

Lawrence Berkeley National Laboratory

Recent Work

Title

BIS[BIS(PENTAMETHYLCYCLOPENTADIENYL)YTTERBIUM(III)]DITELLURIDE

Permalink

<https://escholarship.org/uc/item/4v95c2f8>

Authors

Zalkin, A.
Berg, D.J.

Publication Date

1988-03-01



Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

Materials & Chemical Sciences Division

RECEIVED
LAWRENCE
BERKELEY LABORATORY

MAY 10 1988

LIBRARY AND
DOCUMENTS SECTION

Submitted to Acta Crystallographica

**Bis[bis(pentamethylcyclopentadienyl)
ytterbium(III)]ditelluride**

A. Zalkin and D.J. Berg

March 1988

TWO-WEEK LOAN COPY

*This is a Library Circulating Copy
which may be borrowed for two weeks.*



DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

Bis[bis(pentamethylcyclopentadienyl)ytterbium(III)]ditelluride

By Allan Zalkin and David J. Berg

Molecular and Chemical Sciences Division, Lawrence Berkeley Laboratory,
University of California, Berkeley, California 94720 USA

Abstract. $[(\text{Me}_5\text{C}_5)_2\text{Yb}]_2\text{Te}_2$, $\text{C}_{40}\text{H}_{60}\text{Te}_2\text{Yb}_2$, $M_r = 1142.20$, monoclinic, $P2_1/n$, $a = 15.517(3)$ Å, $b = 10.611(2)$ Å, $c = 13.166(3)$ Å, $\beta = 114.34(2)^\circ$, $V = 1975.1$ Å³, $Z = 2$, $D_x = 1.92 \text{ g cm}^{-3}$, $\lambda(\text{MoK}\alpha) = 0.71073$ Å, $\mu = 61.71 \text{ cm}^{-1}$, $F(000) = 1088$, $T = 296^\circ\text{K}$, $R = 0.036$ for $3330 [F^2 > 2\sigma(F^2)]$ of 4560 total unique data. The Te_2^{2-} ion is on a center of symmetry and lies perpendicular to and in between the two Yb atoms. Each Yb atom is bonded approximately tetrahedrally to two cyclopentadienyl rings centers and the Te_2^{2-} ion. Distances (Å) are: ave. Yb-C 2.626(17), ave. Yb-Cp(centroid) 2.332(18), Te-Te 2.7686(11) Å.

Experimental. The complex was isolated from the reaction of $\text{Yb}[\text{C}_5(\text{CH}_3)_5]_2 \cdot \text{O}(\text{C}_2\text{H}_5)_2$ with a large excess of tellurium powder in hexane after stirring for 2 days at room temperature. Black air-sensitive crystals were sealed inside quartz capillaries under argon. Crystal, $0.25 \times 0.25 \times 0.33$ mm with eight faces; modified Picker automatic diffractometer, graphite monochromator; cell dimensions from 28 reflections, $20 < 2\theta < 54^\circ$; analytical absorption correction, range 2.4-3.8; max. $(\sin\theta/\lambda) = 0.65 \text{ \AA}^{-1}$; $h -20$ to 20, $k 0$ to 13, $l -17$ to 17; three standard reflections, average decay 0.8%, intensities adjusted accordingly; 8993 data, 4560 unique, $R_{\text{int}} = 0.023$; structure solved by Patterson and Fourier methods; refined on F , f' and f'' terms included, 199 parameters; hydrogen atoms not observed in difference maps and were not included; anisotropic thermal parameters for all atoms refined; $R = 0.036$ for 3330 reflections for which $F^2 > 2\sigma(F^2)$; $R = 0.055$ all data; $wR = 0.052$; $S = 1.38$; $w = 4F_o/[\sigma^2(F_o^2) + (0.06F_o^2)^2]$; max. $(\text{shift}/\sigma) < 0.05$; extinction correction $F_{\text{corr}} = (1 + 4.8 \times 10^{-8} I)F_o$, extinction parameter estimated after each series of refinements, max correction 11%; max. and min. of ΔF synthesis 1.6 and -1.4 e \AA^{-3} ; atomic f , f' and f'' for neutral Yb, Te and C from International Tables for X-ray Crystallography (1974); local unpublished programs and ORTEP (Johnson, 1965).

Atomic parameters are listed in Table 1, and distances and angles are listed in Table 2.* Fig. 1 shows the molecule and numbering scheme.

* Lists of structure factors, anisotropic thermal parameters, additional distances and angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. (13 pp). Copies may be obtained through The Executive Secretary International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related Literature. Comparable distance for Te-Te is 2.78 \AA in AuTe_2X , $\text{X} = \text{Cl}$ or I (Haendler, Mootz, Rabenau & Rosenstein, 1974) and $2.802(1) \text{ \AA}$ in

[$\{(\text{CH}_3\text{C}(\text{CH}_2\text{P}(\text{C}_6\text{H}_5)_2)_3\text{Ni}\}_2[\mu\text{-Te}_2]\cdot 2\text{C}_4\text{H}_8\text{O}$] (Di Vaira, Peruzzini & Stoppioni, 1986). The latter is the only structurally characterized compound with the same geometry for the Te_2 unit as that found in the present work. A comparable distance for Yb-Cp is 2.347(2) Å in $(\text{Me}_5\text{C}_5)_2\text{Yb}(\text{C}_6\text{H}_5\text{S})(\text{NH}_3)$ (Zalkin, Henly & Andersen, 1987).

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the US Department of Energy under Contract No. DE-AC03-76SF00098.

References

- Di Vaira, M., Peruzzini, M., Stoppioni, P. (1986) J. Chem. Soc. Chem. Comm., 1986, 374.
- Haendler, H. M., Mootz, D., Rabenau, A. & Rosenstein, G. (1974) J. Solid State Chem., 10, 175-181.
- International Tables for X-ray Crystallography (1974). Vol. IV, Table 2.2, pp. 71-102. Birmingham: Kynoch Press. (present distributor D. Reidel, Dordrecht.)
- Johnson, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- Zalkin, A., Henly, T.J. & Andersen, R. A. (1987). Acta Cryst. C43, 233-236.

Table 1. Atomic Parameters

$$B_{eq} = \sum B_{ij} a_i^* a_j^* a_i \cdot a_j / 3$$

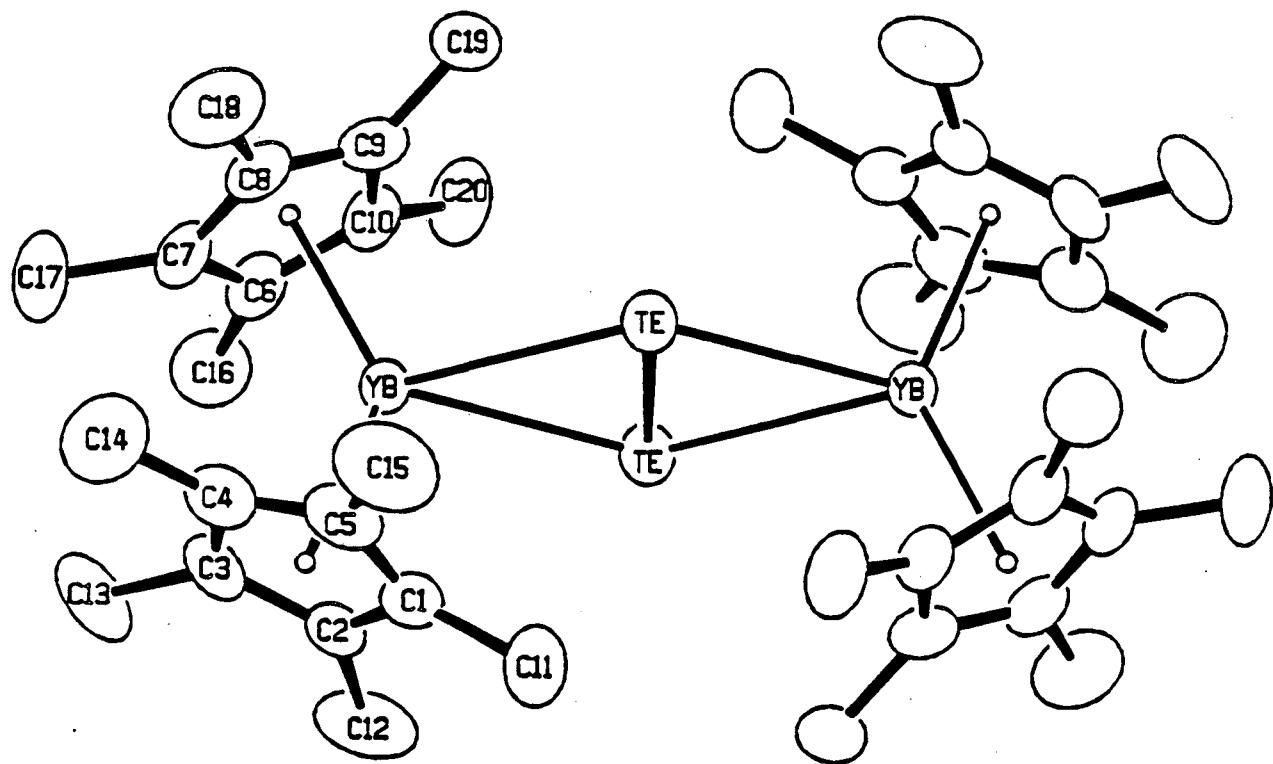
Atom	x	y	z	B _{eq}
Yb	0.16540(2)	0.07854(3)	-0.02377(2)	2.656(9)
Te	0.05403(3)	-0.07689(4)	0.08828(4)	3.43(2)
C1	0.1697(6)	0.2522(7)	0.1192(7)	3.7(2)
C2	0.1706(5)	0.3167(6)	0.0264(7)	3.6(2)
C3	0.2584(6)	0.2928(7)	0.0192(7)	4.1(3)
C4	0.3112(6)	0.2094(7)	0.1103(8)	4.4(3)
C5	0.2554(6)	0.1844(7)	0.1702(7)	4.2(3)
C6	0.1835(5)	0.0476(7)	-0.2110(6)	3.5(2)
C7	0.2764(5)	0.0245(8)	-0.1270(6)	3.5(2)
C8	0.2717(6)	-0.0846(7)	-0.0692(7)	3.7(2)
C9	0.1786(6)	-0.1345(7)	-0.1208(7)	3.8(3)
C10	0.1232(5)	-0.0508(7)	-0.2093(6)	3.5(2)
C11	0.0950(8)	0.2726(10)	0.1658(10)	6.3(4)
C12	0.0970(9)	0.4095(8)	-0.0467(10)	6.7(4)
C13	0.2962(10)	0.3655(10)	-0.0528(12)	7.8(6)
C14	0.4163(7)	0.1799(12)	0.1501(12)	8.1(5)
C15	0.2877(10)	0.1090(11)	0.2775(9)	7.0(5)
C16	0.1544(7)	0.1523(9)	-0.2991(8)	5.1(3)
C17	0.3649(7)	0.0846(9)	-0.1232(9)	5.5(4)
C18	0.3546(7)	-0.1478(10)	0.0237(7)	5.5(3)
C19	0.1504(7)	-0.2631(7)	-0.0948(9)	5.1(4)
C20	0.0263(6)	-0.0778(9)	-0.2988(7)	4.8(3)

Table 2. Selected Distances (\AA) and Angles ($^\circ$)

Cp1 - Yb	2.319	Te - Yb - Cp1	107.67
Cp2 - Yb	2.344	Te - Yb - Cp2	113.83
C1 - Yb	2.616(7)	Te - Yb - Cp1	109.31
C2 - Yb	2.605(7)	Te - Yb - Cp2	112.59
C3 - Yb	2.627(7)	Cp1 - Yb - Cp2	133.32
C4 - Yb	2.621(8)	Te - Yb - Te	52.04(2)
C5 - Yb	2.607(8)	Yb - Te - Yb	127.96(2)
C6 - Yb	2.614(7)	Yb - Te - Te	63.82(2)
C7 - Yb	2.659(7)	Yb - Te - Te	64.14(2)
C8 - Yb	2.625(7)		
C9 - Yb	2.646(7)		
C10 - Yb	2.638(7)		
Te - Yb	3.1513(9)		
Te - Yb	3.1598(7)		
Te - Te	2.7686(11)		

Cp1 and Cp2 represent the centroids of cyclopentadienyl atoms C1-C5 and C6-C(10) respectively.

Fig 1. ORTEP (Johnson, 1965) drawing of molecule; 50% probability ellipsoids.



Supplemental Materials

Bis[bis(pentamethylcyclopentadienyl)ytterbium(III)]ditelluride

By Allan Zalkin and David J. Berg

Molecular and Chemical Sciences Division, Lawrence Berkeley Laboratory,
University of California, Berkeley, California 94720 USA

Abstract. $[(\text{Me}_5\text{C}_5)_2\text{Yb}]_2\text{Te}_2$, $\text{C}_{40}\text{H}_{60}\text{Te}_2\text{Yb}_2$, $M_r = 1142.20$, monoclinic, $P2_1/n$, $a = 15.517(3)$ Å, $b = 10.611(2)$ Å, $c = 13.166(3)$ Å, $\beta = 114.34(2)^\circ$, $V = 1975.1$ Å³, $Z = 2$, $D_x = 1.92$ g cm⁻³, $\lambda(\text{MoK}\alpha) = 0.71073$ Å, $\mu = 61.71$ cm⁻¹, $F(000) = 1088$, $T = 296^\circ\text{K}$, $R = 0.036$ for $3330 [F^2 > 2\sigma(F^2)]$ of 4560 total unique data. The Te_2^{2-} ion is on a center of symmetry and lies perpendicular to and in between the two Yb atoms. Each Yb atom is bonded approximately tetrahedrally to two cyclopentadienyl rings centers and the Te_2^{2-} ion. Distances (Å) are: ave. Yb-C 2.626(17), ave. Yb-Cp(centroid) 2.332(18), Te-Te 2.7686(11) Å.

Supplemental Table 1. Anisotropic Thermal Parameters^a

Atom	B11	B22	B33	B12	B13	B23
Yb	2.861(13)	2.530(13)	3.053(14)	-0.253(10)	1.696(10)	-0.104(10)
Te	3.601(22)	3.206(20)	4.014(23)	-0.047(16)	2.112(18)	0.344(16)
C1	4.4(3)	2.99(28)	4.3(3)	-0.57(26)	2.27(29)	-1.19(26)
C2	3.9(3)	2.28(25)	4.8(4)	-0.53(23)	2.2(3)	-0.32(25)
C3	4.6(4)	3.2(3)	4.9(4)	-1.86(28)	2.4(3)	-1.09(28)
C4	3.6(3)	3.6(3)	5.3(4)	-0.74(27)	1.2(3)	-1.1(3)
C5	5.4(4)	3.4(3)	3.3(3)	-0.9(3)	1.1(3)	-0.67(26)
C6	3.9(3)	4.4(3)	3.07(29)	0.44(26)	2.26(27)	0.29(25)
C7	3.6(3)	4.4(3)	3.7(3)	0.23(27)	2.56(27)	-0.24(27)
C8	3.9(3)	3.9(3)	4.0(3)	0.85(27)	2.44(28)	-0.13(27)
C9	5.5(4)	2.92(28)	4.2(4)	0.69(27)	3.1(3)	-0.20(25)
C10	3.4(3)	4.2(3)	3.3(3)	0.00(25)	1.95(26)	-0.54(24)
C11	6.9(6)	6.8(5)	7.9(6)	-2.8(5)	5.5(5)	-3.6(5)
C12	7.2(6)	3.2(4)	7.4(6)	0.9(4)	0.7(5)	0.9(4)
C13	11.8(9)	5.1(5)	10.3(9)	-3.8(6)	8.3(8)	-0.8(5)
C14	2.8(4)	8.0(7)	11.5(9)	-0.2(4)	1.0(5)	-2.7(6)
C15	9.8(8)	6.2(5)	4.0(4)	0.0(5)	1.7(5)	1.1(4)
C16	6.1(5)	4.8(4)	4.8(4)	1.0(4)	2.5(4)	1.7(3)
C17	4.4(4)	7.5(6)	6.2(5)	-1.5(4)	3.7(4)	-0.9(4)
C18	5.7(5)	6.3(5)	3.7(4)	2.1(4)	1.1(4)	0.7(4)
C19	7.4(5)	2.9(3)	7.3(5)	-0.2(3)	5.1(5)	-0.3(3)
C20	3.6(3)	7.3(5)	3.8(3)	-0.5(3)	1.64(29)	-0.9(4)

^a The isotropic temperature factor has the form

$$T = -\sum(h_i h_j B_{ij} a_i^* a_j^*)/4 \text{ in units of } \text{\AA}^2.$$

Supplemental Table 2. Additional Angles in $[(\text{CH}_3)_5\text{C}_5]_2\text{YbTe}_2\text{Yb}[\text{C}_5(\text{CH}_3)_5]_2$

C2 -C1	1.406(11)	C2 -C1-C5	108.5(7)	C7 -C6 -C10	109.0(7)
C5 -C1	1.415(12)	C2 -C1-C11	123.9(8)	C7 -C6 -C16	126.7(7)
C11-C1	1.535(11)	C5 -C1-C11	126.8(8)	C10-C6 -C16	123.9(7)
C3 -C2	1.427(11)	C1 -C2-C3	108.8(7)	C6 -C7 -C8	107.4(7)
C12-C2	1.514(12)	C1 -C2-C12	126.5(8)	C6 -C7 -C17	124.8(8)
C4 -C3	1.443(12)	C3 -C2-C12	124.3(8)	C8 -C7 -C17	125.8(8)
C13-C3	1.515(12)	C2 -C3-C4	106.5(7)	C7 -C8 -C9	108.5(7)
C5 -C4	1.416(12)	C2 -C3-C13	125.3(9)	C7 -C8 -C18	125.8(8)
C14-C4	1.526(12)	C4 -C3-C13	126.8(9)	C9 -C8 -C18	125.3(8)
C15-C5	1.517(12)	C3 -C4-C5	108.1(8)	C8 -C9 -C10	107.9(7)
C10-C6	1.408(10)	C3 -C4-C14	124.8(9)	C8 -C9 -C19	124.0(8)
C16-C6	1.534(11)	C5 -C4-C14	125.6(10)	C10-C9 -C19	127.5(8)
C8 -C7	1.404(10)	C1 -C5-C4	108.1(7)	C6 -C10-C9	107.0(7)
C17-C7	1.496(11)	C1 -C5-C15	126.5(9)	C6 -C10-C20	125.8(7)
C9 -C8	1.422(12)	C4 -C5-C15	125.1(9)	C9 -C10-C20	126.0(8)
C18-C8	1.517(11)				
C10-C9	1.436(11)				
C19-C9	1.515(11)				
C20-C10	1.506(11)				

Least-Squares Planes

Plane No. 1

Equation of the Plane: $0.18590 a + 0.78983 b + 0.45991 c = 3.31170$

Distance to the plane from

atoms in plane			atoms not in plane		
Atom	$d(\text{\AA})$	$\sigma(d)$	Atom	$d(\text{\AA})$	$\sigma(d)$
C1	0.008	0.011	Yb	-2.319	0.004
C2	-0.007	0.012	C11	0.243	0.019
C3	0.003	0.012	C12	0.120	0.020
C4	0.003	0.013	C13	0.289	0.021
C5	-0.008	0.013	C14	0.298	0.022
			C15	0.097	0.021

Plane No. 2

Equation of the Plane: $-0.55554 a + 0.53294 b + 0.81048 c = -3.57876$

Distance to the plane from

atoms in plane			atoms not in plane		
Atom	$d(\text{\AA})$	$\sigma(d)$	Atom	$d(\text{\AA})$	$\sigma(d)$
C6	0.015	0.011	Yb	2.344	0.004
C7	-0.020	0.011	C16	-0.083	0.017
C8	0.020	0.011	C17	-0.403	0.017
C9	-0.011	0.012	C18	-0.062	0.017
C10	-0.004	0.010	C19	-0.218	0.018
			C20	-0.276	0.016

Angle between the planes = 46.3(4)

STRUCTURE AMPLITUDES continued for (MESCS)2YB TE YB(MESCS)2

page 9

H FOB SG DEL	H FOB SG DEL	H FOB SG DEL	H FOB SG DEL	H FOB SG DEL	H FOB SG DEL	H FOB SG DEL	H FOB SG DEL
2 17 20 9*							
K,L= 13, 5							
-5 5 21 -7*							
-4 11 22 -11*							
-3 0 19 -5*							
-2 111 4 0							
-1 18 19 2*							
0 16 19 -9*							

*LAWRENCE BERKELEY LABORATORY
TECHNICAL INFORMATION DEPARTMENT
UNIVERSITY OF CALIFORNIA
BERKELEY, CALIFORNIA 94720*