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$RbHo_2Cu_3S_5$

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(Cu–S) = 0.001 Å
R factor = 0.023
wR factor = 0.054
Data-to-parameter ratio = 17.7

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

Rubidium diholmium tricopper pentasulfide, $RbHo_2Cu_3S_5$, crystallizes in the orthorhombic space group *Cmcm* and is isostructural with $RbSm_2Ag_3Se_5$. In the asymmetric unit, the site symmetries of atoms Rb, Cu1, and S2 are *mm* and those of the other atoms are *m*. The structure has a three-dimensional tunnel framework, with tunnels built from HoS_6 octahedra and CuS_4 tetrahedra. The tunnels are filled with Rb atoms.

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Comment

Ternary and quaternary rare-earth chalcogenides containing a combination of *d*- and *f*-elements have been reviewed recently (Mitchell & Ibers, 2002). We report here the structure of $RbHo_2Cu_3S_5$, a new member of this large family. $RbHo_2Cu_3S_5$, which has the $RbSm_2Ag_3Se_5$ structure type (Huang & Ibers, 2000), crystallizes in space group *Cmcm* of the orthorhombic system. In the asymmetric unit (Fig. 1), the site symmetries of atoms Rb, Cu1, and S2 are *mm* and those of the other atoms are *m*. The structure of $RbHo_2Cu_3S_5$ (Fig. 2) possesses a three-dimensional tunnel framework built from HoS_6 octahedra and CuS_4 tetrahedra. The tunnel, comprising ten-membered rings of six Cu—S bonds and four Ho—S bonds, is only large enough in cross-section to accommodate one Rb atom. Each Rb atom is surrounded by a bicapped trigonal prism of eight S atoms, with Rb—S separations ranging from 3.293 (1) to 3.460 (1) Å, comparable with those of 3.247 (2)–3.7951 (4) Å in $RbNd_2CuS_4$ (Huang & Ibers, 2000). The Ho—S bond distances range from 2.6497 (8) to 2.787 (1) Å, consistent with those of 2.672 (2)–2.8009 (3) Å in $K_2Ho_4Cu_4S_9$ (Yao *et al.*, 2003), and the Cu—S bond distances range from 2.321 (1) to 2.547 (2) Å, comparable with those of 2.3448 (9) to 2.534 (2) Å in $K_2Ho_4Cu_4S_9$ (Yao *et al.*, 2003).

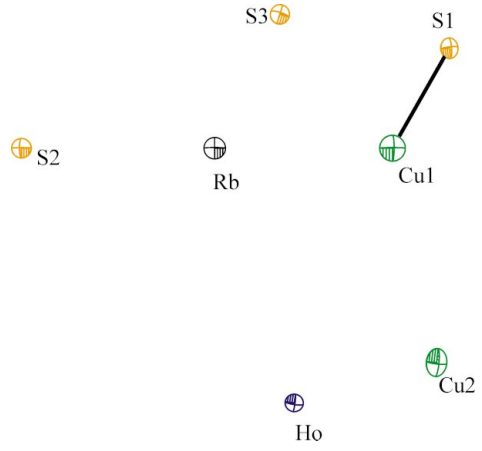

Figure 1
A view of the asymmetric unit of $RbHo_2Cu_3S_5$, with displacement ellipsoids drawn at the 90% probability level.

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Experimental

$RbHo_2Cu_3S_5$ was obtained as yellow needles from a solid-state reaction of Rb_2S_3 (1.2 mmol), Ho (Aldrich, 99%, 1.0 mmol), Cu (Aldrich, 99.999%, 0.5 mmol), and S (Aldrich, 99.5%, 2.0 mmol). The Rb_2S_3 reactive flux (Sunshine *et al.*, 1987) was prepared by the stoichiometric reaction of Rb (Aldrich, 98+%) and S in liquid NH_3. The reactants were loaded into a fused-silica tube under an argon atmosphere in a glove-box. The tube was sealed under a 10^{-4} Torr atmosphere and then placed in a computer-controlled furnace. The sample was heated to 1173 K over a period of 25 h, kept at 1173 K for 3 d, slowly cooled at a rate of 10 K h^{-1} to 473 K, and then cooled rapidly to room temperature.

Crystal data

$RbHo_2Cu_3S_5$
M_r = 766.25
Orthorhombic, *Cmcm*
a = 3.9451 (11) Å
b = 13.915 (4) Å
c = 16.408 (5) Å
V = 900.8 (4) Å^3
Z = 4
D_x = 5.650 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4507 reflections
θ = 2.5–29.0°
μ = 30.77 mm^{-1}
T = 153 (2) K
Needle, yellow
0.42 × 0.068 × 0.030 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: numerical face indexed
T_{min} = 0.047, T_{max} = 0.405
5433 measured reflections
673 independent reflections
661 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 29.0°
$h = -5 \rightarrow 5$
$k = -18 \rightarrow 18$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.054
S = 1.39
673 reflections
38 parameters
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.37 e Å^{-3}
$\Delta\rho_{min}$ = −2.55 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00404 (19)

Table 1
Selected geometric parameters (Å, °).

Rb—S3	3.2929 (15)	Ho—S3^{vi}	2.7865 (10)
Rb—S2^{i}	3.3410 (15)	Cu1—S2^{vii}	2.3212 (10)
Rb—S1^{ii}	3.4604 (12)	Cu1—S1^{iv}	2.5438 (14)
Ho—S2^{iii}	2.6497 (8)	Cu2—S1^{viii}	2.3641 (14)
Ho—S3^{iv}	2.6957 (14)	Cu2—S3^{v}	2.3674 (8)
Ho—S1^{v}	2.6995 (9)	Cu2—S1^{iv}	2.5473 (15)
S2^{iii}—Ho—S3^{iv}	172.13 (4)	S3^{vi}—Ho—S3^{v}	90.13 (4)
S2^{iii}—Ho—S1^{v}	92.47 (3)	S2^{vii}—Cu1—S2^{i}	116.38 (7)
S3^{iv}—Ho—S1^{v}	92.89 (3)	S2^{i}—Cu1—S1^{iv}	105.05 (2)
S1^{v}—Ho—S1^{vi}	93.89 (4)	S1^{iv}—Cu1—S1	120.96 (6)
S2^{iii}—Ho—S3^{vi}	87.95 (4)	S1^{viii}—Cu2—S3^{v}	108.94 (4)
S3^{iv}—Ho—S3^{vi}	86.50 (3)	S3^{v}—Cu2—S3^{vi}	112.86 (5)
S1^{v}—Ho—S3^{vi}	178.05 (3)	S1^{viii}—Cu2—S1^{iv}	115.43 (4)
S1^{vi}—Ho—S3^{vi}	87.99 (3)	S3^{v}—Cu2—S1^{iv}	105.36 (4)

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

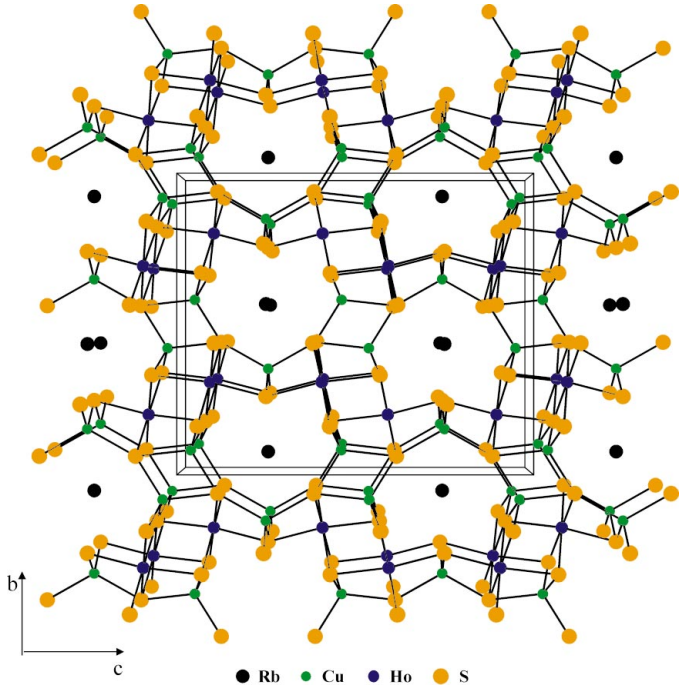

Figure 2
The structure of $RbHo_2Cu_3S_5$, viewed down [100].

The highest residual electron density is 0.87 Å from the Ho site and the deepest hole is 0.70 Å from this same site.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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