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Structures of $LuFeCoO_4$ and $LuFe_2O_4$

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Abstract. Common to both: trigonal, $R\bar{3}m$, $Z = 3$, $T = 295$ K, Mo $K\alpha$, $\lambda = 0.71073$ Å. Cobalt iron lutetium tetraoxide, $LuFeCoO_4$: $M_r = 353.74$, $a = 3.4180$ (1), $c = 25.28$ (1) Å, $V = 255.8$ (1) Å^3, $D_x = 6.89$ Mg m^{-3}, $\mu = 37.64$ mm^{-1}, $F(000) = 468$, $R = 0.016$, 497 unique observed reflections. Diiron lutetium tetraoxide, $LuFe_2O_4$: $M_r = 350.66$, $a = 3.4406$ (1), $c = 25.28$ (1) Å, $V = 259.2$ (1) Å^3, $D_x = 6.74$ Mg m^{-3}, $\mu = 36.56$ mm^{-1}, $F(000) = 465$, $R = 0.029$, 480 unique observed reflections. These structures are of the In_2ZnS_4 type and described as a close packing of O atoms with Lu atoms in octahedral and other metal atoms in trigonal bipyramidal coordination. The thermal parameters U_{33} of Lu atoms are abnormally large.

Experimental. The crystals were prepared from large black crystals grown by Iida, Takekawa & Kimizuka (1990). Intensity data were collected on an Enraf–Nonius CAD-4 single-crystal diffractometer with graphite-monochromated Mo $K\alpha$ radiation in ω–2θ scan mode. Experimental conditions are summarized in Table 1. The intensities were corrected for Lorentz, polarization and absorption factors. The structures were refined on F by least squares by assuming isotropic secondary extinction. The calculation was initiated with the atomic parameters of $YbFe_2O_4$ given by Kato, Kawada, Kimizuka & Katsura (1975). Unit weight was given to all the observed reflections. The atomic scattering factors for neutral atoms and dispersion correction factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 2.* Selected bond distances and angles are listed in Table 3 together with their estimated standard deviations. All of the calculations were performed with program system *SDP* (B. A. Frenz & Associates, Inc., 1985).

Related literature. The structures of the title compounds are essentially of the In_2ZnS_4 type (Lappe, Niggli, Nitsche & White, 1962). They are isostructural with $YbFe_2O_4$ (Kato *et al.*, 1975) and $Yb_{0.5}Eu_{0.5}Fe_2O_4$ (Malaman, Evrard, Tannieres, Aubry, Courtois & Protas, 1975). Kimizuka, Mohri, Matsui & Shiratori (1988) and Kimizuka & Mohri (1989) identified several new compounds of $RAO_3(MO)_n$ (R = Sc, Y, In, Er, Tm, Yb, Lu; A = Al, Fe, Ga; M = Mg, Mn, Fe, Co, Zn, Cd), and estimated the crystal structures through both X-ray powder diffraction and electron diffraction analysis.

Table 1. *Experimental data*

	$LuFeCoO_4$	$LuFe_2O_4$
Crystal shape and	Sphere	Sphere
diameter (mm)	0.120	0.120
Number and θ (°) range for	18	18
lattice parameters	41–46	41–46
Transmission factors	0.054–0.132	0.059–0.137
θ_{max} (°)	60	60
Range of h ($=k$)	0–7	0–7
l	0–60	0–60
Number and variation (%) of	3	3
standard reflections	−0.3	−0.6
Measured reflections	555	564
Observed reflections [$I > 1.5\sigma(I)$]	497	480
R	0.016	0.029
wR	0.019	0.031
S	1.3	2.0
$(\Delta/\sigma)_{max}$	0.01	0.01
Min. and max. $\Delta\rho$ (e Å^{-3})	−1.8, 2.8	−8.6, 8.1
Extinction factor ($\times 10^6$)	8.3 (1)	9.6 (1)

Table 2. *Atomic parameters and thermal parameters* (Å^2)

$x = y = 0$, $U_{11} = U_{22} = 2U_{12}$, $U_{13} = U_{23} = 0$, $B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

		$LuFeCoO_4$	$LuFe_2O_4$
Lu	z	0	0
	U_{11}	0.0037 (1)	0.0041 (1)
	U_{33}	0.0235 (1)	0.0432 (2)
	B_{eq}	0.81 (1)	1.35 (1)
Fe/Co	z	0.21485 (2)	0.21518 (3)
	U_{11}	0.0072 (1)	0.0100 (1)
	U_{33}	0.0074 (1)	0.0090 (2)
	B_{eq}	0.57 (1)	0.76 (1)
O(1)	z	0.1284 (1)	0.1281 (3)
	U_{11}	0.012 (1)	0.019 (1)
	U_{33}	0.017 (1)	0.047 (4)
	B_{eq}	1.1 (1)	2.3 (1)
O(2)	z	0.2923 (1)	0.2926 (2)
	U_{11}	0.009 (1)	0.013 (1)
	U_{33}	0.008 (1)	0.008 (1)
	B_{eq}	0.7 (1)	0.9 (1)

* A list of structure factors has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53010 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 3. *Bond lengths* (Å) *and angles* (°)

	$LuFeCoO_4$	$LuFe_2O_4$
Lu—O(2^i) (6×)	2·230 (1)	2·237 (2)
Lu—O(1) (2×)	3·247 (3)	3·240 (8)
O(2^i)—O(2^{ii}) (6×)	3·418 (1)	3·441 (1)
O(2^i)—O(2^{iii}) (6×)	2·865 (3)	2·861 (4)
O(2^i)—Lu—O(2^{ii}) (6×)	100·1 (1)	100·5 (1)
O(2^i)—Lu—O(2^{iii}) (6×)	79·9 (1)	79·5 (1)
Fe/Co—O(1^i) (3×)	1·989 (1)	2·002 (1)
Fe/Co—O(2)	1·957 (2)	1·957 (4)
Fe/Co—O(1)	2·185 (3)	2·200 (8)
O(1^i)—O(1^{ii}) (3×)	3·418 (1)	3·441 (1)
O(2)—O(1^i) (3×)	2·962 (3)	2·972 (6)
O(1)—O(1^i) (3×)	2·763 (3)	2·782 (8)
O(1^i)—Fe/Co—O(1^{ii}) (3×)	118·4 (1)	118·4 (1)
O(2)—Fe/Co—O(1^i) (3×)	97·3 (1)	97·3 (2)
O(1)—Fe/Co—O(1^i) (3×)	82·7 (1)	82·8 (2)

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

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Structure of [10-(2-Aminophenyl)-5-methyl-1,5,9-triaza-9-decene-*N*,*N*′,*N*″,*N*‴]iodocopper(II) Iodide

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Abstract. $[CuI(C_{14}H_{24}N_4)]I$, $M_r = 565{\cdot}73$, monoclinic, $P2_1/c$, $a = 9{\cdot}277\ (1)$, $b = 18{\cdot}200\ (4)$, $c = 11{\cdot}749\ (1)$ Å, $\beta = 92{\cdot}925\ (8)°$, $V = 1981{\cdot}1\ (5)$ Å^3, $Z = 4$, $D_m = 1{\cdot}91\ (1)$, $D_x = 1{\cdot}897$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 4{\cdot}19$ mm^{-1}, $F(000) = 1084$, room temperature, $R = 0{\cdot}0349$ for 2625 reflections. The Cu^{2+} coordination sphere has a distorted square-pyramidal geometry with four equatorial Cu—N bonds varying from 2·016 (5) to 2·064 (5) Å and an axial Cu—I bond of 2·788 (1) Å. The N atom displacements from the N_4 best plane vary from −0·019 (5) to 0·019 (5) Å and the angle between the Cu—I bond and the N_4 plane is 85·1°. The second iodide which is 5·88 Å from Cu^{2+} acts as a counter-ion in the structure. The three chelate ring conformations can be described as half chair, distorted chair and distorted chair. Both amino groups form hydrogen bonds with the iodide ions.

Experimental. Crystals were from methanol–water solution, density by flotation. Data collected for 0·5 × 0·35 × 0·45 mm crystal on a Syntex $P2_1$ diffractometer, 15 reflections $17 < 2\theta < 25°$ were used to obtain lattice parameters, 3333 unique reflections were measured up to $2\theta = 50°$ (h: ±11, k: 0–21, l: 0–13) using graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, profile analysis according to Lehmann & Larsen (1974). Two standards (054 and

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