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Filled skutterudite structure of europium ruthenium polyphosphide, $EuRu_4P_{12}$

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Key indicators: single-crystal X-ray study; T = 100 K; mean σ(Ru–P) = 0.0003 Å; disorder in main residue; R factor = 0.020; wR factor = 0.024; data-to-parameter ratio = 43.5.

The crystal structure of $EuRu_4P_{12}$ is isotypic with filled skutterudite structures of rare earth transition metal polyphosphides: RFe$_4$P$_{12}$ (R = Ce, Pr, Nd, Sm and Eu), RRu$_4$P$_{12}$ (R = La, Ce, Pr and Nd) and ROs$_4$P$_{12}$ (R = La, Ce, Pr and Nd). The Ru cation is coordinated by six P anions in a distorted octahedral manner. The partially occupied Eu position (site occupancy 0.97) is enclosed by a cage formed by the corner-shared framework of the eight RuP_6 octahedra.

Related literature

The title compound is isotypic with the $Im\bar{3}$ form of $LaFe_4P_{12}$, see: Jeitschko & Braun (1977). For the single-crystal preparation and magnetic and electrical properties of $EuRu_4P_{12}$, see: Sekine *et al.* (2000). For hyperfine interaction in $EuRu_4P_{12}$, see: Grandjean *et al.* (1983); Indoh *et al.* (2002). For the method used to avoid multiple diffraction, see: Takenaka *et al.* (2008).

Experimental

Crystal data

$Eu_{0.97}Ru_4P_{12}$
M_r = 923.37
Cubic, $Im\bar{3}$
a = 8.04163 (10) Å
V = 520.04 (1) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 13.37 mm^{-1}
T = 100 K
0.04 mm (radius)

Data collection

MacScience M06XHF22 four-circle diffractometer
Absorption correction: for a sphere [transmission coefficients for spheres tabulated in International Tables Vol. C (1992), Table 6.3.3.3, were interpolated with Lagrange's method (four-point interpolation; Yamauchi *et al.*, 1965)]
T_{min} = 0.486, T_{max} = 0.526
1564 measured reflections
769 independent reflections
625 reflections with $F > 3\sigma(F)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.024
S = 1.54
1304 reflections
30 parameters
$\Delta\rho_{max}$ = 2.08 e Å^{-3}
$\Delta\rho_{min}$ = −1.14 e Å^{-3}

Table 1
Selected bond lengths (Å).

Eu1—P1	3.1112 (3)	P1—P1^{i}	2.3061 (1)
Eu1—Ru1	3.4821 (1)	P1—P1^{ii}	3.0829 (1)
Ru1—P1	2.3558 (1)		

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

Data collection: *MXCSYS* (MacScience, 1995) and *IUANGLE* (Tanaka *et al.*, 1994); cell refinement: *RSLC-3 UNICS* system (Sakurai & Kobayashi, 1979); data reduction: *RDEDIT* (Tanaka, 2008); program(s) used to solve structure: *QNTAO* (Tanaka *et al.*, 2008); program(s) used to refine structure: *QNTAO*; molecular graphics: *ATOMS for Windows* (Dowty, 2000); software used to prepare material for publication: *RDEDIT*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2130).

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S1. Refinement

Multiple diffraction was avoided by using ψ-scans (Takenaka *et al.*, 2008). Intensities were measured at the equi-temperature region of combination of angles ω and χ of a four-circle diffractometer. The intensities have not been included for the refinement if the multiple diffraction cannot be avoided. In addition, the crystal was cooled to 100 K with an Oxford cryostream cooler installed on a four-circle diffractometer. Since the temperature of the sample depends on the ω and χ-angle and the X-ray diffraction measurement was carried out in the equi-temperature area, the ω and χ-angle had the limitation. Thus completeness of the independent reflection was less than 85%.

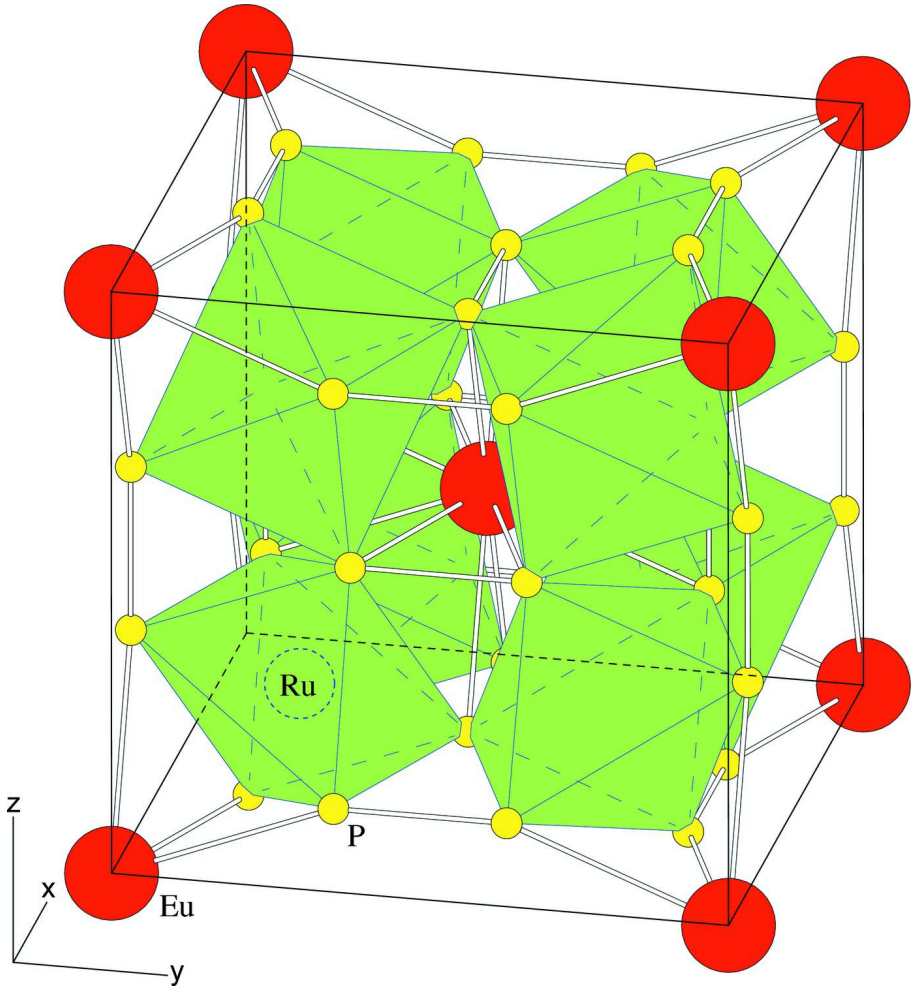

Figure 1

The structure of $EuRu_4P_{12}$ at 100 K. Small yellow and large red spheres, respectively, represent P and Eu atoms. Green distorted octahedron represent RuO_6 units.

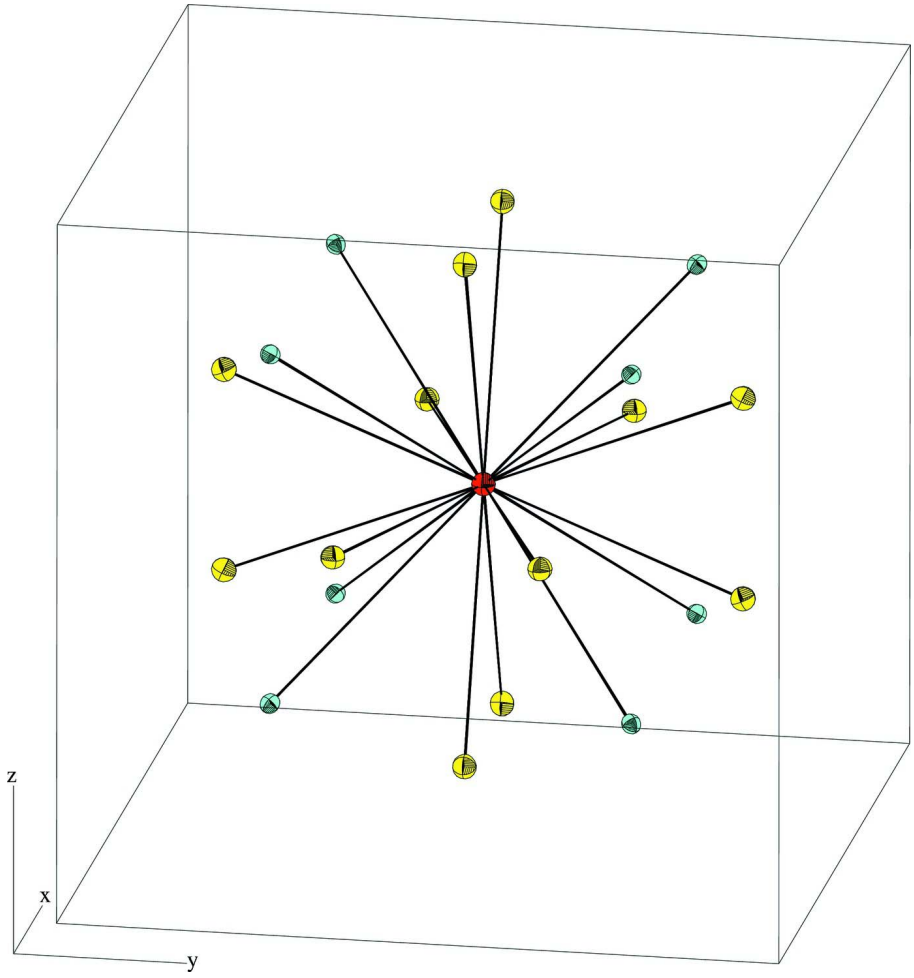

Figure 2

Bonding of Ru_4 and P_{12} around an Eu ion with displacement ellipsoids at the 90% probability level. Red, blue and yellow ellipsoids represent Eu, Ru and P atoms, in Fig. 1.

Europium ruthenium polyphosphide

Crystal data

$Eu_{0.97}Ru_4P_{12}$
M_r = 923.37
Cubic, $Im\bar{3}$
Hall symbol: -I 2 2 3
a = 8.04163 (10) Å
V = 520.04 (1) Å^3
Z = 2
F(000) = 828.4

D_x = 5.925 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 37 reflections
θ = 36.0–37.7°
μ = 13.37 mm^{-1}
T = 100 K
Sphere, black
0.04 mm (radius)

Data collection

MacScience M06XHF22 four-circle diffractometer
Radiation source: fine-focus rotating anode
Graphite monochromator
Detector resolution: 1.25 x 1.25° pixels mm^{-1}

$\omega/2\theta$ scans

Absorption correction: for a sphere
[transmission coefficients for spheres tabulated in International Tables Vol. C (1992), Table 6.3.3.3, were interpolated with Lagrange's method (four-point interpolation; Yamauchi *et al.*, 1965)]
T_{min} = 0.486, T_{max} = 0.526
1564 measured reflections

769 independent reflections
625 reflections with $F > 3\sigma(F)$
R_{int} = 0.016
θ_{max} = 74.2°, θ_{min} = 3.6°
h = −18→20
k = −21→21
l = −18→20

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.024
S = 1.54
1304 reflections
30 parameters

Weighting scheme based on measured s.u.'s
$(\Delta/\sigma)_{max}$ = 0.018
$\Delta\rho_{max}$ = 2.08 e Å^{-3}
$\Delta\rho_{min}$ = −1.14 e Å^{-3}
Extinction correction: B–C type 1 Gaussian isotropic (Becker & Coppens, 1975)
Extinction coefficient: 0.068 (6)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Eu1	0.000000	0.000000	0.000000	0.00270 (2)	0.970 (4)
Ru1	0.250000	0.250000	0.250000	0.001840 (15)	
P1	0.000000	0.359329	0.143386	0.00283 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Eu1	0.00271 (3)	0.00271 (3)	0.00271 (3)	0	0	0
Ru1	0.00185 (3)	0.00185 (3)	0.00185 (3)	0.000108 (17)	0.000108 (17)	0.000108 (17)
P1	0.00268 (10)	0.00301 (10)	0.00285 (10)	0	0	−0.00009 (7)

Geometric parameters (Å, º)

Eu1—P1	3.1112 (3)	Ru1—P1^{i}	2.3558 (1)
Eu1—Ru1	3.4821 (1)	P1—P1^{ii}	2.3061 (1)
Ru1—P1	2.3558 (1)	P1—P1^{i}	3.0829 (1)
Eu1—P1—Ru1	77.78	Ru1—P1—P1^{i}	49.13
Eu1—P1—P1^{ii}	68.25	P1—Ru1—P1^{i}	81.74
Eu1—P1—P1^{i}	109.77	P1^{ii}—P1—P1^{i}	89.59
Ru1—P1—P1^{ii}	111.34		

Symmetry codes: (i) −z+1/2, x+1/2, −y+1/2; (ii) −x, y, −z.