

C7—O71	1.207 (9)	C32—O33	1.186 (6)	Hall, S. R. & Stewart, J. M. (1990). Editors. <i>Xtal3.0 Reference Manual</i> . Univs. of Western Australia, Australia, and Maryland, USA.
C7—O71B	1.20 (2)	C32—C34	1.505 (7)	Kutschabsky, L., Adam, G. & Voigt, B. (1987). <i>Z. Chem.</i> <b>27</b> , 73–74.
C7—O72	1.293 (6)	O72—C73	1.439 (8)	Kutschabsky, L., Reck, G., Adam, G., Argay, G. & Czugler, M. (1981). <i>J. Prakt. Chem.</i> <b>323</b> , 829–835.
C8—C9	1.505 (6)	O111—C112	1.351 (6)	McLaughlin, G. M. (1983). <i>PWREDU</i> . Program for data reduction for Philips PW1100/20 diffractometer. Australian National Univ., Canberra, Australia.
C8—C14	1.515 (6)	C112—O113	1.190 (7)	Nakanishi, K., Endo, M., Näf, U. & Johnson, L. F. (1971). <i>J. Am. Chem. Soc.</i> <b>93</b> , 5579–5581.
C8—C15	1.530 (6)	C112—C114	1.489 (8)	Peter-Katalinic, J., Zsindely, J., Schmid, H. & Oberhänsli, W. E. (1975). <i>Helv. Chim. Acta</i> , <b>58</b> , 2517–2524.
C2—C1—C10	119.3 (4)	C10—C9—C15	115.2 (3)	Sheldrick, G. M. (1985). <i>SHELXS86</i> . Program for the solution of crystal structures. Univ. of Göttingen, Germany.
C1—C2—C3	113.4 (4)	C11—C9—C15	119.6 (4)	
C1—C2—O21	106.2 (4)	C1—C10—C5	122.1 (4)	
C3—C2—O21	99.9 (4)	C1—C10—C9	127.8 (4)	
C2—C3—C4	100.0 (4)	C5—C10—C9	109.5 (4)	
C2—C3—O31	114.8 (4)	C9—C11—C12	113.0 (4)	
C4—C3—O31	109.4 (4)	C9—C11—O111	105.1 (3)	
C3—C4—C5	105.5 (4)	C12—C11—O111	109.6 (4)	
C3—C4—C18	114.1 (4)	C11—C12—C13	110.5 (4)	
C3—C4—C19	98.4 (4)	C12—C13—C14	111.4 (4)	
C5—C4—C18	116.0 (4)	C12—C13—C16	105.3 (4)	
C5—C4—C19	110.2 (4)	C14—C13—C16	103.7 (4)	
C18—C4—C19	111.1 (4)	C8—C14—C13	101.4 (4)	
C4—C5—C6	119.2 (4)	C8—C15—C9	59.0 (3)	
C4—C5—C10	111.0 (4)	C8—C15—C16	104.7 (4)	
C6—C5—C10	104.8 (3)	C9—C15—C16	114.7 (4)	
C5—C6—C7	111.8 (4)	C13—C16—C15	105.6 (4)	
C5—C6—C8	104.2 (3)	C13—C16—O161	127.6 (4)	
C7—C6—C8	111.3 (4)	C15—C16—O161	126.7 (4)	
C6—C7—O71	126.9 (6)	C4—C19—O21	111.3 (4)	
C6—C7—O72	113.7 (4)	C4—C19—O191	128.1 (5)	
O71—C7—O72	118.5 (7)	O21—C19—O191	120.6 (4)	
C6—C8—C9	110.3 (4)	C2—O21—C19	106.0 (3)	
C6—C8—C14	127.2 (4)	C3—O31—C32	117.3 (4)	
C6—C8—C15	116.6 (3)	O31—C32—O33	123.9 (5)	
C9—C8—C14	115.0 (4)	O31—C32—C34	110.0 (4)	
C9—C8—C15	60.3 (3)	O33—C32—C34	126.1 (5)	
C14—C8—C15	108.7 (3)	C7—O72—C73	118.3 (4)	
C8—C9—C10	105.7 (4)	C11—O111—C112	117.3 (4)	
C8—C9—C11	118.6 (4)	O111—C112—O113	123.8 (5)	
C8—C9—C15	60.7 (3)	O111—C112—C114	110.1 (5)	
C10—C9—C11	121.0 (4)	O113—C112—C114	126.1 (5)	

Intensity data were collected with  $\theta$ -scan widths of  $(0.8 + 0.346\tan\theta)^\circ$ , a  $\theta$ -scan rate of  $2^\circ \text{ min}^{-1}$  and background counts of 7 s at each end of every scan. H atoms, with the exception of those on C34 and C114, were observed in a  $\Delta\rho$  synthesis. H atoms were placed at geometrically determined positions (those on C34 and C114 were assumed to be disordered over the two orientations giving rise to staggered conformations with respect to the  $\beta$  atoms) and held fixed during the least-squares refinement, but were periodically recalculated. All non-methyl H atoms were given a common  $U$  parameter which was refined, and methyl H atoms were treated likewise. The absolute configuration was assigned to conform with the known chirality of the compound's precursors. Refinement was by full-matrix least-squares methods.

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 55050 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1000]

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## Structure of $\text{Ni}(\text{H}_2\text{O})_6(\text{NH}_4)_2(\text{PO}_3\text{F})_2$

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## Abstract

The structure of diammonium hexaaquanickel bis(monofluorophosphate) consists of  $\text{Ni}(\text{H}_2\text{O})_6^{2+}$  and  $\text{NH}_4^+$  cations and  $\text{PO}_3\text{F}^{2-}$  anions held together by hydrogen bonds involving all H atoms except one belonging to the ammonium cation. The Ni atoms are octahedrally coordinated by the six water molecules.

## Comment

The present work forms part of a structural study of monofluorophosphates. The corresponding alkali-metal salts have been widely studied (Durand, Cot & Galigné, 1974, 1978; Galigné, Durand & Cot, 1974; Durand, Granier, Cot & Galigné, 1975; Payen, Durand, Cot & Galigné, 1979). However, the fluorophosphates of divalent transition metals have received less attention. Only the structures of

$\text{ZnPO}_3\text{F}\cdot\frac{5}{2}\text{H}_2\text{O}$  (Durand, Larbot, Cot, Duprat & Dabosi, 1983) and  $\text{CoPO}_3\text{F}\cdot 3\text{H}_2\text{O}$  (Durand, Cot, Berraho & Rafiq, 1987) are known. In addition, the structure of  $\text{SnPO}_3\text{F}$  (Berndt, 1974) has been published. In this work, we report the synthesis and crystal structure of the title compound, a new mixed phase which was isolated during experiments aimed at the synthesis of  $\text{NiPO}_3\text{F}\cdot x\text{H}_2\text{O}$ .

The structure is represented in Fig. 1, viewed along the  $a$  axis. It can be described as layers, parallel to the  $ac$  plane, in which  $\text{PO}_3\text{F}^{2-}$  anions and  $\text{NH}_4^+$  and  $\text{Ni}(\text{H}_2\text{O})_6^{2+}$  cations are held together by hydrogen bonds, involving F, N and all the other O atoms. The bridging H atoms are H(2) and H(3) (both belonging to  $\text{NH}_4^+$ ) and H(41), H(51), H(61) and H(62) of the water molecules. The layers are joined by additional hydrogen bonds connecting water molecules [O(4) and O(5)] with O(2) and O(3) of the  $\text{PO}_3\text{F}^{2-}$  group, the bridging atoms being H(1), H(42) and H(52). Thus, H(4) is the only H atom not involved in the hydrogen-bond interactions.

The Ni atoms, located at centres of symmetry, are coordinated by six water molecules forming a distorted octahedron; the Ni—O distances are as expected. The same can be said of the P—O distances. The P—F distance [1.601 (1) Å] is similar to that obtained by theoretical calculations [1.599 Å (Serafini, Labarre, Granier & Cot, 1976)], but is significantly longer than those observed in  $\text{ZnPO}_3\text{F}\cdot\frac{5}{2}\text{H}_2\text{O}$  (Durand, Larbot, Cot, Duprat & Dabosi, 1983) and in  $\text{CoPO}_3\text{F}\cdot 3\text{H}_2\text{O}$  [1.548 (6) and 1.567 (2) Å, respectively (Durand, Cot, Berraho &

Rafiq, 1987)]. Following the interpretation of Durand, Granier, Cot & Galigné (1975), this lengthening could be correlated with a weakening of the P—F bond, owing to the hydrogen-bond interaction which does not occur in the Co and Zn compounds where the F atoms are only bonded to the P atoms. The Ni atoms are only coordinated by water molecules. In contrast, the metal environment in the Co and Zn derivatives is formed by O atoms from both the fluorophosphate anions and the water molecules.

## Experimental

### Crystal data

$\text{Ni}(\text{H}_2\text{O})_6(\text{NH}_4)_2(\text{PO}_3\text{F})_2$	$\theta = 13\text{--}17^\circ$
$M_r = 398.8$	$V = 688.8 (2) \text{ \AA}^3$
Monoclinic	$Z = 2$
$P2_1/c$	$D_x = 1.92 \text{ Mg m}^{-3}$
$a = 6.292 (1) \text{ \AA}$	Mo $K\alpha$
$b = 12.329 (1) \text{ \AA}$	$\lambda = 0.7107 \text{ \AA}$
$c = 9.233 (1) \text{ \AA}$	$\mu = 1.72 \text{ mm}^{-1}$
$\beta = 105.91 (1)^\circ$	$T = 293 \text{ K}$
Cell parameters from 25 reflections	Prismatic
	$0.4 \times 0.3 \times 0.3 \text{ mm}$
	Green

### Data collection

CAD-4 four-circle diffractometer	$R_{\text{int}} = 0.0064$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 30^\circ$
Absorption correction: none	$h = -8 \rightarrow 8$
2104 measured reflections	$k = 0 \rightarrow 17$
1990 independent reflections	$l = 0 \rightarrow 12$
1813 observed reflections	2 standard reflections
	frequency: 60 min
	intensity variation: 27%
	$[I > 2\sigma(I)]$

### Refinement

Refinement on $F$	$(\Delta/\sigma)_{\text{max}} = 0.51$
Final $R = 0.030$	$\Delta\rho_{\text{max}} = 0.54 \text{ e \AA}^{-3}$
$wR = 0.034$	$\Delta\rho_{\text{min}} = -0.71 \text{ e \AA}^{-3}$
$S = 0.996$	Atomic scattering factors
1813 reflections	from <i>International Tables</i>
118 parameters	for <i>X-ray Crystallography</i>
Only H-atom coordinates refined	(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{Å}^2$ )

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	$x$	$y$	$z$	$U_{\text{eq}}$
Ni	0.0	0.0	0.0	0.01497 (9)
P	0.26282 (6)	0.13583 (3)	0.60022 (4)	0.01638 (11)
F	0.2048 (2)	0.06556 (10)	0.44908 (12)	0.03894 (3 <sup>a</sup> )
O(1)	0.3839 (2)	0.05624 (9)	0.71767 (9)	0.02297 (3)
O(2)	0.0410 (2)	0.17322 (10)	0.61413 (15)	0.02721 (3 <sup>a</sup> )
O(3)	0.4086 (2)	0.22550 (10)	0.57021 (15)	0.02701 (3 <sup>a</sup> )
O(4)	0.2926 (2)	0.06702 (10)	-0.00613 (13)	0.02158 (3)
O(5)	-0.0422 (2)	0.11695 (10)	0.15131 (13)	0.02444 (3 <sup>a</sup> )
O(6)	0.1671 (2)	-0.09893 (10)	0.17483 (13)	0.02349 (3 <sup>a</sup> )
N	0.6428 (2)	0.16624 (12)	0.35935 (17)	0.02627 (4 <sup>a</sup> )

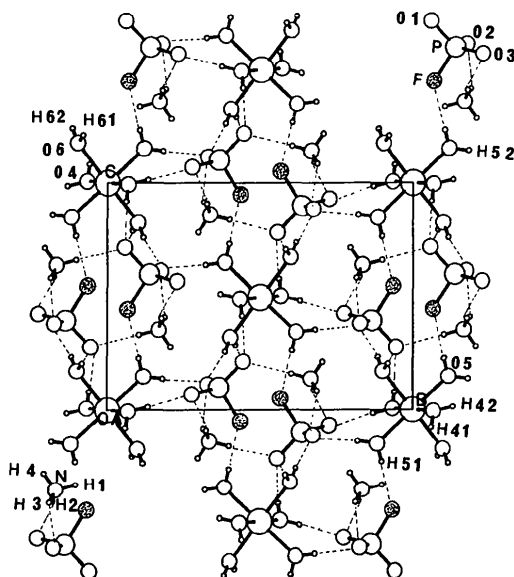


Fig. 1. PLUTO drawing (Motherwell & Clegg, 1978) of the structure of the title compound projected along the  $a$  axis. Dotted lines represent the hydrogen-bond interactions. The F atoms are shaded.

Table 2. Significant bond lengths (Å) and angles (°)

PO <sub>3</sub> F <sup>2-</sup>			
P—F	1.601 (1)	F—P—O(1)	103.10 (7)
P—O(1)	1.508 (1)	F—P—O(2)	104.22 (7)
P—O(2)	1.510 (1)	F—P—O(3)	103.90 (8)
P—O(3)	1.511 (1)	O(1)—P—O(2)	115.73 (7)
		O(1)—P—O(3)	113.40 (7)
		O(2)—P—O(3)	114.50 (7)
Ni(H <sub>2</sub> O) <sub>6</sub> <sup>2+</sup>			
Ni—O(4)	2.034 (1) × 2	O(4)—Ni—O(5)	91.18 (5)
Ni—O(5)	2.079 (1) × 2	O(4)—Ni—O(6)	89.52 (5)
Ni—O(6)	2.070 (1) × 2	O(5)—Ni—O(6)	90.79 (5)
O—H(mean)	0.80 (4)	O(4)—Ni—O(6)	90.48 (5)
		O(5)—Ni—O(6)	89.21 (5)
		O(4)—Ni—O(5)	88.82 (5)
		H—O—H(mean)	107 (4)

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

Table 3. Hydrogen-bond geometry (Å, °)

D...A	D...A	H...A	H atom	Angle at H
O(1)...O(4)	2.774 (2)	1.98 (4)	H(41)	170 (3)
O(1)...O(6)	2.778 (2)	1.99 (4)	H(61)	173 (4)
O(1)...N <sup>iv</sup>	2.830 (2)	2.01 (4)	H(1)	173 (4)
O(2)...O(6 <sup>iv</sup> )	2.789 (2)	1.97 (4)	H(62)	174 (4)
O(2)...N <sup>iv</sup>	2.935 (2)	2.07 (4)	H(3)	148 (4)
O(3)...O(4')	2.703 (2)	1.90 (4)	H(42)	171 (3)
O(3)...N	2.846 (2)	1.99 (4)	H(2)	171 (3)
O(5)...F	2.841 (2)	2.05 (4)	H(51)	176 (5)
O(5)...O(2')	2.683 (2)	1.87 (4)	H(52)	179 (4)

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

The title compound was prepared from a mixture (molar ratio 1:1) of aqueous solutions of (NH<sub>4</sub>)<sub>2</sub>PO<sub>3</sub>F·H<sub>2</sub>O and Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O. Green single crystals were grown by slow evaporation at room temperature. The IR spectrum revealed the presence of both NH<sub>4</sub><sup>+</sup> and PO<sub>3</sub>F<sup>2-</sup> ions. Crystals decomposed in air; to collect data, they were mounted in sealed glass capillaries. In spite of this, crystal decay was observed during data collection. The final intensities of standard reflections dropped by up to 73 % of their initial values. Intensities were collected with a scan amplitude of 1.5° and speed of 0.6° s<sup>-1</sup>. Calculations were performed with *DIRDIF* (Beurskens *et al.*, 1984), *XRAY80* (Stewart, Kundell & Baldwin, 1980), *PE-SOS* (Martínez-Ripoll & Cano, 1975) and *PLUTO* (Motherwell & Clegg, 1978). The weighting scheme applied by *PE-SOS* was  $w = K/F(f)F(s)$ , where  $K = 0.5785$ ,  $F(s) = 6.4971 - 21.0696(\sin\theta/\lambda) + 18.9012(\sin\theta/\lambda)^2$  and  $F(f) = a + bF_{\text{obs}} + cF_{\text{obs}}$ . The values of  $a$ ,  $b$  and  $c$  were as follows:

$a$	$b$	$c$	$F_{\text{obs}} < 7.5$
1.1255	-0.2964	0.0269	0.0
0.2515	0.0118	0.0	$7.5 < F_{\text{obs}} < 20.0$
-0.0032	0.0235	0.0	$20.0 < F_{\text{obs}} < 29.0$
-0.3053	0.0329	0.0	$29.0 < F_{\text{obs}}$

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54998 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1003]

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(±)-*cis*-6-Acetylamino-5-hydroxy-1,3-dioxepane

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## Abstract

The 1,3-dioxepane ring adopts a twist-boat conformation with the acetylamino and hydroxy groups occupying *cis* positions with respect to the ring. Two intramolecular hydrogen bonds [N—H...OH of 2.774(4) and O—H...OCH<sub>2</sub> of 2.808(3) Å] fix the conformation, and to-