Table 4. *Selected geometric parameters* (Å, °) *for* $[Tb_2(CrO_4)_3(H_2O)_5].2H_2O$

Tbl—O3	2.461 (8)	Tb2—O9	2.298 (7)
Tbl—O4	2.438 (7)	Tb2—O10	2.320 (5)
Tbi—O6	2.350 (7)	Tb2—O(*W*)5	2.405 (4)
Tbl—O8	2.410 (5)	Cr1—O1	1.668 (6)
Tbl—O12	2.356 (7)	Cr1—O2	1.664 (6)
Tbl—O(*W*)1	2.499 (6)	Cr1—O3	1.627 (8)
Tbl—O(*W*)2	2.407 (9)	Cr1—O4	1.648 (6)
Tbl—O(*W*)3	2.453 (8)	Cr2—O5	1.678 (6)
Tbl—O(*W*)4	2.518 (9)	Cr2—O6	1.634 (6)
Tb2—O1	2.408 (6)	Cr2—O7	1.638 (9)
Tb2—O1	2.574 (7)	Cr2—O8	1.634 (6)
Tb2—O2	2.507 (7)	Cr3—O9	1.663 (8)
Tb2—O5	2.463 (8)	Cr3—O10	1.670 (5)
Tb2—O5	2.441 (8)	Cr3—O11	1.617 (8)
Tb2—O7	2.608 (7)	Cr3—O12	1.653 (7)
O1—Cr1—O2	101.9 (3)	O6—Cr2—O7	110.3 (4)
O1—Cr1—O3	112.7 (4)	O6—Cr2—O8	109.6 (4)
O1—Cr1—O4	112.2 (3)	O7—Cr2—O8	110.9 (4)
O2—Cr1—O3	110.4 (3)	O9—Cr3—O10	110.4 (3)
O2—Cr1—O4	110.8 (4)	O9—Cr3—O11	108.8 (4)
O3—Cr1—O4	108.7 (4)	O9—Cr3—O12	110.5 (4)
O5—Cr2—O6	112.7 (4)	O10—Cr3—O11	108.6 (4)
O5—Cr2—O7	102.8 (4)	O10—Cr3—O12	110.2 (3)
O5—Cr2—O8	110.4 (3)	O11—Cr3—O12	108.4 (4)

Cell constants were obtained from a least-squares refinement. Lorentz and polarization corrections were applied to the data. Positions of lanthanoid and Cr atoms were established by direct methods and the remaining atoms were located in subsequent $\Delta\rho$ maps. H atoms were not located. All calculations were performed with a MicroVAX 3100 computer using the Enraf-Nonius structure determination package *SDP-Plus* (Frenz, 1978) which incorporates the direct-methods program *MULTAN*11/82 (Main *et al.*, 1982).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71654 (64 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1084]

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A Copper Monofluorophosphate, $Cu(H_2O)_2(NH_4)_2(PO_3F)_2$

M. Berraho

Laboratoire de Chimie de la Coordination,
Faculté de Sciences, Semlalia Université Cadi Ayyad,
Marrakech, Morocco

A. Vegas* and M. Martínez-Ripoll

Departamento de Cristalografía,
Instituto de Química-Física Rocasolano, CSIC,
Serrano 119, E-28006 Madrid, Spain

M. Rafiq

Laboratoire de Chimie Minérale, Faculté de Sciences,
Fès, Morocco

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Abstract

The structure of diammonium diaquabis(monofluorophosphato)copper, $2[NH_4][Cu(PO_3F)_2(H_2O)_2]$, consists of CuO_6 octahedra and NH_4 and PO_3F tetrahedra held together by hydrogen bonds involving one H atom of the ammonium group and the H atoms of the water molecules. The Cu atoms are octahedrally coordinated by two water molecules at the apical positions and four O atoms belonging to four PO_3F groups.

Comment

This work forms part of a structural study of monofluorophosphates (Berraho, R'Kha, Vegas & Rafiq, 1992, and references therein). The structure of the title compound (Fig. 1) can be described on the basis of $Cu(H_2O)_2(PO_3F)_4^{2-}$ complex anions and NH_4^+ cations. The Cu atoms have octahedral coordination, which is somewhat deformed by the Jahn–Teller

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effect, where the two water molecules occupy the apical positions and the four fluorophosphate groups are each coordinated through one O atom (Table 2). In the PO_3F groups, the P—O distances (mean value 1.50 Å) are similar to those found in the Co and Ni compounds (Durand, Cot, Berraho & Rafiq, 1987; Berraho, R'Kha, Vegas & Rafiq, 1992). The P—F distance [1.577 (4) Å] also agrees with values reported for those compounds. The crystal structure is held together mainly by hydrogen bonds of the type N—H···O and O—H···O (Table 3).

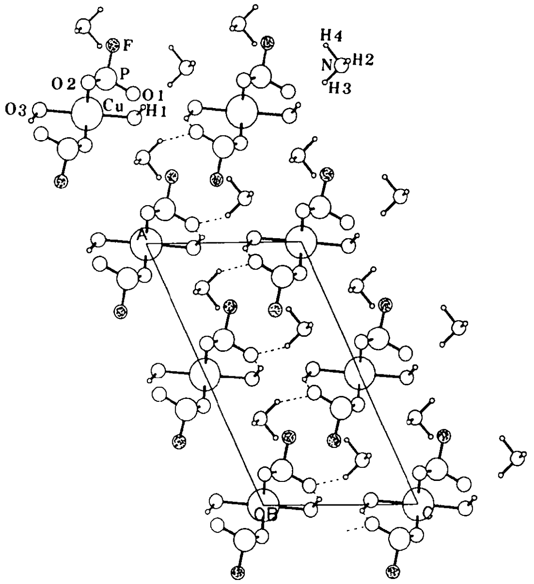

Fig. 1. The structure of the title compound viewed along the *b* axis. Dotted lines represent hydrogen bonds. F atoms are shown as shaded circles.

Experimental

Synthesis was carried out from a mixture of aqueous solutions of $(NH_4)_2PO_3F.H_2O$ and $Cu(NO_3)_2.6H_2O$ in the molar ratio 1:1. Single crystals were grown by slow evaporation at room temperature.

Crystal data

$2[NH_4][Cu(PO_3F)_2(H_2O)_2]$
M_r = 331.6
Monoclinic
$C2/m$
a = 13.454 (2) Å
b = 5.243 (2) Å
c = 7.518 (2) Å
β = 114.59 (2)°
V = 482.2 (4) $Å^3$
Z = 2

D_x = 2.28 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 38 reflections
θ = 10–30°
μ = 7.4 mm^{-1}
T = 293 K
Prismatic
0.30 × 0.20 × 0.15 mm
Blue

Data collection

Four-circle XRD 3000S diffractometer
$\omega/2\theta$ scans (width 1.5°, speed 0.05° s^{-1})
Absorption correction: none
497 measured reflections
461 independent reflections
461 observed reflections

R_{int} = 0.032
θ_{max} = 65°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 6$
$l = -8 \rightarrow 8$
2 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F
R = 0.048
wR = 0.045
S = 0.996
461 reflections
54 parameters
Only coordinates of H atoms refined
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.35

$\Delta\rho_{max}$ = 0.35 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.41 e $Å^{-3}$
Extinction correction: least squares
Extinction coefficient: 0.26 × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($Å^2$)

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

	x	y	z	U_{eq}
Cu	0	0	0	0.007 (1)
P	0.1313 (1)	−1/2	0.2198 (3)	0.010 (1)
F	0.2570 (3)	−1/2	0.3601 (7)	0.025 (2)
O(1)	0.0730 (4)	−1/2	0.3513 (8)	0.017 (2)
O(2)	0.1182 (3)	−0.2618 (8)	0.1003 (6)	0.021 (1)
O(3)	0.4823 (5)	1/2	0.2909 (9)	0.029 (2)
N	0.8313 (6)	1/2	0.2326 (10)	0.019 (3)
H(1)	0.522 (6)	0.672 (16)	0.377 (11)	
H(2)	0.814 (6)	0.650 (15)	0.178 (11)	
H(3)	0.894 (9)	1/2	0.388 (16)	
H(4)	0.754 (9)	1/2	0.274 (15)	

Table 2. *Selected geometric parameters* (Å, °)

P—F	1.577 (4)		N—H(3)	1.12 (10)	
P—O(1)	1.496 (7)		N—H(4)	1.20 (14)	
P—O(2)	1.505 (5)	×2			
O(3)—H(1)	1.11 (8)		Cu—O(2)	1.996 (4)	×4
N—H(2)	0.88 (8)		Cu—O(3)	2.296 (8)	×2
F—P—O(1)	105.6 (2)		O(3)—Cu—O(3)	180.0 (6)	
F—P—O(2)	103.1 (2)	×2	O(2)—Cu—O(2)	180.0 (6)	×2
O(1)—P—O(2)	115.5 (3)	×2	O(2)—Cu—O(2)	93.1 (2)	×2
O(2)—P—O(2)	112.1 (3)		O(2)—Cu—O(2)	86.9 (2)	×2
H(2)—N—H(3)	115 (7)	×2	O(3)—Cu—O(2)	91.3 (2)	×4
H(2)—N—H(4)	92 (6)	×2	O(3)—Cu—O(2)	88.7 (3)	×4
H(3)—N—H(4)	95 (9)	×2			
H(2)—N—H(2)	129 (7)	×2			

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	A	D—H	H···A	D···A	D—H···A
N	H(3)	$O(1^i)$	1.12 (10)	1.82 (12)	2.845 (9)	150 (8)
O(3)	H(1)	$O(1^{ii})$	1.11 (8)	1.89 (9)	2.847 (3)	142 (6)

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

Calculations were performed using *DIRDIF* (Beurskens *et al.*, 1984), *XRAY*80 (Stewart, Kundell & Baldwin, 1980) and *PLUTO* (Motherwell & Clegg, 1978).

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Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71703 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1067]

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A Precise Structure Redetermination of Nickel Ammonium Sulfate Hexahydrate, $Ni(H_2O)_6.2NH_4.2SO_4$

Tahir H. Tahirov and Tian-Huey Lu

Department of Physics, National Tsing Hua University, Hsinchu, Taiwan 300

Chiung-Chia Huang

Department of Applied Chemistry, Chung Cheng Institute of Technology, Ta-Hsi, Taiwan 33508

Chung-Sun Chung

Department of Chemistry, National Tsing Hua University, Hsinchu, Taiwan 300

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Abstract

The $Ni(H_2O)_6$ ion is located at an inversion center. Six octahedral water molecules surround the Ni^{II} ion and form hydrogen bonds with the sulfate groups. Each ammonium group binds to the sulfate groups through hydrogen bonds.

Comment

This structure has been reported by Grimes, Kay & Webb (1963) and Montgomery & Lingafelter (1964), but the present structure is more precise in several respects.

The Ni^{II} ion is located at an inversion center. The six water molecules surrounding the Ni^{II} ion form octahedral geometry, with Ni—O(*W*) distances spanning the narrow range 2.041 (2)–2.067 (2) Å. The water molecules bind the sulfate groups through hydrogen bonds. Hydrogen bonds also exist between the ammonium and sulfate groups. In the sulfate ion, S—O(4) is *ca* 0.02 Å shorter than the other three S—O bonds. This is apparently due to differences in the number of hydrogen bonds accepted by these atoms; O(4) accepts only one hydrogen bond while O(1), O(2) and O(3) each accept three.

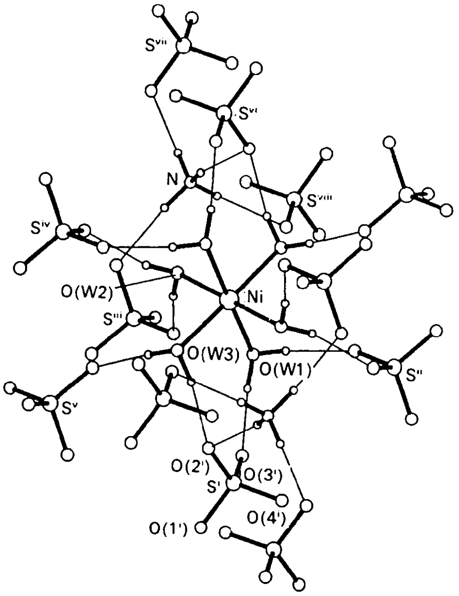

Fig. 1. A perspective view of the molecule with hydrogen bonds (thin lines) in the crystal structure. The Ni atom is situated at the inversion center. Symmetry codes: (i) $x, 0.5-y, -0.5+z$; (ii) $x, -1+y, -1+z$; (iii) $1-x, 1-y, 1-z$; (iv) $-x, 1-y, 1-z$; (v) $x, -1+y, z$; (vi) $-x, -0.5+y, 0.5-z$; (vii) $x, 1.5-y, -0.5+z$; (viii) $1-x, -0.5+y, 0.5-z$.

Experimental

Crystal data

$Ni(H_2O)_6.2NH_4.2SO_4$
$M_r = 394.99$
Monoclinic
$P2_1/c$
$a = 6.244$ (2) Å
$b = 12.469$ (4) Å
$c = 9.195$ (3) Å
$\beta = 106.98$ (3)°

$D_x = 1.916$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 9.5-17.5°$
$\mu = 1.81$ mm^{-1}
$T = 298$ (3) K
Parallelepiped

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