Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	U_{eq}
Tl1	0.18250 (8)	1/2−x	1/2 + x	0.0313 (2)
Tl2	0.44820 (8)	1/2 + x	1−x	0.0349 (2)
Cd1	0.33219 (11)	x	x	0.0214 (2)
Cd2	0.58961 (11)	x	x	0.0203 (2)
S	0.2778 (4)	0.6242 (4)	0.5141 (4)	0.0169 (9)
O1	0.1839 (17)	0.718 (2)	0.462 (2)	0.048 (7)
O2	0.4042 (13)	0.6732 (19)	0.4999 (19)	0.040 (5)
O3	0.259 (2)	0.5121 (18)	0.439 (3)	0.051 (7)
O4	0.253 (2)	0.5981 (16)	0.6447 (13)	0.042 (6)

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

Tl1—O1^{i}	3.25 (2)	Cd1—O4^{iii}	2.27 (2)
Tl1—O3^{i}	3.27 (3)	Cd2—O1^{ii}	2.30 (2)
Tl1—O4	3.04 (2)	Cd2—O2	2.32 (2)
Tl2—O1^{ii}	3.01 (2)	S—O1	1.49 (2)
Tl2—O2	2.95 (2)	S—O2	1.42 (2)
Tl2—O3^{ii}	3.26 (2)	S—O3	1.42 (2)
Cd1—O3	2.31 (2)	S—O4	1.41 (2)
O1—S—O2	109.6 (11)	O2—S—O3	111.6 (12)
O1—S—O3	104.3 (13)	O2—S—O4	109.8 (12)
O1—S—O4	110.9 (12)	O3—S—O4	110.5 (12)

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

Table 3. *Bond-valence sums (v.u.) for the atoms in $Tl_2Cd_2(SO_4)_3$ and $K_2Cd_2(SO_4)_3$*

	$Tl_2Cd_2(SO_4)_3$	$K_2Cd_2(SO_4)_3$†
Tl1	0.61 (3)	0.83 (3) (K1)
Tl2	0.83 (4)	0.88 (3) (K2)
Cd1	2.13 (10)	2.42 (8)
Cd2	2.03 (9)	2.30 (7)
S	6.7 (3)	6.49 (6)
O1	1.96 (9)	2.20 (3)
O2	2.18 (9)	2.17 (3)
O3	2.17 (11)	2.17 (3)
O4	2.25 (9)	2.12 (4)

† 440 K (Percival *et al.*, 1989)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAMEL JOCKEY* (Flack, 1974). Program(s) used to refine structure: *CRYLSQ* in *Xtal*3.2 (Hall, Flack & Stewart, 1992). Molecular graphics: *ORTEP* (Johnson, 1965) in *Xtal*3.2. Software used to prepare material for publication: *BONDLA* in *Xtal*3.2.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: AB1353). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Gadolinium Potassium Hexacyanoferrate(II) Trihydrate

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Abstract

Gadolinium potassium hexacyanoferrate(II) trihydrate, $GdKFe(CN)_6.3H_2O$, crystallizes in the orthorhombic space group *Pnma* (No. 62). The structure consists of non-linear cyanide bridges linking octahedrally coordinated Fe atoms and eight-coordinate Gd atoms. Cavities within the structure are occupied by potassium and zeolitic water molecules which are within hydrogen-bonding distance of the water molecules bonded to the Gd atoms, $GdN_6(H_2O)_2$.

Comment

The first ferrocyanide containing a lanthanide atom, $CeKFe(CN)_6$, was described by Robinson (1909).

Prandtl & Mohr (1938) later reported the preparation of lanthanide (Ln) ferrocyanides containing sodium and potassium and having a number of water molecules associated with the alkali–lanthanide ferrocyanide compounds. Recently, the structural characteristics of the larger rare earth compounds of this series (Ln = La–Nd) have been described (Beall, Mullica & Milligan, 1978; Mullica, Milligan & Oliver, 1979; Mullica, Sappenfield & Perkins, 1988; Milligan, Mullica & Perkins, 1982). Their structure is virtually identical to the $LnFe(CN)_6.5H_2O$ (Ln = La–Nd) series, with the exception that a potassium ion replaces one of the zeolitic water molecules. The heavier lanthanide potassium complexes have been shown to have an orthorhombic C-centered form (Mullica, Sappenfield & Cunningham, 1991), with fewer zeolitic water molecules. Monoclinic $SmKFe(CN)_6$ (Mullica, Sappenfield & Perkins, 1989) is a transitional form between the hexagonal and orthorhombic lanthanide potassium hexacyanide compounds. Structural analysis of these compounds has shown that the potassium ion partially substitutes for the zeolitic water molecule. In effect, the potassium ion occupies a crystallographic site which is almost the same as the zeolitic water molecule. As a result, the occupancy factors for the zeolitic water molecule and the potassium ion are set at 0.5. The $LnKRu(CN)_6$ series also display a phase transition from the hexagonal form (Mullica, Hayward & Sappenfield, 1996*a*) to the orthorhombic C-centered structure (Mullica, Hayward & Sappenfield, 1996*b*). In the orthorhombic lanthanide potassium hexacyanoruthenates, however, the potassium ion and zeolitic water molecules occupy specific crystallographic sites with no disorder. The structural investigation of gadolinium potassium hexacyanoferrate was initiated in order to determine whether the K^+ ion and zeolitic H_2O occupy separate crystallographic sites as in the gadolinium and terbium potassium hexacyanoruthenates (Mullica, Hayward & Sappenfield, 1996*b*).

Unlike the lanthanide iron hexacyanides and lanthanide potassium ruthenium hexacyanides, where the early members of the series crystallize in the hexagonal space group $P6_3/m$ and the later members of the series crystallize in the C-centered orthorhombic space group *Cmcm*, the lanthanide potassium iron hexacyanides appear to undergo a structural transition from the hexagonal structure to the C-centered lattice structure *via* a monoclinic and a primitive orthorhombic form. Gadolinium hexacyanoferrate(III) and gadolinium hexacyanoruthenate(III) crystallize in the orthorhombic C-centered space group *Cmcm* (Mullica & Sappenfield, 1991; Mullica, Hayward & Sappenfield, 1996*b*). On the other hand, gadolinium potassium hexacyanoferrate(II) trihydrate crystallizes in the primitive space group *Pnma* (No. 62). To the best of our knowledge, this is the first observed lanthanide hexacyanide to crystallize in a primitive orthorhombic space group.

A perspective view of the $GdKFe(CN)_6$ structure is shown in Fig. 1. The Gd atom is eight-coordinated by six cyano N and two water O atoms arranged in a square antiprism. The Fe atom is octahedrally bonded to six cyano C atoms. The octahedral Fe and eight-coordinate Gd atoms are linked by non-linear cyanide bridges [the mean Gd—N—C angle is 162 and the mean Fe—C—N angle is 178°], which produce a serpentine-like undulation throughout the crystal lattice. Two mirror-related N1 atoms (N1 and $N1^{v}$) and two mirror-related N2 atoms ($N2^{i}$ and $N2^{vi}$) form one of the square faces of the square antiprism. The opposing square face is formed by two mirror-related N3 atoms ($N3^{ii}$ and $N3^{vii}$) and the O1 and O2 water atoms (mean deviation from the plane is 0.166 Å) (Fig. 1). The dihedral angle between the two square faces of the square antiprism is 1.8°. The zeolitic water molecule and potassium ions occupy cavities within the polymeric structure. The H_2O molecules are within hydrogen-bonding distance [O1···O3 2.91 (1) and O2···O3 2.75 (1) Å] of the coordinated water molecules, O1 and O2. The potassium ions partially substitute for the zeolitic water molecules. Each cavity contains either one K^+ ion or one H_2O molecule, but the ordering of these species is statistical.

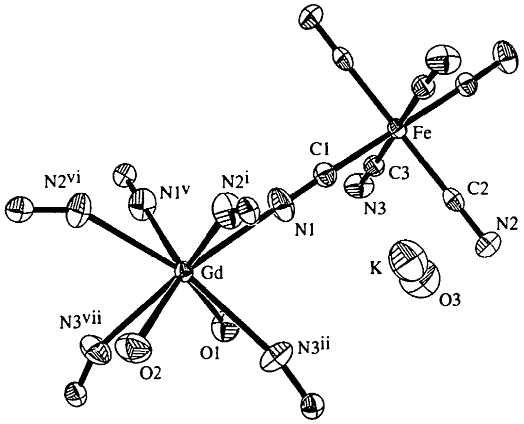

Fig. 1. A perspective view of $GdKFe(CN)_6.3H_2O$ displaying the square-antiprism arrangement about the Gd atom and the octahedral geometry around the Fe atom. N atoms bonded to the Gd atom have been labelled for clarity. The symmetry codes correspond to the codes listed in Table 2. Displacement ellipsoids are shown at 50% probability levels.

Experimental

Clear colorless crystals of gadolinium hexacyanoferrate(II) trihydrate were synthesized by employing the slow diffusion of dilute aqueous solutions of $GdCl_3$ and $K_4Fe(CN)_6$ in a U-tube. The U-tubes were stored in the dark for six months before crystals large enough for crystallographic analysis were harvested. Both starting materials were purchased commercially (reagent grade) and used without further purification.

Crystal data

$GdKFe(CN)_6.3H_2O$
$M_r = 462.35$
Orthorhombic
Pnma
$a = 12.632\,(3)$ Å
$b = 13.618\,(2)$ Å
$c = 7.2249\,(7)$ Å
$V = 1242.9\,(3)$ Å^3
$Z = 4$
$D_x = 2.47$ Mg m^{-3}
$D_m = 2.41\,(5)$ Mg m^{-3}
D_m measured by flotation in bromoform and bromobenzene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 16.24$–$23.96°$
$\mu = 6.829$ mm^{-1}
$T = 292$ K
Clear rod
$0.41 \times 0.13 \times 0.11$ mm
Colorless

Data collection

Enraf–Nonius CAD-4*F* diffractometer
ω–2θ scans
Absorption correction: integration from crystal shape
$T_{\min} = 0.418$, $T_{\max} = 0.477$
1507 measured reflections
1141 independent reflections

1022 observed reflections $[F > 4\sigma(F)]$
$R_{\text{int}} = 0.0218$
$\theta_{\max} = 25°$
$h = -4 \rightarrow 14$
$k = -4 \rightarrow 16$
$l = -2 \rightarrow 8$
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

Refinement on F
$R = 0.023$
$wR = 0.043$
$S = 1.165$
1022 reflections
98 parameters
H atoms were not located
$w = 1/[\sigma^2(F) + 0.0007F^2]$
$(\Delta/\sigma)_{\max} = 0.366$
$\Delta\rho_{\max} = 0.920$ e Å^{-3}
$\Delta\rho_{\min} = -1.101$ e Å^{-3}

Extinction correction: *SHELXTL-Plus* (Sheldrick, 1989)
Extinction coefficient: 0.00044 (8)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

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

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

	x	y	z	U_{eq}
Gd	0.15890 (2)	1/4	−0.00675 (3)	0.0142 (2)
Fe	1/2	1/2	0	0.0163 (3)
C1	0.3807 (4)	0.4123 (4)	−0.0101 (4)	0.023 (1)
N1	0.3088 (4)	0.3602 (4)	−0.0097 (4)	0.035 (1)
C2	0.4177 (3)	0.5871 (3)	0.1451 (5)	0.023 (1)
N2	0.3654 (3)	0.6407 (3)	0.2313 (4)	0.033 (1)
C3	0.5485 (3)	0.4328 (3)	0.2140 (5)	0.022 (1)
N3	0.5782 (3)	0.3884 (3)	0.3427 (4)	0.035 (1)
O1	0.2244 (3)	1/4	0.3257 (5)	0.035 (1)
O2	−0.0409 (3)	1/4	−0.0920 (6)	0.038 (1)
O3†	0.1665 (6)	0.590 (1)	0.0108 (9)	0.056 (4)
K†	0.1677 (2)	0.5791 (2)	−0.0451 (4)	0.053 (1)

† Site occupancy = 0.5.

Table 2. *Geometric parameters (Å, °)*

Gd—N1	2.417 (5)	Fe—C3	1.898 (3)
Gd—N2^{i}	2.427 (3)	C1—N1	1.152 (7)
Gd—N3^{ii}	2.449 (3)	C2—N2	1.165 (5)
Gd—O1	2.541 (4)	C3—N3	1.171 (5)
Gd—O2	2.598 (4)	O1—O3^{iii}	2.91 (1)
Fe—C1	1.924 (5)	O2—O3^{iv}	2.76 (1)
Fe—C2	1.894 (4)		
N1—Gd—N1^{v}	76.8 (3)	C1—Fe—C2^{viii}	91.2 (2)
N1—Gd—N2^{i}	73.2 (1)	C1—Fe—C3	89.1 (2)
N1—Gd—N2^{vi}	118.2 (1)	C1—Fe—C3^{viii}	90.9 (2)
N1—Gd—N3^{ii}	81.5 (1)	C2—Fe—C3^{viii}	88.4 (1)
N1—Gd—N3^{vii}	144.0 (1)	C2—Fe—C3	91.6 (1)
N1—Gd—O1	75.6 (1)	Fe—C1—N1	177.6 (3)
N1—Gd—O2	139.6 (1)	Fe—C2—N2	178.5 (3)
N2^{i}—Gd—N2^{vi}	75.6 (2)	Fe—C3—N3	177.7 (3)
N2^{i}—Gd—N3^{ii}	81.5 (1)	Gd—N1—C1	179.4 (4)
N2^{i}—Gd—N3^{vii}	142.8 (1)	Gd^{iii}—N2—C2	150.8 (3)
N2^{i}—Gd—O1	141.1 (1)	Gd^{ix}—N3—C3	156.2 (3)
N2^{i}—Gd—O2	72.1 (1)	Gd—O1—O3^{iii}	126.1 (2)
N3^{ii}—Gd—N3^{vii}	100.7 (2)	Gd—O2—O3^{iv}	120.5 (2)
N3^{ii}—Gd—O1	71.2 (1)	O3^{iii}—O1—O3^{x}	97.3 (4)
N3^{ii}—Gd—O2	73.2 (1)	O3^{iv}—O2—O3^{xi}	104.4 (5)
O1—Gd—O2	122.7 (1)	O1^{i}—O3—O2^{iv}	77.1 (4)
C1—Fe—C2	88.8 (2)		

Symmetry codes: (i) $\frac{1}{2} - x, 1 - y, z - \frac{1}{2}$; (ii) $x - \frac{1}{2}, y, \frac{1}{2} - z$; (iii) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$; (iv) $-x, 1 - y, -z$; (v) $x, \frac{1}{2} - y, z$; (vi) $\frac{1}{2} - x, y - \frac{1}{2}, z - \frac{1}{2}$; (vii) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} - z$; (viii) $1 - x, 1 - y, -z$; (ix) $\frac{1}{2} + x, y, \frac{1}{2} - z$; (x) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} + z$; (xi) $-x, y - \frac{1}{2}, -z$.

A conoscopic examination between two crossed polarizers on a Zeiss Photomicroscope II confirmed the optical quality and nature of the crystal. The structure was checked for additional symmetry by the *MISSYM* program (Gabe, Le Page, Charland, Lee & White, 1989).

Data collection: *CAD*-4 *Manual* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *Manual*. Data reduction: *SDP* (Enraf–Nonius, 1985). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989) (heavy-atom Patterson method). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus* and *XP* (Siemens, 1994). Software used to prepare material for publication: local *CIFGEN* program.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1153). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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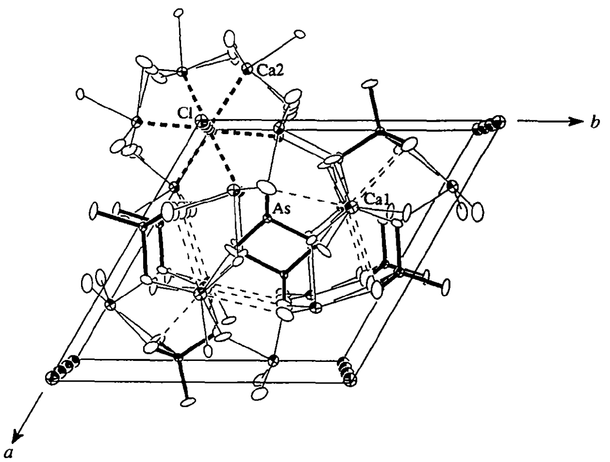

Fig. 1. Projection onto the (001) plane of the structure of $Ca_5(AsO_4)_3Cl$ showing the connectivity of the $Ca1O_9$, $Ca2O_5Cl$ and AsO_4 coordination geometries. Open circles represent O atoms. The Ca2—Cl bonds in $Ca2O_5Cl$ and the long Ca1—O bonds of the tricapped trigonal prism (viewed down its vertical axis) are drawn in thick and thin dotted lines, respectively. The atoms which were refined anisotropically are shown with ellipsoids at the 90% probability level.

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Chlorapatite: $Ca_5(AsO_4)_3Cl$

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Abstract

Chlorapatite (pentacalcium triarsenate chloride), $Ca_5(AsO_4)_3Cl$ crystallizes in the hexagonal system and is closely related to its phosphate analogs, $Ca_5(PO_4)_3X$ (X = Cl, OH), but has a slightly modified structure in terms of anion sites.

Comment

The structure and chemistry of the apatites have been widely studied for many years due to their versatility in applications (Prener, 1967; Kreidler & Hummel, 1970; Elliott, 1994). The apatite structure [general formula $A_5(BO_4)_3X$, where A is a 2+ cation, B = P, As, V, Si or S, and X = F, Cl, Br, OH or CO_3] is adopted by a wide range of compounds.

The title compound adopts virtually the same structural framework as both $Ca_5(PO_4)_3Cl$ (Mackie, Elliott & Young, 1972) and $Ca_5(PO_4)_3OH$ (Kay, Young & Posner, 1964), with a slight variation due to the ordering of anions. The Cl atoms, which are disordered and have half occupancy, are located at (0,0,0.1263) and are symmetrical with respect to the mirror planes at $z = \frac{1}{4}$ and $z = \frac{3}{4}$. No single-crystal study of $Ca_5(AsO_4)_3Cl$ has been reported previously.

Experimental

Crystals of the title compound were grown *via* an eutectic flux of 31% $CaCl_2$ and 69% NaCl (m.p. 773 K) from a reaction mixture of As_2O_5, CuO and Cu_2O (nominal composition $As_2Cu_4O_8$) in a fused-silica ampoule. The reactants were heated at 973 K for 6 d before being slowly cooled to room temperature. Apatite crystals were isolated by washing the reaction product with deionized water using suction filtration.

Crystal data

$Ca_5(AsO_4)_3Cl$
$M_r = 652.61$
Hexagonal
$P6_3/m$
$a = 10.076\,(1)$ Å
$c = 6.807\,(1)$ Å
$V = 598.4\,(2)$ Å^3
$Z = 2$
$D_x = 3.621$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$–$24°$
$\mu = 10.753$ mm^{-1}
$T = 296\,(2)$ K
Chunk
0.3 × 0.2 × 0.2 mm
Colorless

Data collection

Rigaku AFC-5*S* four-circle diffractometer
ω scans
1142 measured reflections
496 independent reflections
$R_{\rm int} = 0.0193$
$\theta_{\rm max} = 27.49°$

$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -8 \rightarrow 8$
3 standard reflections monitored every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0207$
$wR(F^2) = 0.0503$

Extinction correction: *SHELXL*93 (Sheldrick, 1993)

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