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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{N}-\text{C}) = 0.009 \text{ \AA}$
R factor = 0.033
wR factor = 0.095
Data-to-parameter ratio = 20.5

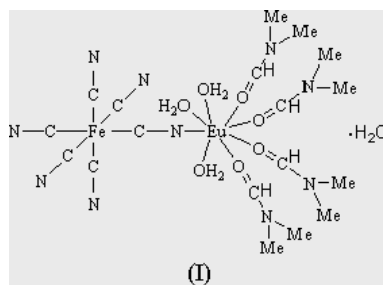
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Pentacyanoferrate(III)- μ -cyano-triaquatetrakis-(dimethylformamide)europium(III) hydrate

The structure of the title compound, $[\text{EuFe}(\text{CN})_6(\text{DMF})_4(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}$ (DMF is dimethylformamide), is comprised of a neutral bimetallic complex $\text{EuFe}(\text{CN})_6(\text{DMF})_4(\text{H}_2\text{O})_3$ and a solvent water molecule. Approximately octahedral sixfold-coordinated Fe^{III} and a slightly distorted square-antiprismatic eightfold-coordinated Eu^{III} ions are bridged by a cyanide group to form a dinuclear complex. A three-dimensional framework is formed through $\text{O}-\text{H} \cdots \text{N}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen-bonding interactions.

Comment

Kou *et al.* (1998) first obtained a dinuclear cyano-bridged Ln-Fe complex, $[\text{SmFe}(\text{CN})_6(\text{DMF})_4(\text{H}_2\text{O})_4] \cdot \text{H}_2\text{O}$ (DMF is dimethylformamide), using $\text{Sm}(\text{NO}_3)_3$ as starting material. Later, in 2000, the group of Kautz reported ferricyanide bimetallic complexes of lighter and heavier rare-earth ions $[\text{LnFe}(\text{CN})_6(\text{DMF})_4(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}$ (Ln = La, Ce, Er, Yb and Lu; Kautz *et al.*, 2000; Mullica *et al.*, 2000). It is interesting that various amounts of coordinated water molecules have been found in these complexes. When Ln = Sm there are four coordinated water molecules in the complex; however, when Ln = La, Ce, Er, Yb and Lu, three coordinated water molecules are found. In order to further investigate the influence of lanthanide contraction on the composition and structure of such complexes, we synthesized a dinuclear Fe-Eu complex, *viz.* $[\text{EuFe}(\text{CN})_6(\text{DMF})_4(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}$, (I), the crystal structure of which is reported here.



As shown in Fig. 1, the structure of (I) is composed of neutral bimetallic $\text{EuFe}(\text{CN})_6(\text{DMF})_4(\text{H}_2\text{O})_3$ complex molecules and solvent water molecules. The Fe^{III} and Eu^{III} ions are bridged by a cyanide group to form a dinuclear complex. The Eu^{III} ion is eightfold coordinated by one N atom of the bridging cyanide group [$\text{Eu}-\text{N} = 2.495(4) \text{ \AA}$], four O atoms of DMF molecules [$\text{Eu}-\text{O}_{\text{DMF}} = 2.353(3)-2.649(3) \text{ \AA}$, with an average distance of $2.469(3) \text{ \AA}$], and three water molecules, for which the three $\text{Eu}-\text{O}_{\text{water}}$ distances are in the range $2.204(3)-2.589(3) \text{ \AA}$, with an average distance of

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2.362 (3) Å. The coordination polyhedron can be described as a slightly distorted square antiprism. A similar situation was found in [Ln(Fe(CN)₆(DMF)₄(H₂O)₃)]·H₂O (Mullica *et al.*, 2000). The Eu1–N1–C1 angle is 165.7 (4)°, deviating slightly from linearity, as was the case in its analog. A three-dimensional framework is formed through O–H···O and O–H···N hydrogen-bonding interactions, with O···O distances of 2.748 (3) Å and average O···N separations of 2.706 (3) Å.

Experimental

The title complex was prepared by mixing a 1:1 molar ratio of EuCl₃·H₂O (0.5 mmol) and K₃[Fe(CN)₆] (165 mg, 0.5 mmol) in a mixed solution of DMF/H₂O/EtOH (*v:v* = 1:2:2). The reaction mixture was filtered and orange single crystals suitable for X-ray analysis were obtained by slow evaporation of the solvent. Yield = 81%. IR spectra [recorded on an FT-IR 170SX (Nicolet) spectrometer, KBr pellet]: 3614, 3380 (broad band center), 2945, 2125, 1653, 1497, 1381, 1115, 675 cm⁻¹.

Crystal data

[EuFe(CN)₆(C₃H₇NO)₄·(H₂O)₃]]·H₂O

M_r = 728.38

Monoclinic, *P*_{2₁}/*n*

a = 17.633 (4) Å

b = 8.8791 (18) Å

c = 19.831 (4) Å

β = 96.12 (3)°

V = 3087.2 (11) Å³

Z = 4

D_x = 1.567 Mg m⁻³

Mo *K*α radiation

Cell parameters from 25 reflections

θ = 30.1–33.9°

μ = 2.54 mm⁻¹

T = 293 (2) K

Prism, orange

0.80 × 0.40 × 0.40 mm

Data collection

Rigaku AFC-7R diffractometer

2θ–ω scans

Absorption correction: ψ scan
(Molecular Structure Corporation & Rigaku, 2000)

T_{min} = 0.271, *T_{max}* = 0.363

7448 measured reflections

7067 independent reflections

5832 reflections with *I* > 2σ(*I*)

R_{int} = 0.050

θ_{max} = 31.0°

h = –7 → 25

k = –11 → 3

l = –22 → 22

3 standard reflections

every 200 reflections

intensity decay: –1.3%

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.033

wR (*F*²) = 0.095

S = 1.01

7067 reflections

344 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 3.1152P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.003

Δρ_{max} = 1.64 e Å⁻³

Δρ_{min} = –1.42 e Å⁻³

Extinction correction: *SHELXL97*

Extinction coefficient: 0.00100 (17)

Table 1

Selected geometric parameters (Å, °).

Eu1–O3W	2.204 (3)	Eu1–N1	2.495 (4)
Eu1–O1W	2.293 (3)	Eu1–O2W	2.589 (3)
Eu1–O11	2.353 (3)	Eu1–O21	2.649 (3)
Eu1–O41	2.383 (3)	N1–C1	1.142 (5)
Eu1–O31	2.489 (3)		
C1–N1–Eu1	165.7 (4)	N1–C1–Fe1	176.7 (3)

