

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

# $(\eta^4\text{-Cyclooctatetraene})(\eta^8\text{-cyclooctatetraene})\text{iodidotantalum(V)}$

Pratik Verma, Victor J. Sussman, William W. Brennessel\* and John E. Ellis

Department of Chemistry, 207 Pleasant Street SE, University of Minnesota, Minneapolis, MN 55455, USA

Correspondence e-mail: [brennessel@chem.rochester.edu](mailto:brennessel@chem.rochester.edu)

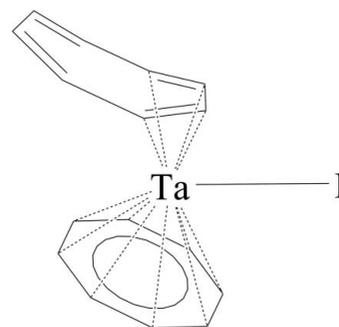
Received 15 May 2014; accepted 27 May 2014

Key indicators: single-crystal X-ray study;  $T = 173\text{ K}$ ; mean  $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$ ; disorder in main residue;  $R$  factor = 0.017;  $wR$  factor = 0.040; data-to-parameter ratio = 16.9.

The title complex,  $[\text{Ta}(\eta^4\text{-C}_8\text{H}_8)(\eta^8\text{-C}_8\text{H}_8)\text{I}]$ , lies across a crystallographic mirror plane that includes the  $\text{Ta}^{\text{V}}$  atom and the iodide ligand. One cyclooctatetraene (cot) ring is  $\eta^4$ -coordinating and is bisected by the mirror plane. The fold angle between the plane of the coordinating butadiene portion and the middle plane of the ring is  $27.4(4)^\circ$ . An additional minor fold angle of  $9.3(7)^\circ$  exists between the final plane in the ring and the middle plane. The other cot ring is  $\eta^8$ -coordinating and is also cut by the mirror plane. In this case, the ring is disordered over the mirror plane, and one position is modeled with appropriate restraints and constraints with respect to distances, angles and displacement parameters (the second position is generated by symmetry). This ring is nearly planar, with an r.m.s. deviation of only  $0.05\text{ \AA}$  when all eight C atoms are included in the calculation. Pairs of intermolecular  $\eta^8$ -cot rings are parallel stacked and slightly off center, with a centroid-centroid distance of  $3.652\text{ \AA}$ . No other significant intermolecular interactions are observed. The compound is of interest as the first structurally characterized mixed halogen-cot complex of the group 5 metals and contains the longest terminal Ta—I distance [ $3.0107(5)\text{ \AA}$ ] reported to date.

## Related literature

For synthesis of the precursor tris(naphthalene)tantalate, see: Brennessel *et al.* (2002). For related  $\text{MX}(\text{cot})_2$ ,  $M = \text{Nb, Ta}$ ,  $X = \text{Cl, Me, Ph}$ , see: Schrock *et al.* (1976). For the only other structurally characterized  $\eta^8$ -coordinated cyclooctatetraene-tantalum species to date, ( $\eta$ -1,4-bis(trimethylsilyl)cot) $\text{Me}_3\text{Ta}$ , see: Clegg & McCamley (2005). For the compound containing the previous longest terminal Ta—I distance, see: Berneri *et al.* (1998). For  $\text{Zr}(\text{cot})_2$ , which also contains both  $\eta^4$ -cot and  $\eta^8$ -cot units, see: Cloke *et al.* (1994). For a description of the Cambridge Structural Database, see: Allen (2002).



## Experimental

### Crystal data

$[\text{Ta}(\text{C}_8\text{H}_8)_2\text{I}]$	$V = 1356.4(2)\text{ \AA}^3$
$M_r = 516.14$	$Z = 4$
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
$a = 14.3626(14)\text{ \AA}$	$\mu = 10.36\text{ mm}^{-1}$
$b = 11.0200(11)\text{ \AA}$	$T = 173\text{ K}$
$c = 9.3467(9)\text{ \AA}$	$0.10 \times 0.10 \times 0.10\text{ mm}$
$\beta = 113.522(2)^\circ$	

### Data collection

Siemens SMART CCD platform diffractometer	8110 measured reflections
Absorption correction: multi-scan ( <i>SADABS</i> ; Sheldrick, 2012)	1636 independent reflections
$T_{\text{min}} = 0.318$ , $T_{\text{max}} = 0.431$	1520 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.020$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$	96 restraints
$wR(F^2) = 0.040$	H-atom parameters constrained
$S = 1.08$	$\Delta\rho_{\text{max}} = 1.04\text{ e \AA}^{-3}$
1636 reflections	$\Delta\rho_{\text{min}} = -0.72\text{ e \AA}^{-3}$
97 parameters	

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This research was supported by the US National Science Foundation and the donors of the Petroleum Research Fund, administered by the American Chemical Society. PV thanks the University of Minnesota for an undergraduate research opportunity program (UROP) award.

Supporting information for this paper is available from the IUCr electronic archives (Reference: LH5707).

## References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.  
 Berneri, P., Calderazzo, F., Englert, U. & Pampaloni, G. (1998). *J. Organomet. Chem.* **562**, 61–69.  
 Brennessel, W. W., Ellis, J. E., Pomije, M. K., Sussman, V. J., Urnezis, E. & Young, V. G. Jr (2002). *J. Am. Chem. Soc.* **124**, 10258–10259.  
 Bruker (2003). *SAINT* and *SMART*. Bruker AXS, Inc., Madison, Wisconsin, USA.

## metal-organic compounds

---

- Clegg, W. & McCamley, A. (2005). Private communication (refcode: SAWWAP). CCDC, Cambridge, England.
- Cloke, F. G. N., Green, J. C., Hitchcock, P. B., Joseph, S. C. P., Mountford, P., Kaltsoyannis, N. & McCamley, A. (1994). *J. Chem. Soc. Dalton Trans.* pp. 2867–2874.
- Schrock, R. R., Guggenberger, L. J. & English, A. D. (1976). *J. Am. Chem. Soc.* **98**, 903–913.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2012). *SADABS*. University of Göttingen, Germany.

## supporting information

*Acta Cryst.* (2014). E70, m245–m246 [https://doi.org/10.1107/S1600536814012379]

**( $\eta^4$ -Cyclooctatetraene)( $\eta^8$ -cyclooctatetraene)iodidotantalum(V)**

**Pratik Verma, Victor J. Sussman, William W. Brennessel and John E. Ellis**

**S1. Comment**

Bis(cyclooctatetraene)iodidotantalum, TaI(cot)<sub>2</sub>, is the first structurally authenticated mixed halogen-cot complex of a group 5 metal (Fig. 1). In prior work, reaction of MCl<sub>5</sub>, M = Nb, Ta, with two equivalents of K<sub>2</sub>[cot] was suggested to afford the chloro analogs, MCl(cot)<sub>2</sub> (Schrock *et al.*, 1976). Although these compounds were not isolated or fully characterized in solution, the <sup>1</sup>H NMR spectrum of the niobium complex in CD<sub>2</sub>Cl<sub>2</sub> at 273 K showed two singlets in a 1:1 ratio at  $\delta$  5.49 and 6.24 p.p.m., in excellent agreement with those observed for TaI(cot)<sub>2</sub> in THF-*d*<sub>8</sub>, see below. Thus, our study provides additional support for the existence of NbCl(cot)<sub>2</sub> and TaCl(cot)<sub>2</sub> and suggests that the latter contain  $\eta^4$ - and  $\eta^8$ -cot groups, as observed in TaI(cot)<sub>2</sub>. Structural features of the  $\eta^4$ -cot group in TaI(cot)<sub>2</sub> are nearly identical to the one present in [Ta(cot)<sub>3</sub>]<sup>-</sup> (Brennessel *et al.*, 2002). For example, the  $\eta^4$ -cot-(centroid)-Ta distances in these two species are 2.023 and 2.025 Å, respectively. The only other structurally characterized  $\eta^8$ -cot-Nb or -Ta complex for comparison is TaMe<sub>3</sub>(cot") (Clegg & McCamley, 2005), where cot" = 1,4-bis(trimethylsilyl)cyclooctatetraene, in which the  $\eta^8$ -cot"-(centroid)-Ta distance, 1.627 Å, is slightly longer than the corresponding distance in TaI(cot)<sub>2</sub>, 1.606 Å, likely owing to the more bulky nature of cot" *versus* cot. The only surprising feature in the structure of TaI(cot)<sub>2</sub> is the unusually long Ta–I distance of 3.0108 (5) Å, which exceeds the prior "record" value of 2.9621 (7) Å, observed in the 7-coordinate complex, TaI(CO)<sub>4</sub>(tmen), tmen = 1,2-bis(dimethylamino)ethane (Berneri *et al.*, 1998). Because the  $\eta^4$  and  $\eta^8$  units in the formally Zr(IV) complex, Zr(cot)<sub>2</sub> (Cloke *et al.*, 1994) and TaI(cot)<sub>2</sub> are quite analogous in structure and binding to the respective metals, the tantalum complex is best formulated to contain Ta(V).

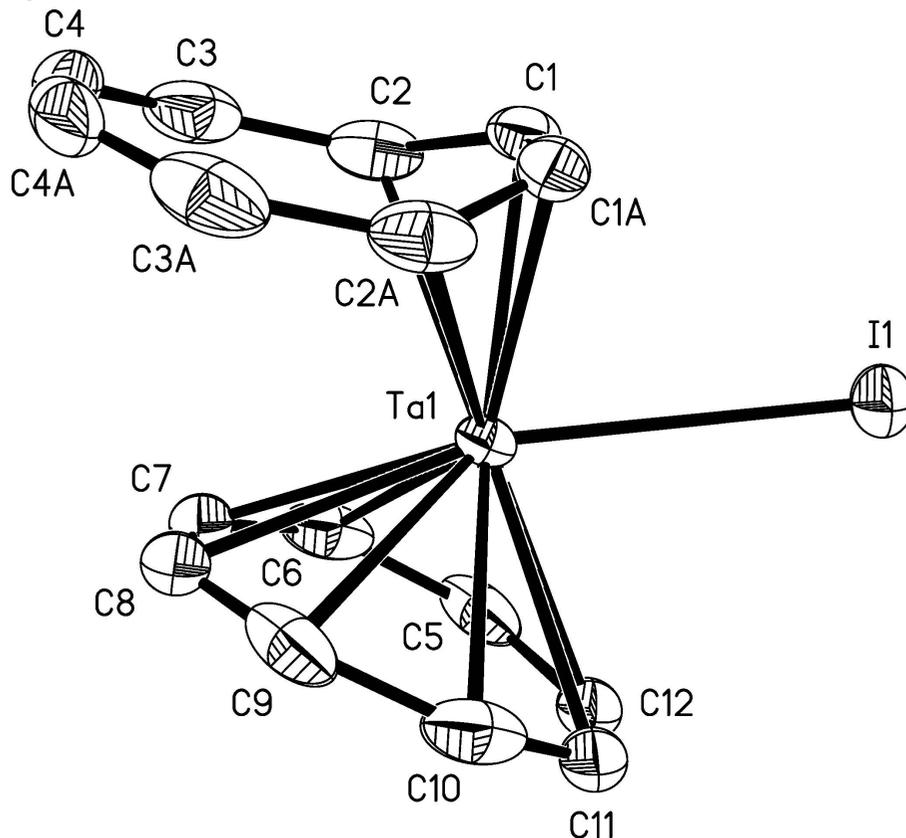
**S2. Experimental**

Under argon, a yellow-orange solution of [Na(THF)<sub>6</sub>][Ta( $\eta^4$ -C<sub>10</sub>H<sub>8</sub>)<sub>3</sub>] (Brennessel *et al.*, 2002) in tetrahydrofuran (THF) was combined with three equiv. of 1,3,5,7-cyclooctatetraene (cot) at room temperature and stirred for 24 h to provide a deep purple solution of [Na(THF)<sub>6</sub>][Ta(cot)<sub>3</sub>]. Cation exchange of the latter (Brennessel *et al.*, 2002) with bis(triphenylphosphine)iminium chloride, PPN<sup>+</sup>Cl<sup>-</sup>, in ethanol under argon, provided a purple-brown precipitate of [PPN][Ta(cot)<sub>3</sub>] in 67% yield. The latter was reacted with one equiv. of elemental iodine in THF at 205 K, followed by warming to room temperature over a four hour period. Following filtration to remove poorly soluble PPN<sup>+</sup>I<sup>-</sup>, a deep red-purple solid of composition TaI(cot)<sub>2</sub> was isolated in 49% yield. <sup>1</sup>H NMR (300 MHz, 298 K, THF-*d*<sub>8</sub>,  $\delta$ , p.p.m.) 5.53 (s, 8H, cot), 6.27 (s, 8H, cot). Suitable single crystals of the product were grown from a pentane-layered THF solution over a period of several days at 273 K.

**S3. Refinement**

The  $\eta^8$ -cot ligand is modeled as disordered over a crystallographic mirror plane (50:50). Bond lengths and angles in the  $\eta^8$ -cot ligand were restrained to be similar to those of their neighbors. Anisotropic displacement parameters for pairs of atoms opposite to each other were constrained to be equivalent, and those of symmetry-related atom pair C11 and C12

were heavily restrained to be similar. H atoms were placed geometrically and treated as riding atoms: C—H = 0.95 Å with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .



**Figure 1**

The structure of the molecule without hydrogen atoms, showing the atom numbering. Only one of the two positions of the disordered ligand is shown. Displacement ellipsoids are drawn at the 50% probability level.

**( $\eta^4$ -Cyclooctatetraene)( $\eta^8$ -cyclooctatetraene)iodidotantalum(V)**

*Crystal data*

[Ta(C<sub>8</sub>H<sub>8</sub>)<sub>2</sub>I]

$M_r = 516.14$

Monoclinic,  $C2/m$

$a = 14.3626$  (14) Å

$b = 11.0200$  (11) Å

$c = 9.3467$  (9) Å

$\beta = 113.522$  (2)°

$V = 1356.4$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 952$

$D_x = 2.527$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 937 reflections

$\theta = 2.4$ – $27.5$ °

$\mu = 10.36$  mm<sup>-1</sup>

$T = 173$  K

Block, red-purple

$0.10 \times 0.10 \times 0.10$  mm

*Data collection*

Siemens SMART CCD platform  
diffractometer

Radiation source: normal-focus sealed tube

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 2012)

$T_{\text{min}} = 0.318$ ,  $T_{\text{max}} = 0.431$

8110 measured reflections

1636 independent reflections

1520 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\text{max}} = 27.5$ °,  $\theta_{\text{min}} = 2.4$ °

$h = -18 \rightarrow 18$   
 $k = -14 \rightarrow 14$

$l = -12 \rightarrow 12$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.017$   
 $wR(F^2) = 0.040$   
 $S = 1.08$   
 1636 reflections  
 97 parameters  
 96 restraints  
 Primary atom site location: structure-invariant  
 direct methods

Secondary atom site location: difference Fourier  
 map  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.018P)^2 + 5.3173P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.003$   
 $\Delta\rho_{\max} = 1.04 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.72 \text{ e } \text{\AA}^{-3}$

*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.** The  $\eta^8$ -cot ligand is modeled as disordered over a crystallographic mirror plane (50:50). Bond lengths and angles in the  $\eta^8$ -cot ligand were restrained to be similar to those of their neighbors. Anisotropic displacement parameters for pairs of atoms opposite to each other were constrained to be equivalent, and those of symmetry-related atom pair C11 and C12 were heavily restrained to be similar.

H atoms were placed geometrically and treated as riding atoms: C—H = 0.95 Å with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ta1	0.28337 (2)	0.5000	0.16766 (2)	0.01915 (6)	
I1	0.26793 (2)	0.5000	-0.16320 (4)	0.02953 (8)	
C1	0.1203 (2)	0.4365 (4)	0.0278 (4)	0.0312 (8)	
H1	0.1043	0.4003	-0.0715	0.037*	
C2	0.1398 (3)	0.3549 (3)	0.1493 (5)	0.0356 (9)	
H2	0.1770	0.2859	0.1413	0.043*	
C3	0.1160 (3)	0.3522 (4)	0.2844 (5)	0.0455 (11)	
H3	0.1214	0.2729	0.3271	0.055*	
C4	0.0875 (3)	0.4358 (5)	0.3660 (5)	0.0531 (13)	
H4	0.0626	0.4012	0.4371	0.064*	
C5	0.4245 (10)	0.3628 (8)	0.2362 (15)	0.039 (3)	0.5
H5	0.4396	0.2896	0.1962	0.047*	0.5
C6	0.3753 (8)	0.3428 (9)	0.3361 (12)	0.038 (4)	0.5
H6	0.3574	0.2602	0.3398	0.046*	0.5
C7	0.3473 (7)	0.4163 (9)	0.4297 (10)	0.042 (2)	0.5
H7	0.3298	0.3703	0.5013	0.050*	0.5
C8	0.3375 (6)	0.5382 (8)	0.4486 (9)	0.042 (3)	0.5
H8	0.3127	0.5560	0.5266	0.051*	0.5
C9	0.3558 (12)	0.6407 (8)	0.3790 (17)	0.039 (3)	0.5
H9	0.3318	0.7123	0.4097	0.047*	0.5
C10	0.4007 (7)	0.6649 (8)	0.2747 (12)	0.038 (4)	0.5
H10	0.4011	0.7488	0.2518	0.046*	0.5

C11	0.4453 (7)	0.5925 (8)	0.1965 (11)	0.042 (2)	0.5
H11	0.4749	0.6393	0.1400	0.050*	0.5
C12	0.4570 (5)	0.4667 (6)	0.1811 (8)	0.042 (3)	0.5
H12	0.4949	0.4481	0.1208	0.051*	0.5

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ta1	0.01532 (9)	0.02046 (10)	0.01810 (10)	0.000	0.00291 (7)	0.000
I1	0.03124 (17)	0.03438 (18)	0.02409 (16)	0.000	0.01224 (13)	0.000
C1	0.0182 (15)	0.045 (2)	0.0268 (17)	-0.0062 (15)	0.0053 (13)	-0.0106 (16)
C2	0.0215 (17)	0.0272 (18)	0.049 (2)	-0.0006 (14)	0.0037 (16)	-0.0047 (16)
C3	0.0277 (19)	0.047 (2)	0.047 (2)	-0.0136 (18)	-0.0006 (18)	0.020 (2)
C4	0.0250 (18)	0.105 (4)	0.027 (2)	-0.011 (2)	0.0080 (16)	0.017 (2)
C5	0.024 (6)	0.055 (6)	0.029 (6)	0.012 (5)	0.001 (3)	-0.019 (5)
C6	0.027 (9)	0.027 (3)	0.045 (13)	-0.007 (3)	-0.002 (5)	0.008 (3)
C7	0.0205 (16)	0.079 (8)	0.0247 (18)	-0.008 (2)	0.0078 (14)	0.001 (2)
C8	0.0210 (15)	0.079 (8)	0.0248 (17)	-0.008 (2)	0.0071 (13)	0.000 (2)
C9	0.024 (6)	0.055 (6)	0.029 (6)	0.012 (5)	0.001 (3)	-0.019 (5)
C10	0.027 (9)	0.027 (3)	0.045 (13)	-0.007 (3)	-0.002 (5)	0.008 (3)
C11	0.0205 (16)	0.079 (8)	0.0247 (18)	-0.008 (2)	0.0078 (14)	0.001 (2)
C12	0.0210 (15)	0.079 (8)	0.0248 (17)	-0.008 (2)	0.0071 (13)	0.000 (2)

*Geometric parameters (Å, °)*

Ta1—C1	2.290 (3)	C3—H3	0.9500
Ta1—C1 <sup>i</sup>	2.290 (3)	C4—C4 <sup>i</sup>	1.416 (11)
Ta1—C6	2.359 (10)	C4—H4	0.9500
Ta1—C9	2.397 (13)	C5—C6	1.396 (9)
Ta1—C5	2.402 (12)	C5—C12	1.410 (9)
Ta1—C10	2.408 (9)	C5—H5	0.9500
Ta1—C7	2.429 (9)	C6—C7	1.365 (9)
Ta1—C11	2.454 (9)	C6—H6	0.9500
Ta1—C8	2.459 (8)	C7—C8	1.369 (9)
Ta1—C12	2.474 (7)	C7—H7	0.9500
Ta1—C2	2.560 (4)	C8—C9	1.381 (9)
Ta1—C2 <sup>i</sup>	2.560 (4)	C8—H8	0.9500
Ta1—I1	3.0107 (5)	C9—C10	1.392 (9)
C1—C2	1.386 (5)	C9—H9	0.9500
C1—C1 <sup>i</sup>	1.400 (8)	C10—C11	1.399 (9)
C1—H1	0.9500	C10—H10	0.9500
C2—C3	1.435 (6)	C11—C12	1.410 (9)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.359 (7)	C12—H12	0.9500
C1—Ta1—C1 <sup>i</sup>	35.6 (2)	C7—Ta1—I1	151.54 (19)
C1—Ta1—C6	109.2 (3)	C11—Ta1—I1	77.9 (2)
C1 <sup>i</sup> —Ta1—C6	141.1 (2)	C8—Ta1—I1	163.56 (16)

C1—Ta1—C9	132.6 (3)	C12—Ta1—I1	73.25 (16)
C1 <sup>i</sup> —Ta1—C9	106.3 (3)	C2—Ta1—I1	101.77 (9)
C6—Ta1—C9	89.0 (3)	C2 <sup>i</sup> —Ta1—I1	101.77 (9)
C1—Ta1—C5	121.5 (3)	C2—C1—C1 <sup>i</sup>	130.4 (2)
C1 <sup>i</sup> —Ta1—C5	155.1 (3)	C2—C1—Ta1	84.5 (2)
C6—Ta1—C5	34.1 (2)	C1 <sup>i</sup> —C1—Ta1	72.20 (10)
C9—Ta1—C5	98.3 (3)	C2—C1—H1	114.8
C1—Ta1—C10	148.7 (2)	C1 <sup>i</sup> —C1—H1	114.8
C1 <sup>i</sup> —Ta1—C10	113.2 (2)	Ta1—C1—H1	118.6
C6—Ta1—C10	99.5 (3)	C1—C2—C3	133.9 (4)
C9—Ta1—C10	33.7 (2)	C1—C2—Ta1	62.89 (19)
C5—Ta1—C10	89.3 (3)	C3—C2—Ta1	115.0 (2)
C1—Ta1—C7	110.4 (3)	C1—C2—H2	113.0
C1 <sup>i</sup> —Ta1—C7	125.5 (2)	C3—C2—H2	113.0
C6—Ta1—C7	33.1 (2)	Ta1—C2—H2	92.4
C9—Ta1—C7	63.2 (3)	C4—C3—C2	135.3 (4)
C5—Ta1—C7	63.4 (3)	C4—C3—H3	112.4
C10—Ta1—C7	87.0 (3)	C2—C3—H3	112.4
C1—Ta1—C11	154.0 (2)	C3—C4—C4 <sup>i</sup>	132.7 (3)
C1 <sup>i</sup> —Ta1—C11	130.1 (2)	C3—C4—H4	113.7
C6—Ta1—C11	88.8 (3)	C4 <sup>i</sup> —C4—H4	113.7
C9—Ta1—C11	63.9 (3)	C6—C5—C12	134.7 (8)
C5—Ta1—C11	64.5 (3)	C6—C5—Ta1	71.3 (5)
C10—Ta1—C11	33.4 (2)	C12—C5—Ta1	76.0 (6)
C7—Ta1—C11	95.0 (3)	C6—C5—H5	112.7
C1—Ta1—C8	118.50 (19)	C12—C5—H5	112.7
C1 <sup>i</sup> —Ta1—C8	111.9 (2)	Ta1—C5—H5	136.9
C6—Ta1—C8	63.8 (3)	C7—C6—C5	133.8 (8)
C9—Ta1—C8	33.0 (2)	C7—C6—Ta1	76.2 (6)
C5—Ta1—C8	87.1 (3)	C5—C6—Ta1	74.6 (6)
C10—Ta1—C8	63.7 (3)	C7—C6—H6	113.1
C7—Ta1—C8	32.5 (2)	C5—C6—H6	113.1
C11—Ta1—C8	86.0 (3)	Ta1—C6—H6	129.8
C1—Ta1—C12	139.89 (19)	C6—C7—C8	137.5 (9)
C1 <sup>i</sup> —Ta1—C12	148.81 (18)	C6—C7—Ta1	70.7 (6)
C6—Ta1—C12	64.7 (2)	C8—C7—Ta1	75.0 (6)
C9—Ta1—C12	87.5 (4)	C6—C7—H7	111.3
C5—Ta1—C12	33.6 (2)	C8—C7—H7	111.3
C10—Ta1—C12	64.3 (3)	Ta1—C7—H7	144.5
C7—Ta1—C12	85.7 (3)	C7—C8—C9	133.9 (9)
C11—Ta1—C12	33.2 (2)	C7—C8—Ta1	72.5 (6)
C8—Ta1—C12	95.1 (2)	C9—C8—Ta1	71.0 (6)
C1—Ta1—C2	32.60 (13)	C7—C8—H8	113.1
C1 <sup>i</sup> —Ta1—C2	62.58 (13)	C9—C8—H8	113.1
C6—Ta1—C2	78.9 (2)	Ta1—C8—H8	143.0
C9—Ta1—C2	122.1 (2)	C8—C9—C10	135.7 (8)
C5—Ta1—C2	101.3 (2)	C8—C9—Ta1	76.0 (6)
C10—Ta1—C2	155.6 (2)	C10—C9—Ta1	73.6 (6)

C7—Ta1—C2	78.5 (3)	C8—C9—H9	112.2
C11—Ta1—C2	165.8 (2)	C10—C9—H9	112.2
C8—Ta1—C2	94.66 (19)	Ta1—C9—H9	134.1
C12—Ta1—C2	132.81 (18)	C9—C10—C11	133.9 (9)
C1—Ta1—C2 <sup>i</sup>	62.58 (13)	C9—C10—Ta1	72.7 (6)
C1 <sup>i</sup> —Ta1—C2 <sup>i</sup>	32.60 (13)	C11—C10—Ta1	75.1 (6)
C6—Ta1—C2 <sup>i</sup>	136.4 (2)	C9—C10—H10	113.0
C9—Ta1—C2 <sup>i</sup>	74.0 (3)	C11—C10—H10	113.0
C5—Ta1—C2 <sup>i</sup>	169.2 (3)	Ta1—C10—H10	135.0
C10—Ta1—C2 <sup>i</sup>	88.2 (2)	C10—C11—C12	135.4 (8)
C7—Ta1—C2 <sup>i</sup>	106.0 (2)	C10—C11—Ta1	71.5 (5)
C11—Ta1—C2 <sup>i</sup>	116.8 (2)	C12—C11—Ta1	74.2 (5)
C8—Ta1—C2 <sup>i</sup>	82.4 (2)	C10—C11—H11	112.3
C12—Ta1—C2 <sup>i</sup>	149.84 (18)	C12—C11—H11	112.3
C2—Ta1—C2 <sup>i</sup>	77.28 (17)	Ta1—C11—H11	141.1
C1—Ta1—I1	76.97 (9)	C11—C12—C5	133.7 (8)
C1 <sup>i</sup> —Ta1—I1	76.97 (9)	C11—C12—Ta1	72.6 (5)
C6—Ta1—I1	118.5 (2)	C5—C12—Ta1	70.4 (6)
C9—Ta1—I1	132.5 (2)	C11—C12—H12	113.1
C5—Ta1—I1	89.0 (3)	C5—C12—H12	113.1
C10—Ta1—I1	100.3 (2)	Ta1—C12—H12	143.8
C1 <sup>i</sup> —C1—C2—C3	38.6 (5)	C7—C8—C9—C10	-8 (3)
Ta1—C1—C2—C3	99.6 (4)	Ta1—C8—C9—C10	-47.7 (16)
C1 <sup>i</sup> —C1—C2—Ta1	-60.97 (17)	C7—C8—C9—Ta1	39.5 (14)
C1—C2—C3—C4	-18.5 (8)	C8—C9—C10—C11	1 (2)
Ta1—C2—C3—C4	57.1 (5)	Ta1—C9—C10—C11	-47.7 (12)
C2—C3—C4—C4 <sup>i</sup>	-13.5 (6)	C8—C9—C10—Ta1	48.4 (16)
C12—C5—C6—C7	-7 (2)	C9—C10—C11—C12	6 (2)
Ta1—C5—C6—C7	-52.8 (12)	Ta1—C10—C11—C12	-41.4 (14)
C12—C5—C6—Ta1	46.1 (13)	C9—C10—C11—Ta1	47.0 (12)
C5—C6—C7—C8	14 (2)	C10—C11—C12—C5	3 (2)
Ta1—C6—C7—C8	-37.9 (15)	Ta1—C11—C12—C5	-38.2 (13)
C5—C6—C7—Ta1	52.2 (12)	C10—C11—C12—Ta1	40.7 (14)
C6—C7—C8—C9	-2 (3)	C6—C5—C12—C11	-6 (2)
Ta1—C7—C8—C9	-39.1 (15)	Ta1—C5—C12—C11	38.8 (13)
C6—C7—C8—Ta1	36.9 (15)	C6—C5—C12—Ta1	-44.7 (13)

Symmetry code: (i)  $x, -y+1, z$ .