The Crystal Structures of Ammonium Phosphinate (Hypophosphite) and Guanidinium Phosphinate, and the Cell Parameters of Hydrazinium(2+) Phosphinate

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(Received 15th December 1978)

Summary: In $(NH_4)H_2PO_2$ [Cmma, a = 7.515(6), b = 11.50(1), c = 3.988(4) Å, Z = 4] the anion has accurately C_{2v} symmetry, with P-O 1.500 Å and a O-P-O angle of 115.1°. In $(CH_6N_3)H_2PO_2$ [Pnam, a = 9.561(8), b = 7.401(5), c = 8.541(5) Å, Z = 4] the anion occupies a site of symmetry *m*, with similar dimension. In both compounds a hydrogen-bond network extends throughout the crystal (N..O 2.84 to 3.02 Å) and involves all H atoms of the cation. Structures were solved from, respectively, diffractometer data (R 0.074 for 210 reflections) and photographic data (R 0.074 for 488 reflections). The cell parameters of $(N_2H_6)(H_2PO_2)_2$ are: $P2_1/c$, a = 7.74(1), b = 6.44(1), c = 6.77(1) Å, β = 98.1(1)°.

We have recently studied[1] the crystal structures of several phosphinates (hypophosphites) to determine the structural role of the $H_2PO_2^-$ anion. Here we report improved parameters for $(NH_4)H_2PO_2$ [AP], originally studied[2] in 1934, and also the structure of (CH_6N_3)-H_2PO_2 [GP] and the unit cell parameters of (N_2H_6)-$(H_2PO_2)_2$ [HP].

Experimental

Aqueous H_3PO_2 (20%) was neutralized with aqueous ammonia or hydrazine hydrate, or with solid $(CH_6N_3)_2CO_3$. Satisfactory crystals of AP resulted from slow evaporation, and of HP from slow cooling, of the resultant solution; GP was precipitated by the addition of acetone, and was recrystallized from hot acetone: ethanol (4:1).

Crystal Data – AP, $(NH_4)H_2PO_2$, orthorhombic, *Cmma*, *a* = 7.515(6), *b* = 11.50(1), *c* = 3.988(4) Å, Z = 4, D_c = 1.60, D_m = 1.62 g cm^{-3}, F(000) = 176; MoKα radiation (λ = 0.7107 Å), μ = 5.1 cm^{-1}.

GP, $(CH_6N_3)H_2PO_2$, orthorhombic, *Pnam*, *a* = 9.561(8), *b* = 7.401(5), *c* = 8.541(5) Å, D_c = 1.37 g cm^{-3}, Z = 4, F(000) = 264; CuKα radiation (λ = 1.5418 Å), μ = 32.2 cm^{-1}.

HP, $(N_2H_6)(H_2PO_2)_2$, monoclinic, $P2_1/c$, *a* = 7.74(1), *b* = 6.44(1), *c* = 6.77(1) Å, β = 98.1(1)°.

Approximate cell parameters for all three compounds were obtained from NaCl-calibrated Weissenberg photographs. Crystals of HP were clearly sensitive to copper radiation, so this compound was not studied further. Intensity data and improved cell parameters for AP were measured by use of a Hilger linear diffractometer; for GP, they were measured from multi-film equi-inclination Weissenberg photographs by the Science Research Council Microdensitometer Service, Daresbury Laboratory, England. The reciprocal lattice layers scanned were: AP, O-*4kl* and *hk*O-3; GP, *h*O-8 and *hk*O-7. The data for GP was corrected for absorption. The structures were readily solved by the heavy-atom method. All hydrogen atoms in GP, and the anion hydrogens in AP, were located by weighted difference syntheses, and were included without refinement in the final cycles of full-matrix least-squares refinement. The NH_4^+ hydrogens of AP could not be located clearly, so were included at calculated positions (d_{N-H} 1.03 Å). Refinement converged at R 0.074 for both compounds, with anisotropic thermal parameters for all non-hydrogen atoms and unit weights for all reflections (AP, 210 independent reflections above background, 16 parameters; GP, 488 reflections, 38 parameters). Copies of tables of observed and calculated structure factors are available from the author. The SHELX-76 program system[3] was used in all calculations.

Discussion

Positional and thermal parameters for AP and GP are given in Tables 1 and 2, and derived dimensions in Table 3. Standard deviations are not given where hydrogen atoms are involved.

The positions of the N, O, and P atoms in AP were originally deduced[2] from the intensities of the stronger spots in stationary-film photographs. We have re-labelled the axes to conform to a standard space-group, but find the same hydrogen-bond nextwork (Figure 1) that was deduced by the earlier workers, with only minor changes in the cell dimensions and in the phosphorus and oxygen atom positions. Each NH_4^+ ion participates in four hydrogen bonds, and each anion oxygen atom in two. The dimensions of $H_2PO_2^-$ are normal,[1,4] as is the N..O distance of 2.84 Å. The cation and anion have

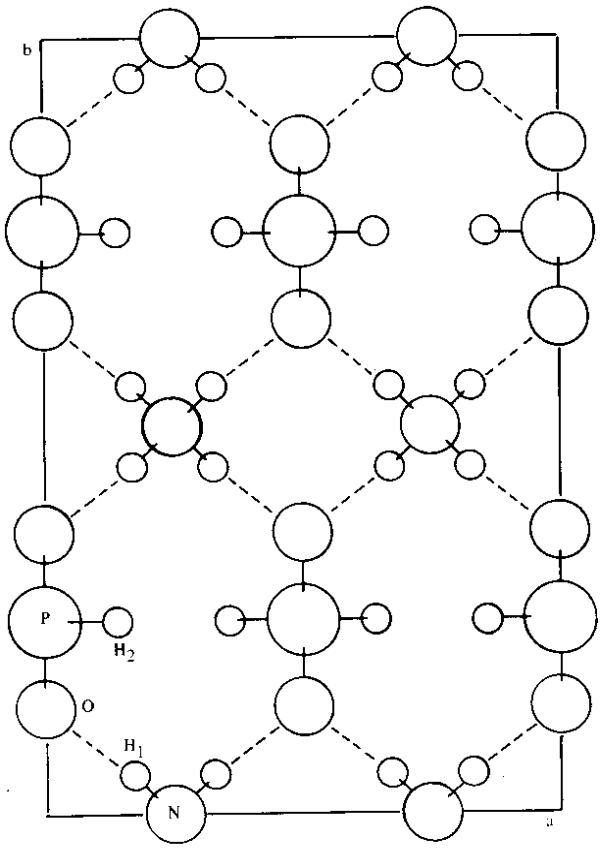

Figure 1. $(NH_4)H_2PO_2$: *c*-axis projection.

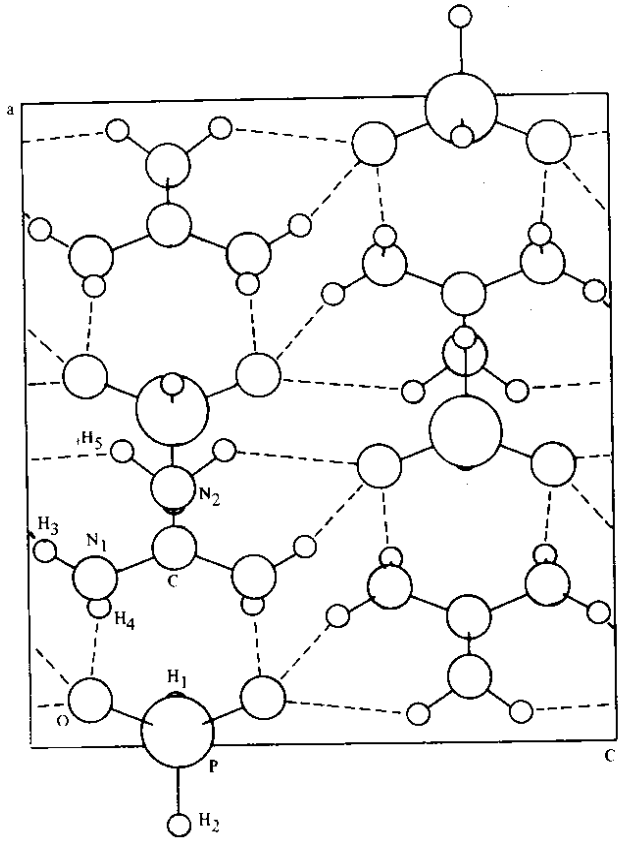

Figure 2. $(CH_6N_3)H_2PO_2$: *b*-axis projection.

222 and *mm* symmetry respectively.

The *b*-axis projection of the structure of GP is shown in Figure 2. Both the cation and anion have crystallographic symmetry *m*. Hydrogen bonds occur between N(1) and a pair of oxygen atoms O, O^{ii} related by the screw axis at (1/4, y, O), and between N(2) and O^{ii}. The hydrogen bond network forms a puckered net centred at x = 1/4. Each oxygen atom is the acceptor in three hydrogen bonds, involving the three independent hydrogen atoms of the cation. In addition, the $-N(2)H_2$ group is also weakly attracted by an oxygen atom O^{iii} of an anion in the adjacent net ('salt bond', not specifically involving a hydrogen atom). The C-N(2) bond is appreciably shorter (by 0.08 Å) than C-N(1). This implies more double-bond character in C-N(2) and more formal positive charge on N(2), and is to be contrasted with the effective trigonal symmetry observed for $CH_6N_3^+$ in the majority of its salts.[5] The cation is planar to within 0.011 Å, with N-C-N angles close to 120°. The dimensions of the anion are again normal.

The space group $P2_1/c$ for HP with Z = 2 implies that the $N_2H_6^{2+}$ cation is centro-symmetric (fully staggered conformation).

Table 1. *Atomic coordinates (x 10^4)*

(a) GP, $(CH_6N_3)H_2PO_2$

	x	y	z
P	198(3)	2400(4)	2500
O	721(6)	3252(8)	1017(6)
N(1)	2589(7)	6273(10)	1149(7)
N(2)	3990(10)	8134(13)	2500
C	3066(10)	6935(14)	2500
H(1)	630	670	2500
H(2)	−1190	2340	2500
H(3)	3140	6690	250
H(4)	1960	5080	1130
H(5)	4530	8250	1680

(b) AP, $(NH_4)H_2PO_2$

	x	y	z
P	0	2500	546(8)
O	0	1377(5)	3549(17)
N	2500	0	0
H(1)[a]	1710	520	1490
H(2)	1420	2500	7740

[a] Calculated position

Table 2 *Anisotropic thermal parameters* (x 10^4)

(a) GP, $(CH_6N_3)H_2PO_2$

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P	440(15)	391(14)	387(13)	0	0	0
O	632(34)	649(36)	340(27)	− 87(29)	25(25)	4(25)
N(1)	540(39)	704(46)	341(31)	−209(34)	−12(30)	32(33)
N(2)	541(58)	534(58)	472(53)	−173(48)	0	0
C	387(55)	352(53)	362(53)	− 9(42)	0	0

(b) AP, $(NH_4)H_2PO_2$

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P	166(13)	147(12)	161(15)	0	0	0
O	331(34)	203(27)	382(38)	0	0	−87(27)
N	220(45)	238(43)	297(57)	0	0	0

Table 3 *Interatomic distances (Å) and interbond angles (°)*

(a) GP, $(CH_6N_3)H_2PO_2$

P-O	1.501(5)	P-H(1)	1.35	N(1)-H(4)	1.07
N(1)-C	1.334(8)	P-H(2)	1.33	N(2)-H(5)	0.87
N(2)-C	1.252(12)	N(1)-H(3)	0.98		
$O\text{-}P\text{-}O^{i}$	115.1(4)	$N(1)\text{-}C\text{-}N(1^{i})$	119.8(1.1)	N(1)-C-N(2)	120.1(5)
O..N(1)	2.864(9)	O^{ii}..N	2.859(9)	O^{ii}..N(2)	3.017(5)
$N(2)..O^{iii}$	3.391(11)				

(b) AP, $(NH_4)H_2PO_2$

P-O	1.500(6)	P-H(2)	1.40	N..O	2.835(8)
$O\text{-}P\text{-}O^{iv}$	118.9(6)	O-P-H(2)	109	$H(2)\text{-}P\text{-}H(2^{v})$	99

Symmetry code: (i) x, y, ½-z (ii) ½-x,½+y,-z (iii) ½+x, 1½-y,z (iv) x, ½-y, z (v) -x, y, z

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR GUANIDINIUM PHOSPHINATE ABS CORRECTED

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	28	27	2	5	0	8	8	5	1	1	12	11	8	5	1	9	−10	6	2	2	16	16
4	0	0	43	42	3	5	0	4	3	6	1	1	18	−17	9	5	1	6	6	7	2	2	9	9
6	0	0	19	−17	4	5	0	3	−2	8	1	1	5	−4	10	5	1	3	−2	8	2	2	7	7
8	0	0	4	4	5	5	0	12	13	1	2	1	4	3	1	6	1	5	7	10	2	2	6	6
10	0	0	4	4	6	5	0	6	7	2	2	1	3	−3	2	6	1	6	4	1	3	2	6	5
1	1	0	24	−24	7	5	0	7	8	3	2	1	8	8	3	6	1	6	−7	2	3	2	10	−11
3	1	0	8	−8	8	5	0	10	10	4	2	1	26	24	4	6	1	3	3	3	3	2	4	4
4	1	0	42	−39	10	5	0	5	−7	5	2	1	9	6	5	6	1	3	0	4	3	2	9	−9
5	1	0	20	18	1	6	0	7	6	6	2	1	16	15	6	6	1	6	5	5	3	2	11	10
6	1	0	29	30	2	6	0	12	−13	7	2	1	10	−10	8	6	1	6	6	6	3	2	15	−15
7	1	0	5	2	3	6	0	3	−2	8	2	1	14	14	0	7	1	14	14	8	3	2	10	−9
10	1	0	6	−6	4	6	0	13	−15	10	2	1	9	9	2	7	1	11	10	9	3	2	3	−3
4	2	0	17	−17	5	6	0	5	3	0	3	1	16	40	4	7	1	7	7	10	3	2	7	−7
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3	3	0	11	12	5	7	0	4	6	8	3	1	11	12	2	0	2	40	−40	6	4	2	11	−10
4	3	0	18	18	6	7	0	5	5	9	3	1	4	4	4	0	2	17	−17	7	4	2	9	−9
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7	3	0	7	−8	3	8	0	4	4	2	4	1	9	−5	10	0	2	9	−8	2	5	2	3	4
8	3	0	11	11	4	8	0	11	11	3	4	1	7	7	1	1	2	5	−5	4	5	2	11	10
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10	4	0	6	6	3	1	1	21	−19	5	5	1	5	−6	4	2	2	21	21	7	6	2	5	6
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4	7	2	7	−7	6	3	3	20	−20	1	1	4	13	13	7	4	4	8	9	3	2	5	4	−3
5	7	2	4	5	7	3	3	9	8	2	1	4	3	−4	8	4	4	7	7	4	2	5	11	11
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4	4	5	8	−8
5	4	5	6	−6
6	4	5	7	−6
8	4	5	8	−8
0	5	5	18	−16
1	5	5	4	3
2	5	5	16	−16
3	5	5	9	−8
4	5	5	8	−7
6	5	5	9	−9
8	5	5	3	−3
1	6	5	5	−5
2	6	5	7	6
3	6	5	6	−6
4	6	5	3	3
6	6	5	6	5
2	6	8	5	−7
2	0	9	5	5
6	0	9	2	−3
2	1	9	11	−11
3	1	9	4	−4
4	1	9	9	−9
6	1	9	7	−8
1	2	9	5	5
4	2	9	3	3
5	2	9	3	3
0	3	9	9	7
2	3	9	8	9
3	3	9	8	7
4	3	9	8	8
5	3	9	3	2
1	4	9	6	5
2	4	9	3	2
3	4	9	4	3
0	0	10	4	−3
2	0	10	6	−6
4	0	10	4	−4
0	2	10	7	8
2	2	10	4	5
3	2	10	4	5
1	3	10	5	5

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR AMMONIUM PHOSPHINATE

H	H	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	0	0	60	70	4	0	1	38	−36	4	8	1	23	−22	6	2	2	21	−22	1	11	2	10	9
4	0	0	70	72	6	0	1	38	−37	6	8	1	27	−26	8	2	2	9	−8	3	11	2	9	9
6	0	0	35	36	8	0	1	18	−17	8	8	1	14	−12	1	3	2	32	29	5	11	2	7	7
8	0	0	29	31	10	0	1	16	−17	1	9	1	7	−6	3	3	2	29	28	0	12	2	16	17
2	2	0	70	−75	1	1	1	16	−14	3	9	1	6	−5	5	3	2	19	19	2	12	2	9	10
4	2	0	23	24	3	1	1	14	−12	7	9	1	5	−1	7	3	2	14	13	4	12	2	14	15
6	2	0	32	−33	5	1	1	8	−5	0	10	1	33	32	0	4	2	41	39	1	13	2	14	−15
8	2	0	15	−15	0	2	1	75	72	2	10	1	21	21	2	4	2	20	20	3	13	2	13	−14
0	4	0	24	25	2	2	1	23	23	4	10	1	28	27	4	4	2	33	32	0	14	2	8	−10
2	4	0	20	−16	4	2	1	43	41	6	10	1	14	14	6	4	2	14	13	2	14	2	11	−15
4	4	0	14	15	6	2	1	18	18	1	11	1	6	−4	8	4	2	16	16	4	14	2	7	−9
6	4	0	4	4	8	2	1	21	21	3	11	1	5	−4	1	5	2	40	−36	0	0	3	6	3
8	4	0	13	14	1	3	1	37	−34	5	11	1	4	−3	3	5	2	29	−28	4	0	3	6	2
0	6	0	8	−11	3	3	1	17	−16	0	12	1	8	−9	5	5	2	25	−22	1	1	3	14	14
2	6	0	30	−28	5	3	1	16	−15	2	12	1	17	−17	7	5	2	16	−15	3	1	3	14	15
4	6	0	8	−8	7	3	1	10	−10	4	12	1	8	−8	0	6	2	17	−17	0	2	3	19	20
6	6	0	21	−22	0	4	1	7	4	6	12	1	12	−12	2	6	2	33	−33	2	2	3	9	8
8	6	0	8	−9	2	4	1	28	−28	1	13	1	12	12	4	6	2	17	−16	4	2	3	17	17
0	8	0	56	55	4	4	1	6	−6	3	13	1	11	10	6	6	2	21	−20	1	3	3	23	−22
2	8	0	36	36	6	4	1	19	−19	0	14	1	13	13	8	6	2	9	−9	3	3	3	20	−21
4	8	0	41	41	8	4	1	7	−7	2	14	1	7	7	1	7	2	10	9	0	4	3	21	−23
6	8	0	21	21	1	5	1	38	34	4	14	1	11	11	3	7	2	11	10	2	4	3	31	−32
8	8	0	21	22	3	5	1	28	28	0	0	2	28	30	5	7	2	8	8	4	4	3	16	−16
0	10	0	31	−31	5	5	1	17	17	2	0	2	10	10	7	7	2	7	6	1	5	3	21	21
2	10	0	38	−38	7	5	1	13	12	4	0	2	29	27	0	8	2	23	23	3	5	3	21	20
4	10	0	22	−22	0	6	1	35	33	6	0	2	10	9	2	8	2	12	11	0	6	3	24	26
6	10	0	25	−25	2	6	1	12	13	8	0	2	15	15	4	8	2	21	20	2	6	3	14	14
8	10	0	11	−12	4	6	1	28	28	10	0	2	5	5	6	8	2	8	9	4	6	3	20	20
0	12	0	16	17	6	6	1	11	11	1	1	2	5	3	8	8	2	13	12	1	7	3	14	−13
2	12	0	7	8	8	6	1	16	16	3	1	2	4	1	1	9	2	4	1	3	7	3	11	−12
4	12	0	14	15	1	7	1	9	−7	5	1	2	4	−3	3	9	2	6	0	0	8	3	7	−1
0	14	0	5	−4	3	7	1	6	−2	7	1	2	5	−4	5	9	2	4	−1	2	8	3	10	−10
2	14	0	10	−11	5	7	1	5	−4	9	1	2	6	−3	0	10	2	8	−8	4	8	3	10	−2
4	14	0	5	−4	7	7	1	5	−4	0	2	2	11	−12	2	10	2	18	−18	1	9	3	9	10
0	0	1	41	49	0	8	1	30	30	2	2	2	34	35	4	10	2	8	8	3	9	3	9	10
2	0	1	93	87	2	8	1	42	41	4	2	2	15	14	6	10	2	13	12	0	10	3	7	9
2	10	3	7	1	1	1	4	21	21	3	3	4	13	14	0	6	4	7	6	3	9	4	13	14
4	10	3	7	7	3	1	4	18	−19	0	4	4	17	18	2	6	4	11	−12	2	10	4	9	−5
1	11	3	13	−12	0	2	4	6	−3	2	4	4	9	10	1	7	4	14	14	1	11	4	13	12
3	11	3	10	−12	2	2	4	9	−10	4	4	4	15	14	3	7	4	13	12	3	11	4	11	11
0	0	4	6	4	4	2	4	8	−2	1	5	4	11	−12	0	8	4	8	5	1	1	5	14	17
2	0	4	8	−3	1	3	4	15	16	3	5	4	11	−11	1	9	4	15	−15	3	1	5	15	15

Acknowledgement

I thank Mr. J.N. Low and Mrs. M. Bern for their assistance with the work on AP.

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