Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Received 11 December 2009; accepted 5 January 2010

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*.

Part of this study was supported by the International Training Programme (ITP) from the Japan Society for the Promotion of Science (JSPS).

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

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supplementary materials

Acta Cryst. (2010). E**66**, i6 [**doi:10.1107/S1600536810000589**]

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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%.

Figures

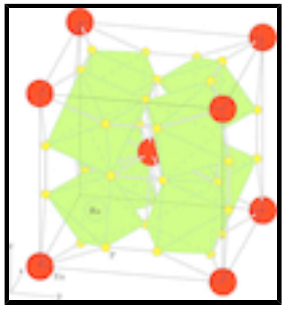

Fig. 1. The structure of $EuRu_4P_{12}$ at 100 K. Small yellow and large red spheres, respectively, represent P and Eu atoms. Green distorted octahedron represent RuO_6 units.

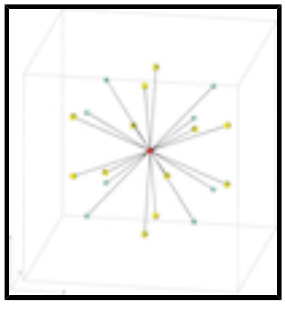

Fig. 2. Bonding of Ru_4 and P_{12} around an Eu ion with displacement ellipsoids at the 90% probability level. Red, blue and yellow ellipsoids represent Eu, Ru and P atoms, in Fig. 1.

Europium ruthenium polyphosphide

Crystal data

$Eu_{0.97}Ru_4P_{12}$	$D_x = 5.925$ Mg m^{-3}
$M_r = 923.37$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cubic, $Im\bar{3}$	Cell parameters from 37 reflections
Hall symbol: -I 2 2 3	$\theta = 36.0$–$37.7°$
$a = 8.04163$ (10) Å	$\mu = 13.37$ mm^{-1}
$V = 520.04$ (1) Å^3	$T = 100$ K
$Z = 2$	Sphere, black

$F(000)$ = 828.4

0.04 mm (radius)

Data collection

MacScience M06XHF22 four-circle diffractometer

Radiation source: fine-focus rotating anode

graphite

Detector resolution: 1.25 x 1.25° pixels mm^{-1}

ω/2θ 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°

$h = -18 \rightarrow 20$

$k = -21 \rightarrow 21$

$l = -18 \rightarrow 20$

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 ($Å^2$)

	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 ($Å^2$)

	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, °)

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, $-z$.

Fig. 1

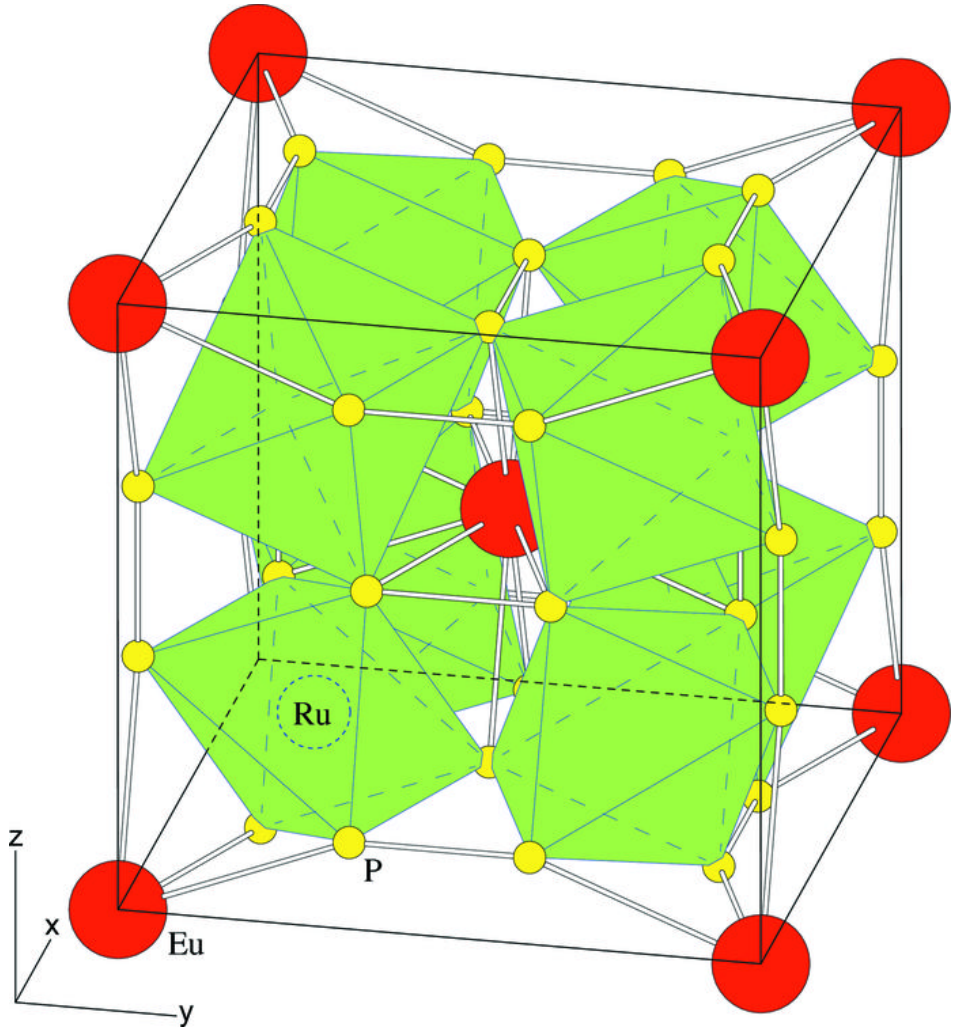

Fig. 2