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Aqua(hexacyanoferrato-*N*)bis(μ-glycine)-glycinelanthanum(III) monohydrate

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The title compound, $[La\{Fe(CN)_6\}(C_2H_5NO_2)_3(H_2O)]_2\cdot 2H_2O$, consists of $Fe(CN)_6$ octahedra connected to an eight-coordinate lanthanum *via* two cyanide bridging La—N—C—Fe links. The La^{3+} ions are bridged by four glycine molecules, forming a binuclear complex: La—(O—gly—O)$_4$—La. The three-dimensional framework is completed through hydrogen-bonding interactions.

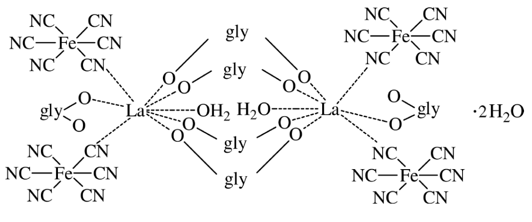
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.011 Å
R factor = 0.056
wR factor = 0.154
Data-to-parameter ratio = 12.7

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

Comment

In a previous paper, we reported the crystal structure of aqua(hexacyanoferrato-*N*)bis(μ-glycine)glycinecerium(III) monohydrate (García-Granda *et al.*, 1996). As a continuation of our studies of the metal–ligand bond interactions in this type of complex, we have determined the crystal structure of the isotypic lanthanum analogue. The La^{3+} ion is eight-coordinated (see *Scheme* below). Only two cyano groups are coordinated to the La atom (La—N1 and La—N2). The rest of the coordination polyhedron is formed by six O atoms, one from a water molecule (O7) and five from glycine moieties. The La^{3+} ions are bridged by four glycine molecules, forming a binuclear complex: La—(O—gly—O)$_4$—La. The low-spin Fe^{3+} ion is octahedrally coordinated to six cyano groups. Bond distances and angles are close to the expected values (García-Granda *et al.*, 1996). A view of the asymmetric unit is shown in Fig. 1.



(I)

Experimental

Crystals suitable for X-ray analysis were obtained by slow evaporation of an aqueous equimolecular solution of lanthanum trichloride, potassium ferrocyanide and a fivefold excess of glycine.

Crystal data

$[LaFe(CN)_6(C_2H_5NO_2)_3(H_2O)]_2\cdot 2H_2O$
M_r = 1224.24
Triclinic, $P\bar{1}$
a = 10.029 (2) Å
b = 10.198 (2) Å
c = 12.233 (2) Å
α = 74.96 (2)°
β = 88.12 (2)°
γ = 61.38 (2)°
V = 1054.5 (3) Å^3
Z = 1
D_x = 1.928 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 25 reflections
θ = 10–30°
μ = 2.75 mm^{-1}
T = 293 (2) K
Prism, red
0.3 × 0.2 × 0.2 mm

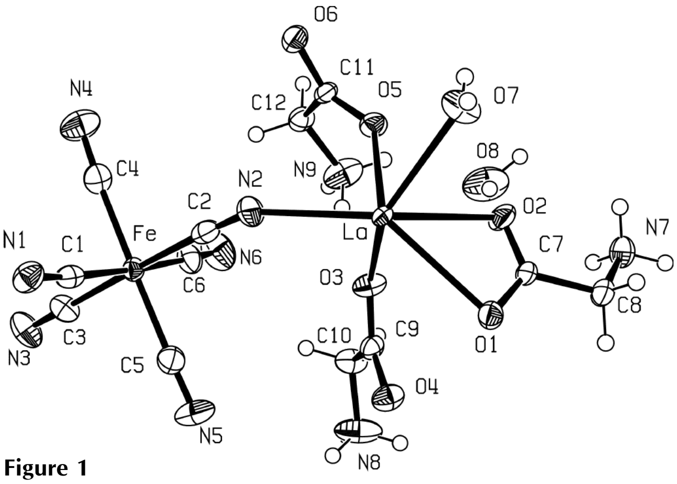


Figure 1
View of the asymmetric unit of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

Data collection

Siemens *P*3/*PC* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.851$, $T_{\max} = 0.890$
7294 measured reflections
3656 independent reflections
3270 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.058$

$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$
3 standard reflections every 200 reflections frequency: 60 min intensity decay: 4.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.155$
$S = 1.06$
3656 reflections
287 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1095P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 2.58$ e Å^{-3}
$\Delta\rho_{\min} = -2.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

La—O1	2.705 (7)	O1—C7	1.265 (9)
La—O2	2.602 (7)	O2—C7	1.237 (10)
La—O3	2.547 (5)	O3—C9	1.258 (10)
La—O5	2.450 (5)	O4—C9	1.237 (9)
La—O7	2.545 (6)	O5—C11	1.253 (10)
La—N2	2.641 (8)	O6—C11	1.267 (11)
La—O1^{i}	2.502 (5)	N1—C1	1.148 (11)
La—O4^{i}	2.532 (5)	N2—C2	1.143 (12)
La—N1^{ii}	2.637 (8)	N3—C3	1.131 (12)
Fe—C1	1.941 (8)	N4—C4	1.133 (14)
Fe—C2	1.939 (9)	N5—C5	1.137 (14)
Fe—C3	1.942 (9)	N6—C6	1.138 (13)
Fe—C4	1.954 (10)	N7—C8	1.489 (13)
Fe—C5	1.944 (10)	N8—C10	1.461 (13)
Fe—C6	1.950 (9)	N9—C12	1.474 (13)
O2—La—O7	71.1 (2)	C1—Fe—C2	89.9 (3)
O3—La—O5	77.43 (18)	C1—Fe—C3	88.7 (3)
O5—La—O7	72.91 (19)	C1—Fe—C4	87.6 (4)
O5—La—N2	71.4 (2)	C1—Fe—C5	92.3 (4)
O1^{i}—La—O5	142.54 (18)	C1—Fe—C6	177.1 (4)
O4^{i}—La—O5	143.60 (18)	La^{ii}—N1—C1	148.9 (7)
O5—La—N1^{ii}	92.1 (2)	La—N2—C2	154.3 (7)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N7—H7*A*$\cdots$O6^{i}	0.89	1.91	2.771 (11)	162
N7—H7*B*$\cdots$N5^{ii}	0.89	2.02	2.890 (13)	164
N7—H7*C*$\cdots$N4^{i}	0.89	2.19	2.984 (12)	148
O7—H7*D*$\cdots$O6^{iii}	0.85	1.92	2.744 (8)	164
O7—H7*E*$\cdots$N1^{ii}	0.85	2.24	3.070 (10)	165
N8—H8*C*$\cdots$N3^{iv}	0.89	2.54	3.045 (11)	117
N8—H8*C*$\cdots$N6^{iv}	0.89	2.55	3.146 (13)	125
N8—H8*D*$\cdots$O6^{v}	0.89	2.16	2.844 (10)	132
O8—H8*F*$\cdots$O2	0.90 (16)	2.14 (15)	3.027 (10)	170 (14)
N9—H9*A*$\cdots$O8^{vi}	0.89	2.34	3.046 (12)	136
N9—H9*A*$\cdots$N4^{vii}	0.89	2.60	3.155 (11)	121
N9—H9*B*$\cdots$O3	0.89	2.16	3.022 (9)	163
N9—H9*C*$\cdots$O8	0.89	2.14	2.921 (14)	147

Symmetry codes: (i) $x-1, y, z$; (ii) $x, y-1, z$; (iii) $1-x, -y, 1-z$; (iv) $-x, 2-y, -z$; (v) $x-1, 1+y, z$; (vi) $-x, 1-y, -z$; (vii) $1-x, 1-y, -z$.

The structure was solved using the atomic coordinates of the isotypic cerium analogue (García-Granda *et al.*, 1996). All H atoms were calculated geometrically (except those of the water molecules, initially located from difference Fourier maps) and included in the refinement, but constrained to ride on their parent atom. The H atoms of the O7 water molecule were poorly determined and refinement gave unacceptable geometry; accordingly, their positions were calculated using the program *HYDROGEN* (Nardelli, 1999) and they were treated like the other calculated H atoms. The isotropic displacement parameters of the H atoms were set to $1.2U_{\text{eq}}$ of their parent atoms.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *PLATON* (Spek, 1990), *PARST* (Nardelli, 1995) and *PARSTCIF* (Nardelli, 1991).

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