Monatsh Chem 139, 1137–1142 (2008)
DOI 10.1007/s00706-008-0908-2
Printed in The Netherlands

Monatshefte für Chemie
Chemical Monthly

Rare earth-rich cadmium compounds RE_4TCd (T = Co, Ru, and Rh) with Gd_4RhIn type structure

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Received 22 January 2008; Accepted 4 February 2008; Published online 21 April 2008

Abstract The rare earth-rich cadmium compounds RE_4TCd (RE = Y, La–Nd, Sm, and Gd–Tm, Lu; T = Co, Ru, and Rh) were prepared from the elements in sealed tantalum ampoules in an induction furnace. All samples were characterized by X-ray powder diffraction. The structures of Y_4RuCd (a = 1362.5(1) pm), La_4RuCd (a = 1415.9(1) pm), Gd_4RuCd (a = 1368.8(2) pm), La_4CoCd (a = 1417.9(4) pm), Gd_4CoCd (a = 1356.1(1) pm), and Gd_4RhCd (a = 1368.7(1) pm) were refined from single crystal X-ray diffractometer data. The RE_4TCd compounds crystallize with the cubic Gd_4RhIn type structure, space group $F\bar{4}3m$. The transition metal atoms have tricapped trigonal prismatic rare earth coordination. The trigonal prisms are condensed *via* common edges, forming a rigid three-dimensional network with adamantane symmetry. Voids in these networks are filled by Cd_4 tetrahedra (304 pm Cd–Cd in Gd_4CoCd) and polyhedra of the RE1 atoms. The crystal chemical peculiarities are briefly discussed.

Keywords Rare earth compounds; Cadmium; Crystal chemistry.

Introduction

The rare earth (RE)-transition metal (T)-cadmium systems have only scarcely been investigated. Besides several equiatomic RETCd (T = Pd, Ag, and Au) compounds with orthorhombic TiNiSi or hexagonal ZrNiAl type structure (depending on the rare earth valence) [1–6], also a series of RE_2T_2Cd (T = Ni, Pd, Pt, and Au) [7, 8, and references therein] compounds with Mo_2B_2Fe or Mn_2B_2Al type structure have been structurally characterized. In the transition metal-rich parts of the phase diagrams several ordered *Laves* phases $CeNi_4$Cd and $RECu_4$Cd (RE = Ho, Er, Tm, and Yb) [9] and the compounds $RECu_{5\text{-}x}Cd_x$ (RE = Ce, Gd, Tb, and Yb) [10] have been synthesized.

Remarkable crystal chemistry occurs for $LaNiCd_2$ [11]. In this compound, the cadmium atoms build up a three-dimensional lonsdaleite-like substructure, similar to the indium atoms in the well-known *Zintl* phase $CaIn_2$. $Pr_6Pd_{13}Cd_4$ [12] contains discrete Pr_6Pd octahedra and can be considered as an intermetallic analogon to the subnitride $Na_{16}Ba_6$N.

In the rare earth metal-rich parts of the RE–T–Cd phase diagrams we recently discovered the compounds RE_4TCd (RE = Tb, Dy, and Ho; T = Co, Rh) [13]. They crystallize with the cubic Gd_4RhIn type structure [14], space group $F\bar{4}3m$. Besides the rigid three-dimensional network of condensed transition metal centered trigonal rare earth prisms, Cd_4 tetra-

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hedra are the central structural motif of these compounds. These materials are highly interesting with respect to their magnetic properties. Depending on the rare earth and transition metal, the magnetic ordering temperatures range from 21 (Dy$_4$RhCd) to 54 K (Tb$_4$CoCd). In continuation of our systematic studies of Gd$_4$RhIn type compounds with cadmium [13] and magnesium [15–17], we have now synthesized the complete series RE_4CoCd, RE_4RuCd, and RE_4RhCd.

Table 1 Lattice parameters (*Guinier* powder data) of ternary cadmium compounds RE_4TCd (T = Co, Ru, Rh). Data marked with an asterisk were taken from Ref. [13] for comparison

Compound	a/pm	V/nm^3
RE_4CoCd		
Y$_4$CoCd	1362.6(5)	2.5298
La$_4$CoCd	1417.9(4)	2.8508
Ce$_4$CoCd	1389.4(15)	2.6819
Pr$_4$CoCd	1387.2(1)	2.6692
Nd$_4$CoCd	1379.8(1)	2.6269
Sm$_4$CoCd	1368.0(3)	2.5600
Gd$_4$CoCd	1356.1(1)	2.4936
Tb$_4$CoCd	1349.0(2)	2.4550
Tb$_4$CoCd*	1346.5(2)	2.4413
Dy$_4$CoCd	1337.5(2)	2.3929
Dy$_4$CoCd*	1341.0(3)	2.4115
Ho$_4$CoCd	1332.8(1)	2.3673
Ho$_4$CoCd*	1334.9(2)	2.3787
Er$_4$CoCd	1332.4(1)	2.3655
Tm$_4$CoCd	1324.9(1)	2.3256
Lu$_4$CoCd	1316.2(1)	2.2803
RE_4RuCd		
Y$_4$RuCd	1362.5(1)	2.5291
La$_4$RuCd	1415.9(1)	2.8386
Ce$_4$RuCd	1393.4(1)	2.7053
Pr$_4$RuCd	1392.4(1)	2.6994
Nd$_4$RuCd	1388.0(1)	2.6742
Sm$_4$RuCd	1375.3(1)	2.6014
Gd$_4$RuCd	1368.8(2)	2.5647
Tb$_4$RuCd	1363.0(2)	2.5319
Dy$_4$RuCd	1355.9(2)	2.4926
Ho$_4$RuCd	1346.7(4)	2.4421
Er$_4$RuCd	1342.5(1)	2.4198
Tm$_4$RuCd	1336.3(1)	2.3863
Lu$_4$RuCd	1331.6(2)	2.3610
RE_4RhCd		
Y$_4$RhCd	1364.3(1)	2.5392
La$_4$RhCd	1422.6(1)	2.8789
Ce$_4$RhCd	1404.2(2)	2.7686
Pr$_4$RhCd	1398.7(1)	2.7361
Nd$_4$RhCd	1388.8(1)	2.6787
Sm$_4$RhCd	1377.2(1)	2.6121
Gd$_4$RhCd	1368.7(1)	2.5639
Tb$_4$RhCd	1359.2(1)	2.5112
Tb$_4$RhCd*	1357.3(1)	2.5005
Dy$_4$RhCd	1353.0(1)	2.4769
Dy$_4$RhCd*	1352.9(1)	2.4763
Ho$_4$RhCd	1345.1(1)	2.4337
Ho$_4$RhCd*	1348.3(1)	2.4511
Er$_4$RhCd	1342.0(2)	2.4169
Tm$_4$RhCd	1338.2(2)	2.3964
Lu$_4$RhCd	1329.6(1)	2.3506

Results and discussion

Thirty-three new RE_4TCd compounds with the cubic Gd$_4$RhIn type structure [14], space group $F\bar{4}3m$, have been synthesized and structurally characterized (Tables 1–5). These series exist with cobalt, ruthenium, and rhodium as transition metal component. The cell volumes of the cerium compounds fit well in the *Iandelli* plots, indicating trivalent cerium. Detailed magnetic studies of these series are under way in order to clarify the magnetic ground states. So far, no RE_4TMg or RE_4TCd compounds with europium or ytterbium as rare earth component have been observed. Most likely europium or ytterbium prefer the divalent state and thus do not meet the electronic requirements for the network of condensed trigonal prisms (Fig. 1). Similar observation has been made for the RE_4TIn compounds [14, 18].

The striking structural motifs in the RE_4TCd compounds are transition metal centered trigonal prisms of the rare earth atoms and Cd$_4$ tetrahedra (304.2(4) pm Cd–Cd in Gd$_4$CoCd, close to *hcp* cadmium (6 × 298 and 6 × 329 pm) [19]). As emphasized in Fig. 1, the RE_6T trigonal prisms are condensed *via* common corners and edges, leading to a rigid three-dimensional network in which the Cd$_4$ tetrahedra and the coordination polyhedra of the RE1 atoms are embedded. For further crystal chemical details we refer to our previous work on the prototype [14–18].

Our recent electronic structure calculations on isotypic La$_4$CoMg [15] and La$_4$RuMg [17] revealed very strong La–Co and La–Ru bonding within the trigonal prismatic units. This goes along with short La–Co and La–Ru distances. Similar situation is observed for the cadmium compounds reported herein. To give an example, the Gd–Rh distances in Gd$_4$RhCd of 282.8(2) and 284.1(2) pm are close to the sum of the covalent radii of 286 pm [20]. Similar

Table 2 Crystal data and structure refinement for RE_4RuCd, Gd_4RhIn type, space group $F\bar{4}3m$, $Z = 16$

Empirical formula	Y_4RuCd	La_4RuCd	Gd_4RuCd
Molar mass/$g \cdot mol^{-1}$	569.11	769.11	842.47
Unit cell dimensions/pm	$a = 1362.5(1)$	$a = 1415.9(1)$	$a = 1368.8(2)$
Calculated density/$g \cdot cm^{-3}$	5.98	7.20	8.73
Crystal size/μm^3	$20 \times 100 \times 100$	$40 \times 90 \times 160$	$20 \times 40 \times 60$
Detector distance/mm	60	60	60
Exposure time/min	5	5	5
ω range/° increment/°	0–180; 1.0	0–180; 1.0	0–180; 1.0
Integr. param. A, B, EMS	13.5, 3.5, 0.014	13.5, 3.5, 0.012	13.5, 3.5, 0.014
Transm. ratio (max/min)	0.375/0.144	0.403/0.081	0.627/0.113
Absorption coefficient/mm^{-1}	41.7	28.4	46.2
$F(000)$	3968	5120	5568
θ range/°	2–33	2–34	2–34
Range in *hkl*	± 20, $-19/20$, ± 20	± 21, $-21/20$, ± 21	$-21/19$, ± 21, $-20/21$
Total no. reflections	7384	9834	4831
Independent reflections	530 ($R_{int} = 0.143$)	605 ($R_{int} = 0.123$)	518 ($R_{int} = 0.079$)
Reflections with $I > 2\sigma(I)$	374 ($R_\sigma = 0.111$)	512 ($R_\sigma = 0.055$)	402 ($R_\sigma = 0.073$)
Data/parameters	530/19	605/19	518/19
Goodness-of-fit on F^2	0.644	0.906	0.751
Final R indices $[I > 2\sigma(I)]$	$R1 = 0.030$ $wR2 = 0.043$	$R1 = 0.026$ $wR2 = 0.053$	$R1 = 0.025$ $wR2 = 0.044$
R indices (all data)	$R1 = 0.051$ $wR2 = 0.046$	$R1 = 0.034$ $wR2 = 0.054$	$R1 = 0.038$ $wR2 = 0.045$
Extinction coefficient	0.000186(11)	0.000116(9)	0.000128(6)
Flack parameter	−0.01(2)	0.04(6)	0.01(4)
Largest diff. peak and hole/eÅ^3	1.25/−1.27	3.36/−1.88	6.05/−2.02

Table 3 Crystal data and structure refinement for RE_4TCd (T = Co, Rh), Gd_4RhIn type, space group $F\bar{4}3m$, $Z = 16$

Empirical formula	La_4CoCd	Gd_4CoCd	Gd_4RhCd
Molar mass	726.97 g/mol	800.33 g/mol	844.31 g/mol
Unit cell dimensions	$a = 1417.9(4)$ pm	$a = 1356.1(1)$ pm	$a = 1368.7(1)$ pm
Calculated density	6.78 g/cm^3	8.51 g/cm^3	8.75 g/cm^3
Crystal size	$20 \times 60 \times 75$ µm	$20 \times 30 \times 75$ µm	$20 \times 30 \times 75$ µm
Detector distance	80 mm	80 mm	80 mm
Exposure time	5 min	5 min	10 min
ω range; increment	0–180°; 1.0°	0–180°, 1.0°	0–180°; 1.0°
Integr. param. A, B, EMS	13.5, 3.5, 0.010	13.5, 3.5, 0.012	14.0, 4.0, 0.018
Transm. ratio (max/min)	0.352/0.201	0.463/0.102	no. abs. corr.
Absorption coefficient	28.5 mm^{-1}	47.7 mm^{-1}	46.4 mm^{-1}
$F(000)$	4848	5296	5584
θ range	2–31°	2–30°	2–30°
Range in *hkl*	± 20, ± 20, $-20/18$	$-19/16$, ± 19, ± 19	± 19, ± 19, $-16/19$
Total no. reflections	7812	6661	3975
Independent reflections	485 ($R_{int} = 0.153$)	420 ($R_{int} = 0.177$)	395 ($R_{int} = 0.161$)
Reflections with $I > 2\sigma(I)$	356 ($R_\sigma = 0.089$)	274 ($R_\sigma = 0.130$)	257 ($R_\sigma = 0.143$)
Data/parameters	485/19	420/19	395/19
Goodness-of-fit on F^2	0.691	0.516	0.529
Final R indices $[I > 2\sigma(I)]$	$R1 = 0.027$ $wR2 = 0.047$	$R1 = 0.025$ $wR2 = 0.041$	$R1 = 0.030$ $wR2 = 0.044$
R indices (all data)	$R1 = 0.044$ $wR2 = 0.049$	$R1 = 0.052$ $wR2 = 0.046$	$R1 = 0.057$ $wR2 = 0.050$
Extinction coefficient	0.000107(9)	0.000073(7)	0.000074(9)
Flack parameter	−0.08(13)	−0.05(8)	−0.03(7)
Largest diff. peak and hole	1.88/−1.10 e/Å^3	1.73/−1.43 e/Å^3	2.43/−1.48 e/Å^3

Table 4 Atomic coordinates and isotropic displacement parameters (pm^2) of RE_4TCd. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor. Note that the Gd_4CoCd, La_4RuCd, Gd_4RuCd, and Gd_4RhCd crystals had the other absolute structure

Atom	*Wyckoff* site	*x*	*y*	*z*	U_{eq}
La_4CoCd					
La1	24*g*	0.55994(9)	1/4	1/4	136(3)
La2	24*f*	0.19044(9)	0	0	124(3)
La3	16*e*	0.34745(7)	*x*	*x*	121(3)
Co	16*e*	0.14088(16)	*x*	*x*	169(8)
Cd	16*e*	0.57796(8)	*x*	*x*	110(4)
Gd_4CoCd					
Gd1	24*g*	0.43771(10)	3/4	3/4	83(3)
Gd2	24*f*	0.81074(11)	0	0	72(3)
Gd3	16*e*	0.65221(8)	*x*	*x*	78(3)
Co	16*e*	0.8585(2)	*x*	*x*	113(10)
Cd	16*e*	0.42068(10)	*x*	*x*	72(5)
Y_4RuCd					
Y1	24*g*	0.56116(11)	1/4	1/4	84(4)
Y2	24*f*	0.18943(11)	0	0	57(3)
Y3	16*e*	0.34737(9)	*x*	*x*	78(4)
Ru	16*e*	0.14038(7)	*x*	*x*	114(4)
Cd	16*e*	0.57958(7)	*x*	*x*	84(3)
La_4RuCd					
La1	24*g*	0.44021(6)	3/4	3/4	115(2)
La2	24*f*	0.80952(6)	0	0	99(2)
La3	16*e*	0.65318(4)	*x*	*x*	89(2)
Ru	16*e*	0.86213(7)	*x*	*x*	133(3)
Cd	16*e*	0.42193(6)	*x*	*x*	107(2)
Gd_4RuCd					
Gd1	24*g*	0.43889(8)	3/4	3/4	86(2)
Gd2	24*f*	0.80944(8)	0	0	71(2)
Gd3	16*e*	0.65280(5)	*x*	*x*	62(2)
Ru	16*e*	0.86091(9)	*x*	*x*	92(4)
Cd	16*e*	0.42073(8)	*x*	*x*	77(3)
Gd_4RhCd					
Gd1	24*g*	0.43603(11)	3/4	3/4	84(3)
Gd2	24*f*	0.80958(12)	0	0	74(3)
Gd3	16*e*	0.65074(9)	*x*	*x*	66(4)
Rh	16*e*	0.85794(14)	*x*	*x*	82(6)
Cd	16*e*	0.42026(12)	*x*	*x*	76(5)

to the magnesium based compounds, the geometry of the trigonal prisms strongly depends on the valence electron concentration. As an example, the Gd_6T units of Gd_4CoCd, Gd_4RuCd, and Gd_4RhCd are presented in Fig. 2 together with the relevant interatomic distances. While the cobalt and rhodium compound with the higher valence electron concentration have almost equal Gd–*T* distances, the Gd2–Ru and Gd3–Ru distances in Gd_4RuCd differ by

Table 5 Interatomic distances (pm) in the structures of Gd_4CoCd, Gd_4RuCd, and Gd_4RhCd. Standard deviations are given in parentheses. All distances within the first coordination spheres are listed

		Gd_4CoCd	Gd_4RuCd	Gd_4RhCd
Gd1				
2	Cd	328.2(2)	331.4(2)	330.3(2)
2	*T*	345.9(1)	348.0(1)	351.0(1)
2	Gd3	346.1(1)	348.1(1)	351.1(1)
4	Gd2	359.0(1)	361.6(1)	362.5(1)
4	Gd1	360.0(2)	365.7(2)	360.1(2)
Gd2				
2	*T*	279.0(3)	278.3(1)	282.8(2)
2	Cd	348.8(1)	350.4(1)	350.4(2)
4	Gd1	359.0(1)	361.6(1)	362.5(1)
2	Gd3	362.5(1)	365.3(1)	363.9(1)
4	Gd2	363.0(2)	368.9(2)	368.6(2)
Gd3				
3	*T*	280.5(3)	286.1(2)	284.1(2)
3	Cd	343.7(2)	348.1(1)	344.1(2)
3	Gd1	346.1(1)	348.0(1)	351.1(1)
3	Gd2	362.5(1)	365.3(1)	363.9(1)
3	Gd3	375.1(3)	376.3(2)	384.3(3)
T				
3	Gd2	279.0(3)	278.3(1)	282.8(2)
3	Gd3	280.5(3)	286.1(2)	284.1(2)
3	Gd1	345.9(1)	348.1(1)	351.0(1)
Cd				
3	Cd	304.2(4)	306.9(3)	308.7(5)
3	Gd1	328.1(2)	331.4(2)	330.3(2)
3	Gd3	343.7(2)	348.1(1)	344.1(2)
3	Gd2	348.8(1)	350.4(1)	350.4(2)

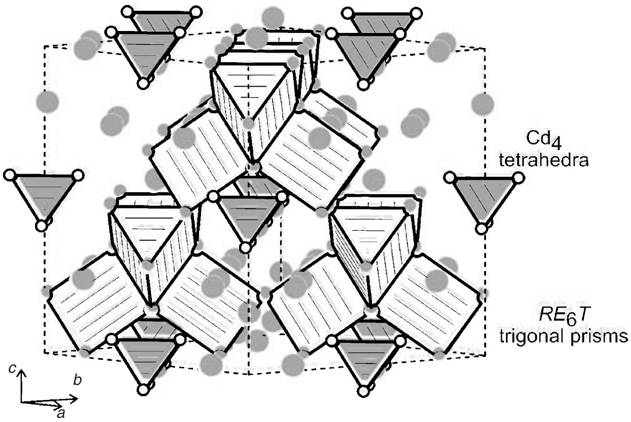

Fig. 1 View of the RE_4TCd structure approximately along one of the plane diagonals

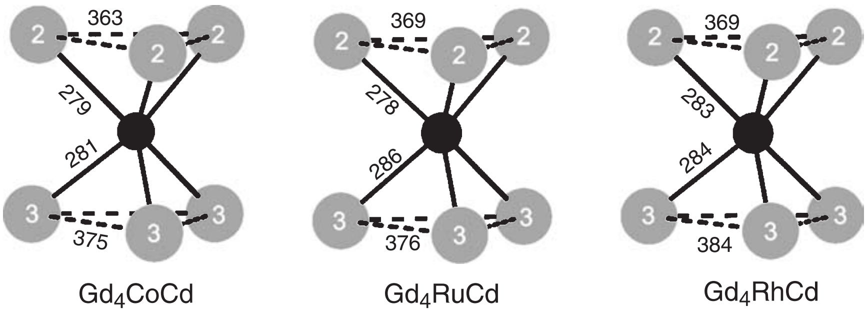

Fig. 2 The trigonal prismatic building units in Gd_4TCd (T = Co, Ru, and Rh). Atom designations and relevant interatomic distances are given

8 pm (Table 5). In view of the similar courses of the interatomic distances (Mg *vs.* Cd compounds), we can thus safely apply a rigid band model for the electronic structures of the cadmium compounds.

Experimental

Synthesis

Starting materials for the preparation of the RE_4TCd samples were ingots of the rare earth elements (Johnson Matthey or smart elements), cobalt powder (Sigma-Aldrich, 100 mesh), ruthenium and rhodium powder (*ca.* 200 mesh, Degussa-Hüls), and a cadmium rod (Johnson-Matthey), all with a stated purity better than 99.9%. Pieces of the rare earth ingots (except for samarium and thulium) were first arc-melted [21] into small buttons under argon. The argon was purified before with molecular sieves, silica gel and titanium sponge (900 K). The elements were then weighed in the ideal 4:1:1 atomic ratio and sealed in small tantalum tubes under argon. The tantalum ampoules were then placed in a water-cooled quartz sample chamber of a high frequency furnace (Hüttinger Elektronik, Freiburg, type TIG 1.5/300) under flowing argon [22] and first heated at 1370 K (La, Ce, Pr compounds), respectively 1420 K (Nd–Lu compounds) for about three minutes. The samples were then cooled to 1020 K within 5 min and kept at that temperature for another 3 h. The temperature was controlled through a Sensor Therm Methis MS09 pyrometer with an accuracy of ±30 K. The samples could easily be separated from the tantalum tubes. No reaction with the tantalum containers was observed. The RE_4TCd samples are stable in moist air over months in powdered as well as in polycrystalline form. Single crystals exhibit metallic lustre while ground powder is dark grey.

The samples for crystal growth were also prepared in inert containers. The elements were cold-pressed to small pellets and sealed in tantalum ampoules which were sealed in silica tubes for oxidation protection. The tubes were heated within 4 h to 1420 K in a muffle furnace, kept at that temperature for 6 h, cooled to 1020 K at a rate of 2 K/h and finally annealed at that temperature for 7 days. The ampoules were then cooled to room temperature within 6 h.

Scanning electron microscopy

The single crystals investigated on the diffractometers have been analyzed by EDX measurements using a LEICA 420 I scanning electron microscope with the rare earth trifluorides, cobalt, ruthenium, rhodium, and cadmium as standards. Since the crystals were mounted by beeswax on glass fibres, they were first coated with a thin carbon film. No impurity elements were detected. The semiquantitative analyses were in agreement with the ideal compositions.

X-Ray film data and structure refinements

The RE_4TCd samples were characterized through *Guinier* powder patterns (Cu $K\alpha_1$ radiation; α-quartz (a = 491.30, c = 540.46 pm) as internal standard). The *Guinier* camera was equipped with an imaging plate system (Fujifilm BAS-1800). The cubic lattice parameters (Table 1) were obtained from least-squares fits to the powder data. The correct indexing was ensured through intensity calculations [23]. The powder and single crystal lattice parameters agreed well.

Well shaped single crystals of Y_4RuCd, La_4RuCd, Gd_4RuCd, La_4CoCd, Gd_4CoCd, and Gd_4RhCd were selected from the annealed samples and first examined by *Laue* photographs on a *Buerger* precession camera (equipped with an imaging plate system Fujifilm BAS-1800) in order to establish suitability for intensity data collection. Intensity data were collected on a *Stoe* IPDS II diffractometer (graphite monochromatized Mo $K\alpha$ radiation; oscillation mode) and numerical absorption corrections were applied to the data sets. The Gd_4RhCd crystal consisted of three domains of which only one was taken for data integration. No absorption correction was applied to this data set. Also the Gd_4RuCd crystal was twinned and only non-overlapping reflections have been used for the structure refinement. All relevant crystallographic details for the data collections and evaluations are listed in Tables 2 and 3.

The systematic extinctions of the six data sets were compatible with space group $F\bar{4}3m$, in agreement with the earlier investigations on the isotypic magnesium compounds [15–17]. The atomic parameters of La_4RuMg [17] were used as starting values and the structures were refined using SHELXL-97 (full-matrix least-squares on F_o^2) [24] with anisotropic atomic displacement parameters for all sites. Since some of the magnesium based compounds revealed RE1/Mg mixing, the occupancy parameters for all crystals were refined in separate series of least-squares cycles. All sites were fully occupied within three standard deviations and in the final cycles the ideal occupancy parameters were assumed again. Refinement of the correct absolute structure was ensured through calculation of the *Flack* parameter [25, 26]. Final difference *Fourier* syntheses revealed no significant residual peaks (Tables 2 and 3). The higher residual peak for Gd_4RuCd most likely results from the overlapping domains (*vide infra*). The positional parameters and interatomic distances (exemplarily for the gadolinium compounds) are listed in Tables 4 and 5. Further details on the structure refinements may be obtained from Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen (Germany), by quoting the Registry Nos. CSD-419070 (Y_4RuCd), CSD-419069 (La_4RuCd), CSD-419068 (Gd_4RuCd), CSD-419067 (La_4CoCd), CSD-419066 (Gd_4CoCd), and CSD-419065 (Gd_4RhCd).

Acknowledgements

We thank Dipl.-Ing. *U.Ch. Rodewald* for the intensity data collections. This work was financially supported by the Deutsche Forschungsgemeinschaft.

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