.69 (3)
C2	0.3465 (3)	0.9003 (1)	0.4862 (2)	3.60 (4)
C3	0.0440 (3)	0.8595 (1)	0.6279 (3)	4.41 (4)
N	0.3663 (2)	0.74068 (8)	0.1197 (2)	2.43 (2)
H1	0.119 (4)	0.934 (1)	0.083 (3)	5.9 (6)
H2	0.597 (3)	0.954 (1)	0.156 (2)	4.1 (4)

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *for the phosphate derivative*

P	O1	O2	O3	O4
O1	1.506 (2)	2.510 (2)	2.543 (2)	2.512 (2)
O2	109.4(1)	1.568 (2)	2.470 (2)	2.462 (3)
O3	116.2 (1)	107.8(1)	1.488 (2)	2.492 (3)
O4	109.7 (1)	103.6 (1)	109.3(1)	1.566(2)

P—O2—H1	112 (2)	P—O4—H2	112 (3)
C1—N	1.492 (3)	N—C1—C2	108.8 (2)
C1—C2	1.503 (4)	N—C1—C3	108.8 (3)
C1—C3	1.501 (4)	C2—C1—C3	113.2 (3)

O(N)—H···O	O(N)—H	H···O	O(N)···O	O(N)—H···O
O2—H1···O1	0.85 (4)	1.73 (4)	2.567 (2)	173 (4)
O4—H2···O1	0.73 (3)	1.91 (3)	2.631 (2)	170 (4)
N—H1N···O3	0.93 (3)	1.87 (3)	2.804 (3)	174 (3)
N—H2N···O3	0.92 (3)	2.00 (3)	2.870 (3)	157 (3)
N—H3N···O3	0.92 (3)	1.86 (3)	2.778 (3)	175 (3)

Phosphite derivative

Crystal data

$C_3H_{10}N^+.H_2O_3P^-$
$M_r = 141.107$
Orthorhombic
$P2_12_12_1$
$a = 8.125$ (9) Å
$b = 12.677$ (10) Å
$c = 7.172$ (9) Å
$V = 739$ (2) Å^3
$Z = 4$
$D_x = 1.269$ Mg m^{-3}

Ag $K\alpha$ radiation
$\lambda = 0.5608$ Å
Cell parameters from 25 reflections
$\theta = 8.50$–13.80°
$\mu = 0.167$ mm^{-1}
$T = 293$ K
Short prism
0.32 × 0.32 × 0.24 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
θ scans
3817 measured reflections

$\theta_{max} = 30$°
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 11$

1073 independent reflections
1627 observed reflections
[$I > 3.0\sigma(I)$]
$R_{\rm int}$ = 0.01

2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
Final R = 0.043
wR = 0.045
S = 0.536
1073 reflections
121 parameters
All H-atom parameters refined
w = 1

$(\Delta/\sigma)_{\rm max}$ = 0.04
$\Delta\rho_{\rm max}$ = 0.498 e Å^{-3}
$\Delta\rho_{\rm min}$ = 0.359 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 3. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for the phosphite derivative*

$B_{\rm eq} = (1/3)\Sigma_i\Sigma_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$B_{\rm eq}/B_{\rm iso}$
P	0.44767 (9)	0.27094 (8)	0.1702 (1)	2.97 (1)
O1	0.3434 (3)	0.2567 (2)	0.3392 (3)	3.97 (5)
O2	0.3703 (3)	0.2499 (3)	−0.0143 (3)	4.19 (6)
O3	0.3925 (3)	0.7044 (3)	0.3005 (4)	4.57 (6)
C1	0.0039 (5)	0.4381 (3)	0.3689 (6)	3.81 (8)
C2	0.1759 (5)	0.9690 (4)	0.1386 (7)	5.0 (1)
C3	0.1038 (7)	0.4893 (4)	0.2139 (9)	6.6 (1)
N	0.0163 (3)	0.3208 (2)	0.3515 (4)	2.94 (5)
HO3	0.307 (6)	0.715 (4)	0.391 (6)	5 (1)

Table 4. *Main interatomic distances* (Å) *and bond angles* (°) *for the phosphite derivative*

P	O1	O2	O3	H
O1	1.490 (2)	2.546 (3)	2.459 (3)	2.35 (5)
O2	117.5(1)	1.489 (3)	2.529 (3)	2.42 (5)
O3	107.3 (1)	111.9(2)	1.563 (3)	2.19 (5)
H	109 (2)	114 (2)	95(2)	1.40(5)

P—O3—HO3	116 (3)		
C1—N	1.495 (5)	N—C1—C2	108.6 (3)
C1—C2	1.514 (6)	N—C1—C3	109.1 (3)
C1—C3	1.522 (7)	C2—C1—C3	112.2 (4)

O(N)—H···O	O(N)—H	H···O	O(N)···O	O(N)—H···O
O3—HO3···O2	0.96 (5)	1.65 (5)	2.581 (3)	161 (4)
N—H1N···O2	0.87 (4)	1.97 (4)	2.839 (4)	175 (4)
N—H2N···O1	0.87 (4)	1.93 (4)	2.781 (3)	163 (4)
N—H3N···O1	0.82 (4)	2.02 (4)	2.803 (3)	158 (4)

The monophosphate was prepared by slow evaporation at room temperature of a diluted aqueous solution containing H_3PO_4 and isopropylamine with the required stoichiometry. Several dissolutions followed by carefully controlled recrystallizations are often necessary to obtain good quality crystals. A similar process was used for the synthesis of the phosphite derivative.

Data collection: Nonius CAD-4 software. Cell refinement: Nonius CAD-4 software. Data reduction: *SDP* (Enraf-Nonius, 1977). Program used to solve structures: *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977). Program used to refine structure: *SDP*. Molecular graphics: *STRUPLO* (Fischer, 1985). Software used to prepare material for publication: *SDP*.

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 55751 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1023]

References

Averbuch-Pouchot, M. T., Durif, A. & Guitel, J. C. (1988). *Acta Cryst.* C**44**, 1907-1909.
Enraf-Nonius (1977). *Structure Determination Package*, version RSX11M. Enraf-Nonius, Delft, The Netherlands.
Fischer, R. X. (1985). *J. Appl. Cryst.* **18**, 258-262.
Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs of York, England, and Louvain, Belgium.

Acta Cryst. (1993). C**49**, 815–818

Structures of Glycinium Phosphite and Glycylglycinium Phosphite

MARIE-THÉRÈSE AVERBUCH-POUCHOT

Laboratoire de Cristallographie, Associé à l'Université J. Fourier, CNRS, BP 166, 38042 Grenoble CEDEX 09, France

(*Received* 20 *May* 1992; *accepted* 9 *October* 1992)

Abstract

The structure of glycinium phosphite consists of a layer arrangement of planes containing infinite $[\mathrm{H(HPO_3)}]_n^{n-}$ chains alternating with planes built by the organic anions, both layers developing parallel to the *bc* plane. Strong hydrogen bonds corresponding to O—O distances of 2.482 and 2.518 Å are responsible for the cohesion between the HPO_3 tetrahedra along this 'macroanion'. The same type of infinite $[\mathrm{H(HPO_3)}]_n^{n-}$ chain is observed in the glycylglycinium derivative which differs from the first compound in that the organic entities are connected by hydrogen bonds. A comparison between these two atomic arrangements and the corresponding monophosphates is given.

Comment

The atomic arrangement in glycine phosphite has a typical layer organization. As shown in Fig. 1, planes built by the phosphoric entities alternate with planes containing the organic groups, the two kinds of layer being parallel to the *bc* plane and separated by a

0108-2701/93/040815-04$06.00