

Table 3. Distances (Å) and angles (°) between atoms possibly involved in hydrogen bonding

N(1)···O(1)	3.078 (7)	Pt—N(1)···O(1)	99.0 (2)
N(1)···O(2)	3.040 (7)	Pt—N(1)···O(2)	102.9 (2)
N(1)···O(3)	2.960 (8)	Pt—N(1)···O(3)	88.7 (2)
N(1)···O(4)	3.158 (7)	Pt—N(1)···O(4)	95.4 (2)
N(2)···O(1)	2.992 (8)	Pt—N(2)···O(1)	101.9 (2)
N(2)···O(2)	2.998 (8)	Pt—N(2)···O(2)	104.4 (2)
N(2)···O(3)	3.149 (7)	Pt—N(2)···O(3)	83.7 (2)
N(2)···O(4)	3.236 (8)	Pt—N(2)···O(4)	93.2 (2)

strong. In $\text{NH}_4[\text{TcO}_4]$ the hydrogen-bonded distances, $\text{N}\cdots\text{O}$, were found to be 2.899 (2) and 2.984 (2) Å at 298 K (Faggiani, Lock & Pocé, 1980).

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Crystal Chemistry of *cyclo*-Hexaphosphates. V. Structure of Cadmium Sodium *cyclo*-Hexaphosphate Tetradecahydrate

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Abstract. $\text{Cd}_2\text{Na}_2\text{P}_6\text{O}_{18}\cdot 14\text{H}_2\text{O}$, $M_r = 996.816$, triclinic, $P\bar{1}$, $a = 7.709$ (1), $b = 11.028$ (6), $c = 9.231$ (2) Å, $\alpha = 108.25$ (5), $\beta = 110.06$ (5), $\gamma = 79.77$ (5)°, $V = 698$ (1) Å³, $Z = 1$, $D_x = 2.371$ Mg m⁻³, $\lambda(\text{Ag } K\alpha) = 0.5608$ Å, $\mu = 1.036$ mm⁻¹, $F(000) = 492$, $T = 294$ K, final $R = 0.019$ for 3182 reflections. The atomic arrangement can be described as rows of CdO_6 octahedra alternating with the P_6O_{18} anions along the c axis. These rows are interconnected by chains of edge-sharing and corner-sharing octahedra parallel to the a axis. Cd/Na disorder is observed on a general position while a water molecule belonging to a sodium coordination sphere is statistically distributed on two general positions. The hydrogen-bond scheme is described.

Introduction. During the course of a systematic investigation of *cyclo*-hexaphosphate crystal chemistry we previously reported the chemical preparation and crystal structure of the first example of a hydrated monovalent divalent *cyclo*-hexaphosphate: $\text{Li}_2\text{Mn}_2\text{P}_6\text{O}_{18}\cdot 10\text{H}_2\text{O}$ (Averbuch-Pouchot, 1989).

In the present work we describe the chemical preparation and crystal structure for a second compound of this type: $\text{Cd}_2\text{Na}_2\text{P}_6\text{O}_{18}\cdot 14\text{H}_2\text{O}$.

This salt exhibits several features rather unusual in the crystal chemistry of condensed phosphates.

Experimental. Crystals of the title compound are prepared by adding an aqueous solution of cadmium chloride to an aqueous solution of sodium *cyclo*-hexaphosphate to obtain a Cd/Na stoichiometry of 1/2 in the final solution. By evaporation at room temperature, diamond-like thick plates of $\text{Cd}_2\text{Na}_2\text{P}_6\text{O}_{18}\cdot 14\text{H}_2\text{O}$ appear in the solution.

Crystal size: $0.32 \times 0.32 \times 0.15$ mm. Density not measured. Nonius CAD-4 diffractometer, graphite monochromator. 20 reflections ($10.0 < \theta < 11.5^\circ$) for refining unit-cell dimensions. ω scan, scan width: 1.20° , scan speed variable between 0.02 and $0.06^\circ \text{ s}^{-1}$, total background measuring time: between 30 and 10 s. 3380 reflections collected, $2 < \theta < 27.5^\circ$, $\pm h$, $\pm k$, l , $h_{\text{max}} = 12$, $k_{\text{max}} = 17$, $l_{\text{max}} = 15$. Two orientation (520 and $\bar{5}20$) and two intensity (430 and $\bar{4}30$) control reflections without any significant varia-

tion. 3182 reflections kept after averaging Friedel pairs ($R_{\text{int}} = 0.02$). Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods (*MULTAN77*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977). H atoms located by difference-Fourier synthesis. Anisotropic full-matrix least-squares refinements (on F), isotropic for H atoms. Unit weights. Final refinement cycles with 3182 reflections (no rejection). Final $R = 0.019$ ($wR = 0.022$), $S = 0.741$, max. $\Delta/\sigma = 0.03$, max. peak height in the final difference-Fourier synthesis $0.752 \text{ e } \text{Å}^{-3}$. No extinction correction. Scattering factors for neutral atoms and f' , f'' from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius (1977) *SDP* used for all calculations. Computer used: MicroVAXII. Several unusual problems occurred during this crystal structure determination. They are discussed below.

Discussion. Table 1 reports the final atomic coordinates.*

As shown by a projection along the c axis (Fig. 1), the atomic arrangement can be clearly described as follows: along the c axis centrosymmetrical CdO_6 octahedra and P_6O_{18} ring anions alternate. These rows are themselves interconnected by a chain of edge-sharing octahedra parallel to the a axis. The CdO_6 octahedra alternating with the phosphoric anions along the c axis are made up of four external O atoms of the P_6O_{18} ring and two water molecules. These octahedra are slightly distorted with Cd—O distances ranging from 2.338 to 2.249 Å and O—Cd—O angles from 88.03 to 88.85°. The P_6O_{18} rings located half-way between these octahedra are very significantly distorted since P—P—P angles vary from 94.25 to 142.81°.

In the chain of octahedra connecting these rows, the arrangement is more complicated. One Na atom and one Cd atom are statistically distributed on a general position $(-0.409, 0.394, -0.367)$ close to the inversion center at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. These atoms are surrounded by octahedra built up of five water molecules and one external O atom of the phosphoric ring. These octahedra have a common edge [$\text{O}(W6)\cdots\text{O}(W6)$] and are connected to the $\text{Na}(1)\text{O}_6$ octahedra by two common corners [$\text{O}(E21)$ and $\text{O}(W4)$] to form an infinite chain: $\cdots\text{Na}(1)\text{O}_6\cdots(\text{Na,Cd})\text{O}_6\cdots(\text{Na,Cd})\text{O}_6\cdots\text{Na}(1)\text{O}_6\cdots$ running along the a axis. Such a chain in projection along the b axis is reported in Fig. 2.

The average (Cd,Na)—O distance in the mixed (Cd,Na) O_6 octahedron is 2.359 Å, almost halfway

Table 1. *Final atomic coordinates and B_{eq} values for non-H atoms*

Starred atoms were refined isotropically. Estimated standard deviations are given in parentheses. Occupancy factors: 0.5 for H(W7), H(W8), O(W7) and O(W8).

$$B_{\text{eq}} = (4/3)\sum_i\sum_j\beta_{ij}a_i\cdot a_j$$

	x	y	z	$B_{\text{eq}}(\text{Å}^2)$
Na(1)	0	$\frac{1}{2}$	$\frac{1}{2}$	2.26 (3)
Na(Cd)	-0.40959 (1)	0.39358 (3)	-0.36685 (4)	1.700 (5)
Cd(1)	0	0	0	1.272 (4)
P(1)	-0.15104 (7)	-0.19810 (5)	0.14057 (6)	1.072 (9)
P(2)	-0.04156 (8)	0.20697 (5)	-0.46901 (6)	1.231 (9)
P(3)	-0.29220 (7)	0.07088 (5)	0.23180 (6)	1.057 (9)
O(E11)	0.2407 (2)	0.3167 (2)	0.8917 (2)	1.68 (3)
O(E12)	0.0769 (2)	0.1831 (2)	-0.0181 (2)	1.48 (3)
L(L13)	0.3007 (2)	0.0808 (1)	0.8205 (2)	1.31 (3)
O(L12)	-0.0159 (2)	0.1754 (2)	0.6959 (2)	1.43 (3)
O(E21)	0.1526 (3)	0.6678 (2)	0.4698 (2)	2.05 (4)
O(E22)	0.1379 (2)	0.1887 (2)	0.4980 (2)	2.03 (3)
O(L23)	0.8332 (2)	0.0916 (2)	0.4148 (2)	1.68 (3)
O(E31)	0.1899 (2)	0.8905 (2)	0.8565 (2)	1.58 (3)
O(E32)	0.5179 (2)	0.1308 (2)	0.2210 (2)	1.78 (3)
O(W1)	0.6284 (3)	0.3279 (2)	0.0486 (2)	2.44 (4)
O(W2)	0.5350 (3)	0.1945 (2)	0.6327 (3)	3.17 (5)
O(W3)	0.2193 (3)	0.5607 (2)	0.1080 (2)	2.75 (4)
O(W4)	0.6952 (3)	0.5446 (2)	0.3075 (2)	2.31 (4)
O(W5)	0.7568 (2)	0.9819 (2)	0.7602 (2)	1.95 (3)
O(W6)	0.4231 (3)	0.3831 (2)	0.3631 (2)	2.70 (4)
O(W7)	0.9488 (5)	0.5980 (4)	0.7600 (5)	2.36 (9)
O(W8)	0.0721 (5)	0.3402 (3)	0.2854 (4)	2.53 (7)
H(1W1)	0.682 (6)	0.263 (4)	0.069 (5)	3 (1)*
H(2W1)	0.540 (6)	0.319 (4)	1.010 (5)	3 (1)*
H(1W2)	0.397 (5)	0.860 (4)	0.317 (5)	2 (1)*
H(2W2)	0.553 (6)	0.822 (4)	0.392 (5)	3 (1)*
H(1W3)	0.775 (6)	0.514 (4)	0.950 (5)	3 (1)*
H(2W3)	0.761 (5)	0.399 (4)	0.932 (5)	2 (1)*
H(1W4)	0.685 (5)	0.483 (4)	0.244 (5)	2 (1)*
H(2W4)	0.287 (5)	0.400 (4)	0.723 (4)	2.2 (9)*
H(1W5)	0.216 (5)	0.071 (4)	0.323 (5)	2.4 (9)*
H(2W5)	0.313 (6)	0.053 (4)	0.231 (5)	2 (1)*
H(1W6)	0.469 (6)	0.308 (4)	0.313 (5)	3 (1)*
H(2W6)	0.327 (6)	0.374 (4)	0.342 (5)	4 (1)*
H(W7)	0.98 (1)	0.531 (8)	0.80 (1)	3 (2)*
H(W78)	0.958 (6)	0.680 (4)	0.825 (5)	3 (1)*
H(W8)	0.91 (1)	0.717 (8)	0.677 (9)	2 (2)*

between the average Cd—O in the $\text{Cd}(1)\text{O}_6$ octahedron (2.286 Å) and the average in the $\text{Na}(1)\text{O}_6$ octahedron (2.446 Å).

The situation around the second associated cation involved in this chain [$\text{Na}(1)$] is very unusual. Its coordination polyhedron is built up of four water molecules and two O atoms. During refinements, one of these water molecules [$\text{O}(W7)$] was affected by a very high thermal factor and in the difference-Fourier syntheses run to localize the H atoms an abnormally high residual electron density was observed close to this water molecule, suggesting that this water molecule must be statistically distributed on two positions, denoted $\text{O}(W7)$ and $\text{O}(W8)$. Refinements of the occupancy factors for these two positions lead to values close to 0.5 [0.470 for $\text{O}(W7)$ and 0.563 for $\text{O}(W8)$] and to normal thermal factors (see Table 1), showing this assumption to be correct.

Even though statistical distributions of cations have been previously observed by the author in some condensed phosphates: Na/Pb disorder in $\text{PbNa}_4(\text{P}_3\text{O}_9)_2$ (Averbuch-Pouchot & Durif, 1983), Hg/Na disorder in $(\text{NH}_4)_2\text{Na}_2\text{Hg}(\text{P}_3\text{O}_9)_2$ (Averbuch-Pouchot

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52174 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

& Durif, 1986), disorder in one component of a cation polyhedron has up to now never been observed in condensed-phosphate crystal chemistry.

Apart from the disorder occurring in O(W7)/O(W8), the hydrogen-bond scheme is as expected in this kind of compound. Its main geometrical features

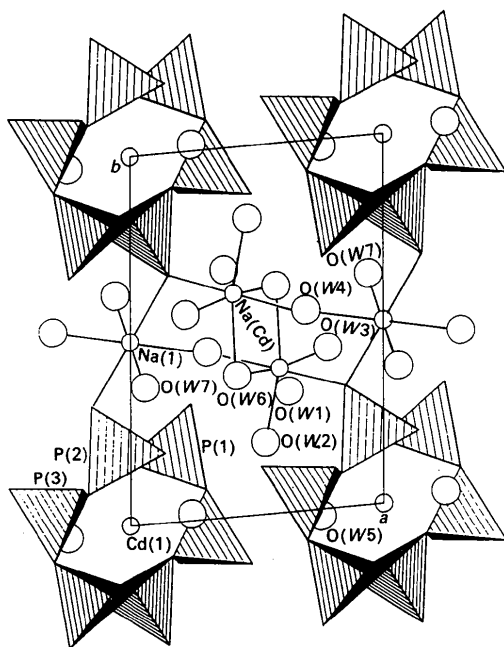


Fig. 1. Projection along the *c* axis of the atomic arrangement of $\text{Cd}_2\text{Na}_2\text{P}_6\text{O}_{18}\cdot 14\text{H}_2\text{O}$. The H atoms have been omitted and the disordered O(W7) and O(W8) water molecules are represented by a single circle.

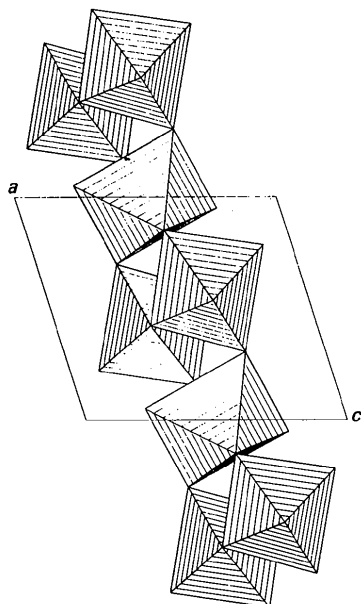


Fig. 2. Projection along the *b* axis of one chain of octahedra.

Table 2. Main interatomic distances (Å) and bond angles (°) in the atomic arrangement of $\text{Cd}_2\text{Na}_2\text{P}_6\text{O}_{18}\cdot 14\text{H}_2\text{O}$

E.s.d.'s are given in parentheses.

P_6O_{18} ring anion

P(1)O₄ tetrahedron

P(1)	O(E11)	O(E12)	O(L13)	O(L12)
O(E11)	1.484 (2)	2.571 (3)	2.474 (2)	2.546 (2)
O(E12)	119.4 (1)	1.494 (2)	2.548 (2)	2.465 (2)
O(L13)	105.9 (1)	110.1 (1)	1.613 (1)	2.508 (2)
O(L12)	111.5 (1)	105.9 (1)	102.9 (1)	1.595 (1)

P(2)O₄ tetrahedron

P(2)	O(L12)	O(E21)	O(E22)	O(L23)
O(L12)	1.606 (2)	2.519 (3)	2.546 (3)	2.398 (2)
O(E21)	108.8 (1)	1.491 (2)	2.551 (3)	2.538 (2)
O(E22)	111.0 (1)	118.2 (1)	1.483 (2)	2.529 (3)
O(L23)	96.7 (1)	110.1 (1)	110.0 (1)	1.604 (2)

P(3)O₄ tetrahedron

P(3)	O(L13)	O(L23)	O(E31)	O(E32)
O(L13)	1.596 (2)	2.444 (2)	2.553 (3)	2.489 (2)
O(L23)	99.8 (1)	1.599 (2)	2.518 (3)	2.535 (2)
O(E31)	111.1 (1)	108.7 (1)	1.499 (2)	2.540 (3)
O(E32)	108.0 (1)	110.8 (1)	117.1 (1)	1.479 (2)

P(2)—P(1)—P(3)	94.25 (2)	P(1)—P(2)	2.9065 (8)
P(1)—P(2)—P(3)	142.81 (3)	P(1)—P(3)	2.9370 (7)
P(1)—P(3)—P(2)	110.06 (2)	P(2)—P(3)	2.8960 (6)
P(1)—O(L13)—P(3)	132.4 (1)		
P(1)—O(L12)—P(2)	130.5 (1)		
P(2)—O(L23)—P(3)	129.4 (1)		

(Cd,Na)O₆ octahedron

(Na,Cd)—O(E21)	2.387 (2)	(Na,Cd)—O(W4)	2.382 (2)
(Na,Cd)—O(W2)	2.308 (2)	(Na,Cd)—O(W6)	2.358 (2)
(Na,Cd)—O(W3)	2.285 (2)	(Na,Cd)—O(W6)	2.436 (2)

Na(1)O₆ octahedron

Na(1)—O(E21)	2.491 (2) (× 2)	Na(1)—O(W7)	2.441 (4) (× 2)
Na(1)—O(W4)	2.489 (2) (× 2)	Na(1)—O(W8)	2.358 (3) (× 2)

Cd(1)O₆ octahedron

Cd(1)—O(E12)	2.271 (2) (× 2)	Cd(1)—O(E31)	2.249 (2) (× 2)
Cd(1)—O(W5)	2.338 (2) (× 2)		

The hydrogen-bond scheme

O(W)—H...O	O(W)—H	H...O	O(W)...O	O(W)—H...O
O(W1)—H(1W1)...O(E31)	0.80 (4)	2.00 (4)	2.795 (3)	174 (5)
O(W1)—H(2W1)...O(E11)	0.66 (4)	2.19 (4)	2.846 (2)	172 (4)
O(W2)—H(1W2)...O(W5)	0.86 (4)	2.10 (4)	2.955 (3)	169 (3)
O(W2)—H(2W2)...O(E22)	0.68 (4)	2.24 (4)	2.886 (3)	159 (5)
O(W3)—H(1W3)...O(E11)	0.84 (4)	1.99 (4)	2.821 (2)	173 (5)
O(W3)—H(2W3)...O(W1)	0.71 (5)	2.10 (5)	2.786 (4)	161 (4)
O(W4)—H(1W4)...O(W1)	0.74 (4)	2.04 (4)	2.775 (2)	171 (5)
O(W4)—H(2W4)...O(E11)	0.80 (5)	2.19 (5)	2.936 (3)	157 (3)
O(W5)—H(1W5)...O(E22)	0.87 (4)	1.94 (4)	2.803 (2)	176 (4)
O(W5)—H(2W5)...O(E32)	0.75 (5)	1.97 (5)	2.716 (3)	174 (4)
O(W6)—H(1W6)...O(E32)	0.89 (4)	1.90 (4)	2.765 (3)	164 (5)
O(W6)—H(2W6)...O(W7)	0.71 (5)	2.02 (4)	2.693 (4)	157 (6)
O(W6)—H(2W6)...O(W8)	0.71 (5)	1.93 (5)	2.640 (4)	177 (5)
O(W7)—H(W7)...O(W3)	0.89 (10)	2.46 (10)	3.075 (6)	127 (7)
O(W7)—H(W78)...O(E12)	0.92 (4)	2.00 (4)	2.845 (4)	151 (5)
O(W8)—H(W8)...O(E22)	0.78 (10)	2.08 (9)	2.823 (5)	159 (7)
O(W8)—H(W78)...O(E12)	0.92 (4)	2.00 (4)	2.804 (4)	144 (4)

H—O(W)—H angles

O(W1)	110 (5)	O(W5)	102 (4)
O(W2)	106 (5)	O(W6)	103 (4)
O(W3)	106 (5)	O(W7)	122 (6)
O(W4)	111 (5)	O(W8)	117 (6)

are reported in Table 2. For the disordered water molecule, one H atom [H(W78)] is common to the fractions of the molecule while two H atoms [H(W7) and H(W8)], refined with occupancy rates of 0.5, belong respectively to the O(W7) and O(W8) water-molecule fractions.

In addition it must be noticed that one of the water molecules [O(W1)] is not involved in cation coordination.

The drawings have been made using the *STRUPL*O program (Fischer, 1985).

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Room-Temperature Structure of Diaquafluorocopper(II)diiron(III)

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Abstract. $\text{CuFe}_2\text{F}_8(\text{H}_2\text{O})_2$, $M_r = 363.25$, monoclinic, $C2/c$, $a = 7.541$ (3), $b = 7.501$ (3), $c = 13.027$ (7) Å, $\beta = 90.52$ (4)°, $V = 736.8$ (15) Å³, $Z = 4$, $D_x = 3.28$ Mg m⁻³, $\text{Mo K}\alpha$, $\lambda = 0.71069$ Å, $\mu = 6.88$ mm⁻¹, $F(000) = 692$, $R = 0.036$ for 2321 unique observed reflections, hydrothermal growth. The room-temperature structure of $\text{CuFe}_2\text{F}_8(\text{H}_2\text{O})_2$ derives from that of $\text{Fe}_3\text{F}_8(\text{H}_2\text{O})_2$. It is described in terms of infinite $[\text{FeF}_{4/2}\text{F}_2]_n$ layers of corner-sharing octahedra connected by copper polyhedra or in terms of shifted $[\text{CuF}_{4/2}(\text{H}_2\text{O})_2, 2\text{FeF}_{6/2}]$ Kagomé layers. Copper octahedra are so strongly distorted {two short Cu—O distances [1.935 (2) Å], two short Cu—F distances [1.909 (1) Å] and two long Cu—F distances [2.451 (2) Å]} that the coordination of copper is better described by a $\text{CuF}_2(\text{H}_2\text{O})_2$ square plane, as shown by a bond-valence analysis.

Introduction. Several $M^{\text{II}}\text{Fe}_2\text{F}_8(\text{H}_2\text{O})_2$ phases with $M = \text{Fe}, \text{Mn}$ were recently grown by hydrothermal synthesis (Herdtschek, 1983; Leblanc, Férey, Calage & De Pape, 1984). Owing to the presence in the structure of triangles of corner-sharing octahedra, frustration of antiferromagnetic interactions occurs and peculiar magnetic properties can be observed (Guillot, Marchand, Leblanc & Férey, 1987), for example, 'idle spin' behaviour of Fe^{2+} was evidenced in $\text{Fe}_3\text{F}_8(\text{H}_2\text{O})_2$. We report here the structure of $\text{CuFe}_2\text{F}_8(\text{H}_2\text{O})_2$, in which the Jahn–Teller distortion of Cu^{2+} octahedra is expected to influence the super-exchange Fe—F—Cu angles and therefore the magnetic properties.

Experimental. Hydrothermal growth from CuF_2 and FeF_3 in 40% HF, 743 K, 185 MPa, 5M NaF solution, $(\text{NaF}/\text{CuF}_2/\text{FeF}_3) = (1/1/2)$. Prismatic light-green crystal $0.05 \times 0.04 \times 0.14$ mm. Data collected on a Siemens AED-2 four-circle diffractometer, ω – 2θ step scan mode in N steps of $\Delta\omega^\circ$, $37 \leq N \leq 50$, $0.023 \leq \Delta\omega \leq 0.027$. Time per step: 2 s. Profile-fitting data analysis (Clegg, 1981); isotropic linewidth $\omega = (0.85 + 0.05 \tan\theta)^\circ$. Aperture $D = 4.0$ mm. Lattice constants based on 24 reflections at $2\theta = 30^\circ$. Absorption correction by the Gauss method, $A_{\text{max}} = 0.76$, $A_{\text{min}} = 0.64$. Intensity measurement of one independent set to $2\theta_{\text{max}} = 90^\circ$ within the range $-14 \leq h \leq 14$, $0 \leq k \leq 14$, $0 \leq l \leq 25$. Standard reflections 400, 008, $0\bar{4}1$, intensity variation 1.0%, 3478 reflections measured, 2321 independent reflections used for refinements [$|F| > 6\sigma(|F|)$]. Structure solved from Patterson interpretation with *SHELXS86* program (Sheldrick, 1985). F magnitudes used in least-squares refinements: 70 parameters refined; mean $\Delta/\sigma = 0.007$, max. $\Delta/\sigma = 0.070$; secondary-extinction factor: $x = 1.6(2) \times 10^{-7}$; atomic scattering factors for H, O, F, Fe, Cu from *International Tables for X-ray Crystallography* (1974); calculations with *SHELX76* (Sheldrick, 1976) on a MicroVAX II computer. Patterson interpretation gives a solution with two metallic atoms. Then, refinements of atomic positions and isotropic thermal motion, followed by Fourier or difference Fourier maps, lead to the localization of all non-H atoms ($R = 0.075$). The residual decreases to $R = 0.037$ when refining the anisotropic thermal parameters. Positions of H atoms in two