Redetermination of Ba_2CdTe_3 from single-crystal X-ray data

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The previous structure determination of the title compound, dibarium tritelluridocadmate, was based on powder X-ray diffraction data [Wang & DiSalvo (1999). *J. Solid State Chem.* **148**, 464–467]. In the current redetermination from single-crystal X-ray data, all atoms were refined with anisotropic displacement parameters. The previous structure report is generally confirmed, but with some differences in bond lengths. Ba_2CdTe_3 is isotypic with Ba_2MX_3 (M = Mn, Cd; X = S, Se) and features $^1_\infty[CdTe_{2/2}Te_{2/1}]^{4-}$ chains of corner-sharing $CdTe_4$ tetrahedra running parallel [010]. The two Ba^{2+} cations are located between the chains, both within distorted monocapped trigonal–prismatic coordination polyhedra. All atoms in the structure are located on a mirror plane.

Related literature

For the previous determination of Ba_2CdTe_3, see: Wang & DiSalvo (1999). For isotypic compounds, see: Grey & Steinfink (1971) for Ba_2MnS_3 and Ba_2MnSe_3; Iglesias *et al.* (1974) for Ba_2CdSe_3 and Ba_2CdS_3.

Experimental

Crystal data

Ba_2CdTe_3	V = 892.85 (3) Å^3
M_r = 769.88	Z = 4
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
a = 9.8405 (2) Å	μ = 20.59 mm^{-1}
b = 4.7502 (1) Å	T = 293 K
c = 19.1008 (4) Å	$0.07 \times 0.03 \times 0.03$ mm

Data collection

Bruker APEXII CCD diffractometer	4014 measured reflections
Absorption correction: multi-scan ($SADABS$; Bruker, 2005)	1098 independent reflections
T_{min} = 0.348, T_{max} = 0.627	858 reflections with $I > 2\sigma(I)$
	R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025	38 parameters
$wR(F^2)$ = 0.041	$\Delta\rho_{max}$ = 1.44 e Å^{-3}
S = 0.99	$\Delta\rho_{min}$ = −1.27 e Å^{-3}
1098 reflections	

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

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Iglesias, J. E., Pachali, K. E. & Steinfink, H. (1974). *J. Solid State Chem.* **9**, 6–14.
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supporting information

Redetermination of Ba$_2$CdTe$_3$ from single-crystal X-ray data

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S1. Comment

Single crystals of Ba$_2$CdTe$_3$ were obtained unintentionally from a Bi-flux reactions for exploration of possible new ternary phases in the Ba—Cd—Te system.

The structure of Ba$_2$CdTe$_3$ is isotypic with Ba$_2$MnX_3 (X = S, Se; Grey & Steinfink, 1971) and Ba$_2$CdX_3 (X = S, Se; Iglesias *et al.*, 1974). The structural set-up can be described as a packing of polyanionic chains composed of corner-sharing CdTe$_4$ tetrahedra. These chains run parallel to [010]; inbetween the chains the two Ba^{2+} cations are located (Fig. 1), both with a coordination number of 7 and surrounded in form of monocapped trigonal-prismatic polyhedra of Te atoms. All atoms are located on a mirror plane x, 1/4, z (Wyckoff symbol 4c).

In comparison with the previous structure model on basis of powder X-ray data (Wang & DiSalvo, 1999), the most important improvement of the current redetermination is reflected in the higher precision of the atomic coordinates and the use of anisotropic displacemenet parameters for all atoms. Although the coordination spheres of Cd and the two Ba atoms can still be described as a distorted CdTe$_4$ tetrahedron and two distorted monocapped trigonal BaTe$_7$ prisms, respectively, the results of the redetermination indicate some differences in terms of Cd—Te and Ba—Te bond lengths (mean σ for the bond length of the powder model in the range 0.003 Å; 0.0006 for the current model). For example, the longest Ba—Te bonds are 3.6722 (8) and 3.6796 (8) Å for Ba1 and Ba2. The previous powder study revealed distances of 3.638 (5) and 3.500 (5) Å, respectively.

S2. Experimental

The title compound was synthesized through a high temperature metal flux reaction. All starting elements were handled inside an Argon-filled glove box with controlled oxygen and moisture levels below 0.1 p.p.m.. The reaction conditions were optimized as follows: Ba, Cd, Te and Bi in a molar ratio of 2:1:3:10 were loaded in an alumina crucible, which were subsequently flame-sealed in a fused silica tube. The reactants were heated quickly to 973 K and allowed to dwell at this temperature for 20 h. After a slow cooling process down to 773 K at a rate of 5 K/h and the removal of the Bi flux by centrifugation, high-quality single crystals of Ba$_2$CdTe$_3$ were obtained.

S3. Refinement

The full occupancies for all sites were verified by freeing the site occupation factor for an individual atom, while other remaining parameters were kept fixed. This proved that all positions are fully occupied with corresponding deviations from full occupancy within 3σ. The residual electron densities show a maximum peak of 1.44 e/Å^3 and a minimum hole of -1.27 e/Å^3, which are 0.86 and 0.81 Å from Te3 and Te2, respectively.
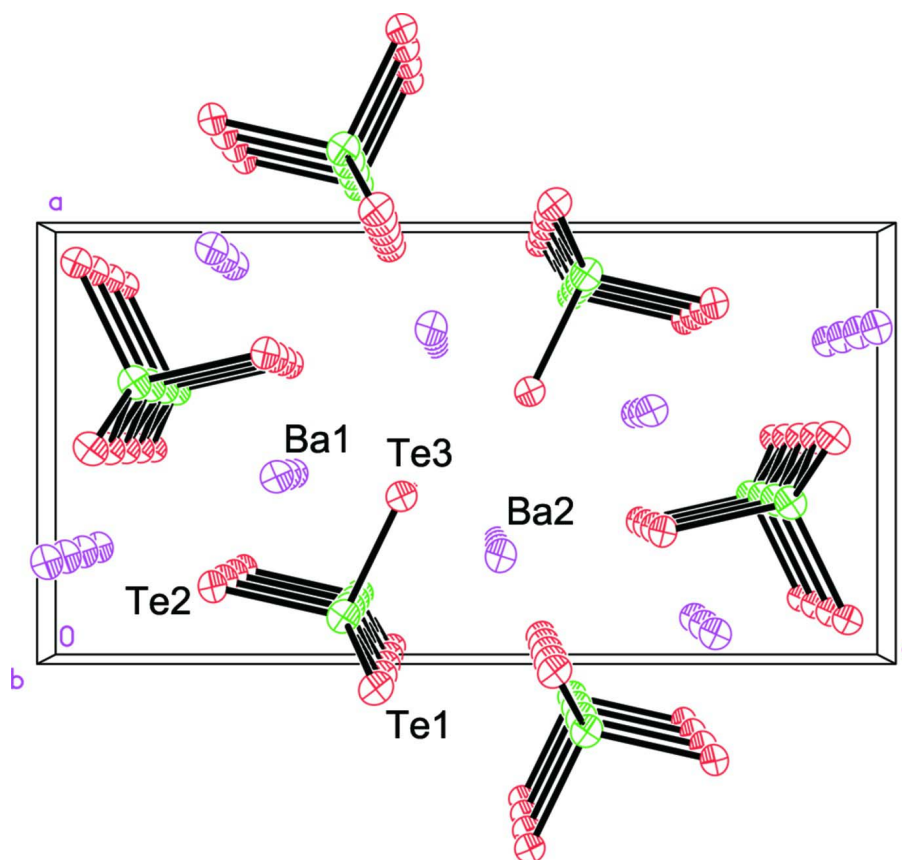
Figure 1

View of the structure of Ba₂CdTe₃ along the *b*-axis. The barium, cadmium and tellurium atoms are plotted as purple, green and red ellipsoids, respectively. Ellipsoids are drawn at the 90% probability level.

dibarium tritelluridocadmate

Crystal data

Ba_2CdTe_3	$F(000) = 1264$
$M_r = 769.88$	$D_x = 5.727$ Mg m⁻³
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2n	Cell parameters from 1715 reflections
$a = 9.8405\ (2)$ Å	$\theta = 3.0–28.2°$
$b = 4.7502\ (1)$ Å	$\mu = 20.59$ mm⁻¹
$c = 19.1008\ (4)$ Å	$T = 293$ K
$V = 892.85\ (3)$ Å³	Needle, red
$Z = 4$	$0.07 \times 0.03 \times 0.03$ mm

Data collection

Bruker APEXII CCD diffractometer	4014 measured reflections
Radiation source: fine-focus sealed tube	1098 independent reflections
Graphite monochromator	858 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.031$
Absorption correction: multi-scan	$\theta_{max} = 27.1°$, $\theta_{min} = 2.1°$
(*SADABS*; Bruker, 2005)	$h = -12 \rightarrow 9$
$T_{min} = 0.348$, $T_{max} = 0.627$	$k = -3 \rightarrow 6$
	$l = -24 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.025$	$w = 1/[\sigma^2(F_o^2) + (0.0142P)^2]$
$wR(F^2) = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
1098 reflections	$\Delta\rho_{max} = 1.44$ e Å^{-3}
38 parameters	$\Delta\rho_{min} = -1.27$ e Å^{-3}
0 restraints	Extinction correction: *SHELXTL* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00112 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	U_{iso}*/U_{eq}
Ba1	0.07317 (6)	0.2500	0.78653 (3)	0.01857 (16)
Ba2	0.24597 (6)	0.2500	0.03873 (3)	0.01918 (16)
Cd1	0.12996 (7)	0.2500	0.36470 (3)	0.01947 (18)
Te1	0.01147 (6)	0.2500	0.59692 (3)	0.01802 (17)
Te2	0.19335 (6)	0.2500	0.22108 (3)	0.01748 (16)
Te3	0.38656 (6)	0.2500	0.42865 (3)	0.01745 (17)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ba1	0.0190 (3)	0.0171 (3)	0.0196 (4)	0.000	−0.0003 (3)	0.000
Ba2	0.0209 (3)	0.0191 (3)	0.0175 (3)	0.000	−0.0013 (3)	0.000
Cd1	0.0197 (4)	0.0201 (4)	0.0186 (4)	0.000	0.0014 (3)	0.000
Te1	0.0187 (4)	0.0154 (3)	0.0200 (4)	0.000	0.0024 (3)	0.000
Te2	0.0178 (4)	0.0203 (3)	0.0144 (4)	0.000	−0.0008 (3)	0.000
Te3	0.0158 (4)	0.0200 (3)	0.0165 (4)	0.000	0.0004 (3)	0.000

Geometric parameters (Å, º)

Ba1—Te2[i]	3.5331 (6)	Cd1—Te1[iii]	2.8488 (5)
Ba1—Te2[ii]	3.5331 (6)	Cd1—Te1[iv]	2.8488 (5)
Ba1—Te2[iii]	3.5413 (6)	Te1—Cd1[iii]	2.8488 (5)
Ba1—Te2[iv]	3.5413 (6)	Te1—Cd1[iv]	2.8488 (5)
Ba1—Te3[i]	3.6287 (6)	Te1—Ba2[i]	3.5459 (6)
Ba1—Te3[ii]	3.6287 (6)	Te1—Ba2[ii]	3.5459 (6)

Ba1—Te1	3.6722 (8)	Te1—Ba2^{vii}	3.6796 (8)
Ba2—Te3^v	3.4297 (6)	Te2—Ba1^{vi}	3.5331 (6)
Ba2—Te3^{vi}	3.4297 (6)	Te2—Ba1^v	3.5331 (6)
Ba2—Te2	3.5213 (8)	Te2—Ba1ⁱⁱⁱ	3.5413 (6)
Ba2—Te1^{vi}	3.5459 (6)	Te2—Ba1^{iv}	3.5413 (6)
Ba2—Te1^v	3.5459 (6)	Te3—Ba2ⁱ	3.4297 (6)
Ba2—Te3^{vii}	3.5913 (8)	Te3—Ba2ⁱⁱ	3.4297 (6)
Ba2—Te1^{viii}	3.6796 (8)	Te3—Ba2^{viii}	3.5913 (8)
Cd1—Te3	2.8050 (9)	Te3—Ba1^v	3.6287 (6)
Cd1—Te2	2.8133 (9)	Te3—Ba1^{vi}	3.6287 (6)
Te2ⁱ—Ba1—Te2ⁱⁱ	84.480 (18)	Te1ⁱⁱⁱ—Cd1—Te1^{iv}	112.97 (3)
Te2ⁱ—Ba1—Te2ⁱⁱⁱ	156.629 (18)	Cd1ⁱⁱⁱ—Te1—Cd1^{iv}	112.97 (3)
Te2ⁱⁱ—Ba1—Te2ⁱⁱⁱ	90.930 (5)	Cd1ⁱⁱⁱ—Te1—Ba2ⁱ	165.46 (2)
Te2ⁱ—Ba1—Te2^{iv}	90.930 (5)	Cd1^{iv}—Te1—Ba2ⁱ	81.436 (14)
Te2ⁱⁱ—Ba1—Te2^{iv}	156.629 (18)	Cd1ⁱⁱⁱ—Te1—Ba2ⁱⁱ	81.436 (14)
Te2ⁱⁱⁱ—Ba1—Te2^{iv}	84.241 (18)	Cd1^{iv}—Te1—Ba2ⁱⁱ	165.46 (2)
Te2ⁱ—Ba1—Te3ⁱ	75.741 (13)	Ba2ⁱ—Te1—Ba2ⁱⁱ	84.104 (18)
Te2ⁱⁱ—Ba1—Te3ⁱ	129.32 (2)	Cd1ⁱⁱⁱ—Te1—Ba1	80.038 (19)
Te2ⁱⁱⁱ—Ba1—Te3ⁱ	123.41 (2)	Cd1^{iv}—Te1—Ba1	80.038 (19)
Te2^{iv}—Ba1—Te3ⁱ	70.888 (14)	Ba2ⁱ—Te1—Ba1	101.413 (18)
Te2ⁱ—Ba1—Te3ⁱⁱ	129.32 (2)	Ba2ⁱⁱ—Te1—Ba1	101.413 (18)
Te2ⁱⁱ—Ba1—Te3ⁱⁱ	75.741 (13)	Cd1ⁱⁱⁱ—Te1—Ba2^{vii}	80.46 (2)
Te2ⁱⁱⁱ—Ba1—Te3ⁱⁱ	70.888 (14)	Cd1^{iv}—Te1—Ba2^{vii}	80.46 (2)
Te2^{iv}—Ba1—Te3ⁱⁱ	123.41 (2)	Ba2ⁱ—Te1—Ba2^{vii}	104.904 (17)
Te3ⁱ—Ba1—Te3ⁱⁱ	81.769 (17)	Ba2ⁱⁱ—Te1—Ba2^{vii}	104.904 (17)
Te2ⁱ—Ba1—Te1	76.026 (15)	Ba1—Te1—Ba2^{vii}	144.28 (2)
Te2ⁱⁱ—Ba1—Te1	76.026 (15)	Cd1—Te2—Ba2	175.65 (3)
Te2ⁱⁱⁱ—Ba1—Te1	80.624 (16)	Cd1—Te2—Ba1^{vi}	78.412 (18)
Te2^{iv}—Ba1—Te1	80.624 (16)	Ba2—Te2—Ba1^{vi}	104.737 (17)
Te3ⁱ—Ba1—Te1	139.101 (8)	Cd1—Te2—Ba1^v	78.412 (18)
Te3ⁱⁱ—Ba1—Te1	139.101 (8)	Ba2—Te2—Ba1^v	104.737 (17)
Te3^v—Ba2—Te3^{vi}	87.658 (19)	Ba1^{vi}—Te2—Ba1^v	84.480 (18)
Te3^v—Ba2—Te2	123.399 (16)	Cd1—Te2—Ba1ⁱⁱⁱ	82.866 (18)
Te3^{vi}—Ba2—Te2	123.399 (16)	Ba2—Te2—Ba1ⁱⁱⁱ	93.918 (17)
Te3^v—Ba2—Te1^{vi}	155.78 (2)	Ba1^{vi}—Te2—Ba1ⁱⁱⁱ	161.261 (19)
Te3^{vi}—Ba2—Te1^{vi}	89.100 (12)	Ba1^v—Te2—Ba1ⁱⁱⁱ	92.594 (5)
Te2—Ba2—Te1^{vi}	77.814 (17)	Cd1—Te2—Ba1^{iv}	82.866 (18)
Te3^v—Ba2—Te1^v	89.100 (12)	Ba2—Te2—Ba1^{iv}	93.918 (17)
Te3^{vi}—Ba2—Te1^v	155.78 (2)	Ba1^{vi}—Te2—Ba1^{iv}	92.594 (5)
Te2—Ba2—Te1^v	77.814 (17)	Ba1^v—Te2—Ba1^{iv}	161.261 (19)
Te1^{vi}—Ba2—Te1^v	84.104 (18)	Ba1ⁱⁱⁱ—Te2—Ba1^{iv}	84.240 (18)
Te3^v—Ba2—Te3^{vii}	74.447 (16)	Cd1—Te3—Ba2ⁱ	85.679 (19)
Te3^{vi}—Ba2—Te3^{vii}	74.447 (16)	Cd1—Te3—Ba2ⁱⁱ	85.679 (19)
Te2—Ba2—Te3^{vii}	71.552 (17)	Ba2ⁱ—Te3—Ba2ⁱⁱ	87.658 (19)
Te1^{vi}—Ba2—Te3^{vii}	127.482 (15)	Cd1—Te3—Ba2^{viii}	164.18 (3)
Te1^v—Ba2—Te3^{vii}	127.482 (15)	Ba2ⁱ—Te3—Ba2^{viii}	105.553 (16)
Te3^v—Ba2—Te1^{viii}	80.695 (16)	Ba2ⁱⁱ—Te3—Ba2^{viii}	105.553 (16)

Te3^{vi}—Ba2—Te1^{viii}	80.695 (16)	Cd1—Te3—Ba1^{v}	76.852 (18)
Te2—Ba2—Te1^{viii}	143.22 (2)	Ba2^{i}—Te3—Ba1^{v}	162.44 (2)
Te1^{vi}—Ba2—Te1^{viii}	75.096 (17)	Ba2^{ii}—Te3—Ba1^{v}	92.687 (10)
Te1^{v}—Ba2—Te1^{viii}	75.096 (17)	Ba2^{viii}—Te3—Ba1^{v}	91.275 (17)
Te3^{vii}—Ba2—Te1^{viii}	145.23 (2)	Cd1—Te3—Ba1^{vi}	76.852 (18)
Te3—Cd1—Te2	103.01 (3)	Ba2^{i}—Te3—Ba1^{vi}	92.687 (10)
Te3—Cd1—Te1^{iii}	109.13 (2)	Ba2^{ii}—Te3—Ba1^{vi}	162.44 (2)
Te2—Cd1—Te1^{iii}	111.05 (2)	Ba2^{viii}—Te3—Ba1^{vi}	91.275 (17)
Te3—Cd1—Te1^{iv}	109.13 (2)	Ba1^{v}—Te3—Ba1^{vi}	81.769 (17)
Te2—Cd1—Te1^{iv}	111.05 (2)		

Symmetry codes: (i) −x+1/2, −y+1, z+1/2; (ii) −x+1/2, −y, z+1/2; (iii) −x, −y, −z+1; (iv) −x, −y+1, −z+1; (v) −x+1/2, −y, z−1/2; (vi) −x+1/2, −y+1, z−1/2; (vii) x−1/2, y, −z+1/2; (viii) x+1/2, y, −z+1/2.