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### SYNTHESIS AND STRUCTURE OF A PENTAMETHYLCYCLOPENTADIENYL THULIUM CHLORIDE COMPLEX, $[(C_5Me_5)_2Tm(\mu_3-Cl)_2K(THF)]_n$

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# SYNTHESIS AND STRUCTURE OF A PENTAMETHYLCYCLOPENTADIENYL THULIUM CHLORIDE COMPLEX, $[(C_5Me_5)_2Tm(\mu_3-Cl)_2K(THF)]_n^*$

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The first structural data on a pentamethylcyclopentadienyl complex of thulium,  $[(C_5Me_5)_2Tm(\mu_3-Cl)_2K(THF)]_n$ , **1**, are reported.  $TmCl_3$  reacts with 2 equivalents of  $KC_5Me_5$  in THF to form **1** in > 80% yield. **1** crystallizes from THF/hexanes in the space group Cc with  $a = 11.1491(13)$  Å,  $b = 28.107(2)$  Å,  $c = 8.5921(8)$  Å,  $\beta = 102.485(8)^\circ$ ,  $V = 2628.8(4)$  Å<sup>3</sup>, and  $D_{\text{calcd}} = 1.570$  Mg/m<sup>3</sup> for  $Z = 4$ . Least-squares refinement of the model based on 3114 reflections ( $F^2 > 3.0\sigma(F^2)$ ) converged to a final  $R_F = 1.9\%$ . The polymeric structure of **1** is generated by triply bridging chlorides, each of which is connected to one  $(C_5Me_5)_2Tm$  bent metallocene unit and two K(THF) moieties in a T-shaped geometry around the chloride.

*Keywords:* Thulium; pentamethylcyclopentadienyl; bridging chlorides; coordination polymer

## INTRODUCTION

Historically, thulium has not been one of the heavily investigated metals in the lanthanide series, since other metals of similar size appear to accomplish similar chemistry<sup>1</sup> and are easier to study due to their lower magnetic moments.<sup>2</sup> Thulium is distinctive in the lanthanide series in that it has a very reactive divalent state: the Tm(III)/Tm(II) reduction potential is estimated to be  $-2.3$  V vs. NHE.<sup>3</sup> Previously, this highly reducing divalent

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state was not of chemical consequence since it had only been identified transiently in solution or in the solid state.<sup>4</sup> Recently, however, a crystallographically characterizable complex of Tm(II) has been isolated,  $\text{TmI}_2(\text{MeOCH}_2\text{CH}_2\text{OMe})_3$ ,<sup>5</sup> which may be a good precursor for new thulium-based chemistry. Future development of the chemistry of thulium will require X-ray crystallographic data on various classes of thulium compounds, but to date very few thulium structures have been reported. Structural studies on organometallic thulium complexes and related species are particularly sparse.<sup>6-9</sup> To our knowledge there are no crystallographic data in the literature on pentamethylcyclopentadienyl thulium complexes. Given the pervasive use of  $\text{C}_5\text{Me}_5$  as a co-ligand in organolanthanide chemistry,<sup>1</sup> data on  $\text{C}_5\text{Me}_5/\text{Tm}$  complexes should be available for comparison. We report here on the structure of  $[(\text{C}_5\text{Me}_5)_2\text{Tm}(\mu_3\text{-Cl})_2\text{K}(\text{THF})]_n$ , **1**.

## EXPERIMENTAL

All of the chemistry described below was performed under nitrogen with rigorous exclusion of air and water using standard Schlenk, vacuum line, and glove box techniques. Solvents were dried and distilled<sup>10</sup> and thulium trichloride (Rhône-Poulenc) was dried<sup>11</sup> as described previously.  $\text{KC}_5\text{Me}_5$  was prepared from  $\text{KH}$  and  $\text{C}_5\text{Me}_5\text{H}$  (Strem) in THF and dried *in vacuo*. Magnetic susceptibility measurements were recorded on a General Electric GN500 following the Evans' method.<sup>12</sup> Infrared spectra were obtained on a Perkin Elmer 1600 FT-IR spectrometer.

### $[(\text{C}_5\text{Me}_5)_2\text{Tm}(\mu_3\text{-Cl})_2\text{K}(\text{THF})]_n$ , **1**

In the glovebox,  $\text{TmCl}_3$  (79 mg, 0.287 mmol) and THF (5 mL) were combined and  $\text{KC}_5\text{Me}_5$  (100 mg, 0.574 mmol) was added to the resulting slurry. The solution immediately turned a cloudy, bright yellow and was allowed to stir overnight. The reaction mixture was centrifuged to remove solids and the THF was removed by rotary evaporation, leaving yellow solid **1** (152 mg, 86%). IR (KBr): 2966 s, 2905 s, 2719 w, 1484 w, 1436 s, 1376 m, 1261 w, 1179 w, 1053 s, 1020 m, 900 m, 867 w, 797 w, 667 w ( $\text{cm}^{-1}$ ). *Anal.* Calcd. for  $[(\text{C}_5\text{Me}_5)_2\text{TmCl}_2\text{K}(\text{THF})]_n$ : Tm, 27.2. Found: Tm, 28.1. Magnetic susceptibility (293 K)  $\chi_M = 2.30 \times 10^{-2}$ ;  $\mu_{\text{eff}} = 7.3 \mu_B$ . Crystals suitable for X-ray diffraction were obtained from slow diffusion of hexanes into a THF solution of **1** at ambient temperature.

**X-ray Data Collection, Structure Determination and Refinement for  $[(C_5Me_5)_2Tm(\mu_3-Cl)_2K(THF)]_n$ , 1**

A yellow crystal of approximate dimensions  $0.25 \times 0.31 \times 0.33$  mm was mounted on a glass fiber and transferred to the Syntex P2<sub>1</sub> diffractometer. Determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by previously described techniques similar to those of Churchill.<sup>13</sup> Low temperature (183 K) intensity data were collected *via* a  $2\theta$ - $\theta$  scan technique with MoK $\alpha$  radiation under the conditions given in Table I.

All 3265 data were corrected for absorption<sup>14</sup> and for Lorentz and polarization effects and placed on an approximately absolute scale. Any reflection with  $I(\text{net}) < 0$  was assigned the value  $F_0 = 0$ . The systematic extinctions observed were  $hkl$  for  $h + k = 2n + 1$  and  $h0l$  for  $l = 2n + 1$ ; the diffraction symmetry was  $2/m$ . The two possible monoclinic space groups are Cc [C<sub>s</sub><sup>4</sup>; No. 9] or C2/c [C<sub>2h</sub><sup>6</sup>; No. 15]. The non-centrosymmetric space group was chosen and later shown to be the correct choice.

TABLE I Experimental data for the X-ray diffraction study

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Formula: C <sub>24</sub> H <sub>38</sub> Cl <sub>2</sub> KOTm
Fw: 621.5
Temperature (K): 183
Crystal system: monoclinic
Space group: Cc
$a = 11.1491(13)$ Å
$b = 28.107(2)$ Å
$c = 8.5921(8)$ Å
$\beta = 102.485(8)^\circ$
$V = 2628.9(4)$ Å <sup>3</sup>
$Z = 4$
$D_{\text{calcd}}$ , Mg/m <sup>3</sup> = 1.570
Diffractometer: Syntex P2 <sub>1</sub> (R3m/V system)
Radiation: MoK $\alpha$ ( $\lambda = 0.710730$ )
Monochromator: Highly oriented graphite
Data collected: $+h, +k, \pm l$
Scan type: $2\theta$ - $\theta$
Scan range: $1.20^\circ$
Scan speed: $3.0 \text{ deg min}^{-1}$ (in $\omega$ )
$2\theta$ range: $4.0$ - $55.0^\circ$
$\mu(\text{MoK})$ , $\text{mm}^{-1} = 3.749$
Absorption correction: semi-empirical ( $\varphi$ -scan method)
Reflections collected: 3265
Reflections with $F_0 > 3.0\sigma(F_0)$ : 3114
No. of variables: 261
RF = 1.9%, R <sub>w</sub> F = 2.5%
Goodness of fit: 1.15

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All crystallographic calculations were carried out using the UCI modified version of the UCLA Crystallographic Computing Package<sup>15</sup> and the SHELXTL PLUS program set.<sup>16</sup> The analytical scattering factors for neutral atoms were used throughout the analysis;<sup>17a</sup> both the real ( $\Delta f'$ ) and imaginary ( $i\Delta f''$ ) components of anomalous dispersion<sup>17b</sup> were included. The quantity minimized during least-squares analysis was  $\sum w(F_0 - F_c)^2$  where  $w^{-1} = \sigma^2(F_0) + 0.0003(F_0)^2$ .

The structure was solved by direct methods (SHELXTL PLUS); and refined by full-matrix least-squares techniques. Hydrogen atoms were included using a riding model with  $d(C-H) = 0.96 \text{ \AA}$  and  $U(\text{iso}) = 0.08 \text{ \AA}^2$ . Refinement of positional and thermal parameters led to convergence with  $R_F = 1.9\%$ ,  $R_{wF} = 2.5\%$  and  $\text{GOF} = 1.15$  for 261 variables refined against those 3114 data with  $F_0 > 3.0\sigma(F_0)$ . The absolute structure was determined

TABLE II Atomic coordinates ( $\times 10^5$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^4$ )

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq)*
Tm(1)	50000	37115(1)	50000	197(1)
K(1)	37092(13)	51919(4)	45389(12)	302(3)
Cl(1)	47988(14)	43740(4)	28239(13)	297(3)
Cl(2)	36524(13)	42448(4)	64503(13)	280(3)
C(1)	28182(66)	33190(22)	38731(88)	355(19)
C(2)	34234(57)	33083(22)	26023(69)	305(17)
C(3)	44235(52)	30006(19)	30152(69)	372(17)
C(4)	44406(54)	28116(17)	45424(71)	380(17)
C(5)	34590(57)	30223(20)	50907(63)	382(17)
C(6)	16289(60)	35865(30)	38268(100)	581(25)
C(7)	29910(77)	35330(30)	9877(74)	592(24)
C(8)	52657(66)	28710(28)	19049(98)	635(28)
C(9)	51724(97)	23874(21)	52814(138)	739(39)
C(10)	30512(85)	29110(33)	66148(89)	712(31)
C(11)	69324(45)	41954(16)	63996(56)	255(13)
C(12)	73680(44)	38938(18)	53153(59)	270(13)
C(13)	73296(58)	34216(19)	58913(71)	254(15)
C(14)	68719(57)	34283(23)	72749(74)	309(17)
C(15)	66324(45)	39115(19)	76220(56)	280(14)
C(16)	69186(52)	47311(17)	63154(78)	388(17)
C(17)	79013(56)	40428(25)	39305(71)	421(19)
C(18)	79465(59)	30055(21)	52409(82)	466(21)
C(19)	68492(63)	30240(24)	84255(83)	502(21)
C(20)	62755(56)	40839(27)	91144(63)	449(20)
O(1)	13111(45)	53838(18)	39615(60)	539(16)
C(21)	5892(64)	57600(26)	31234(84)	527(23)
C(22)	-1602(63)	59468(29)	42541(96)	603(25)
C(23)	-3138(95)	55165(40)	52206(128)	869(43)
C(24)	6135(104)	51776(46)	49482(177)	1150(59)

\*Equivalent isotropic *U* defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

by refinement of the Rogers'  $\eta$ -parameter [= 1.06(3)].<sup>18</sup> A final difference-Fourier map yielded  $\rho(\text{max}) = 0.41 \text{ e}\text{\AA}^{-3}$ . Atomic coordinates and equivalent isotropic displacement coefficients are given in Table II.

## RESULTS AND DISCUSSION

$\text{TmCl}_3$  reacts with  $\text{KC}_5\text{Me}_5$  in THF to form a polymeric KCl adduct,  $[(\text{C}_5\text{Me}_5)_2\text{Tm}(\mu_3\text{-Cl})_2\text{K}(\text{THF})]_n$ , **1**, in > 80% yield. **1** was characterized by X-ray crystallography (Figure 1) and found to be isostructural and isomorphous with the cerium analog,  $[(\text{C}_5\text{Me}_5)_2\text{Ce}(\mu_3\text{-Cl})_2\text{K}(\text{THF})]_n$ , **2**.<sup>19</sup> In both compounds, the lanthanide metal is formally eight coordinate and adopts the geometry typical for trivalent  $(\text{C}_5\text{Me}_5)_2\text{Ln}$  complexes, namely a bent metallocene unit with two monodentate ligands attached. Complexes **1** and **2** are polymeric *via* the coordination sphere of the potassium ion. With only one molecule of solvation, the potassium attaches to four triply-bridging chlorides to generate the polymeric structure shown in

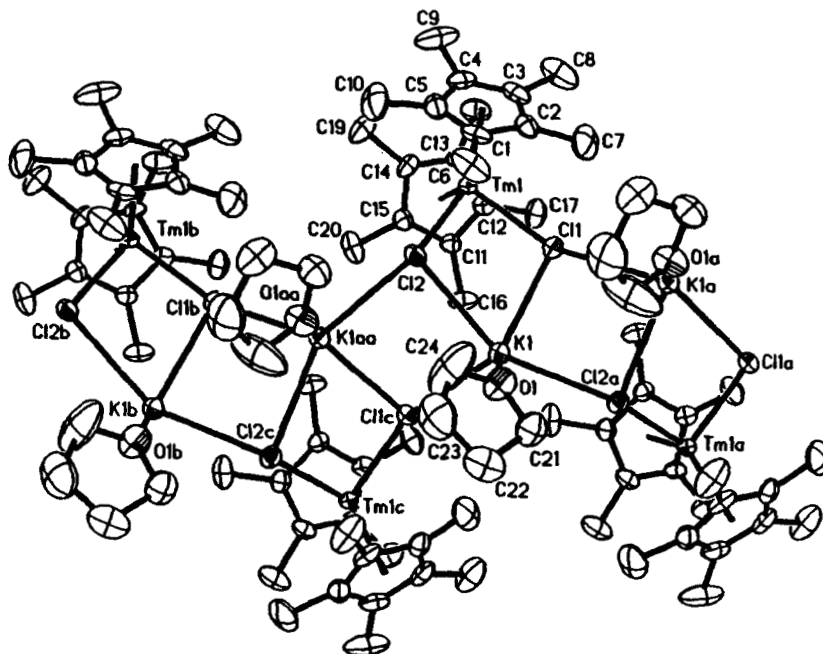


FIGURE 1 Thermal ellipsoid plot of  $[(\text{C}_5\text{Me}_5)_2\text{Tm}(\mu_3\text{-Cl})_2\text{K}(\text{THF})]_n$ , **1**, drawn at the 50% probability level.

Figure 1. The chloride ions have a distorted T-shaped geometry with two M–Cl–M angles in the 90–100° range and one M–Cl–M angle of 142–154° (M = K or Tm).

The bond distances and angles in **1** (Table III) are as expected compared to **2** when the difference in the radii of the metals is considered. For example, the Ln–C(C<sub>5</sub>Me<sub>5</sub>) average distances are 2.64(2) Å and 2.79(2) Å for **1** and **2**, respectively, and the difference in their eight coordinate radii is 0.149 Å.<sup>20</sup> Similarly, the Ln–Cl average distances are 2.617(3) Å and 2.777(1) Å for **1** and **2**, respectively.

The formation of alkali metal halide adducts in lanthanide chemistry is not unusual and many pentamethylcyclopentadienyl complexes of the general formula (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>LnX<sub>2</sub>M(solvent)<sub>n</sub> are known where M = alkali metal, X = halides or pseudohalides, solvent = ethers and other coordinating solvents.<sup>21–25</sup> In most cases, the alkali metal is solvated by two donor ligands, as in the crystallographically characterized complexes (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Yb(μ-I)<sub>2</sub>Li(OEt<sub>2</sub>)<sub>2</sub>,<sup>23</sup> (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Yb(μ-Cl)<sub>2</sub>Li(OEt<sub>2</sub>)<sub>2</sub>,<sup>23</sup> (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Y(μ-Cl)<sub>2</sub>Li(THF)<sub>2</sub>,<sup>21</sup> (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Ce(μ-Cl)<sub>2</sub>Li(OEt<sub>2</sub>)<sub>2</sub>,<sup>24</sup> and (C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Pr(μ-Cl)<sub>2</sub>Na(MeOCH<sub>2</sub>CH<sub>2</sub>OMe)<sub>2</sub>.<sup>25</sup> However, it was previously shown that the CeCl<sub>3</sub>/2KC<sub>5</sub>Me<sub>5</sub> combination crystallizes from THF as a monosolvate, [(C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Ce(μ<sub>3</sub>-Cl)<sub>2</sub>K(THF)]<sub>n</sub>, **2**, which fills the remaining space in the

TABLE III Bond distances (Å) and angles (°) for [(C<sub>5</sub>Me<sub>5</sub>)<sub>2</sub>Tm(μ<sub>3</sub>-Cl)<sub>2</sub>K(THF)]<sub>n</sub>, **1**

Tm(1)–K(1)	4.393(1)	Tm(1)–C(11)	2.611(5)
Tm(1)–Cl(1)	2.614(1)	Tm(1)–C(12)	2.645(5)
Tm(1)–Cl(2)	2.620(1)	Tm(1)–C(13)	2.671(6)
Tm(1)–C(1)	2.656(7)	Tm(1)–C(14)	2.654(6)
Tm(1)–C(2)	2.656(6)	Tm(1)–C(15)	2.633(4)
Tm(1)–C(3)	2.615(6)	Tm(1)–Cnt(1)	2.341
Tm(1)–C(4)	2.614(5)	Tm(1)–Cnt(2)	2.353
Tm(1)–C(5)	2.602(6)		
K(1)–Cl(1)	3.115(2)	K(1)–K(1A)	4429(1)
K(1)–Cl(2)	3.136(2)	K(1)–Cl(1A)	3.074(2)
K(1)–O(1)	2.667(5)	K(1)–Cl(2A)	3.079(2)
Cl(1)–Tm(1)–Cl(2)	87.9(1)	Cl(2)–K(1)–Cl(1A)	84.5(1)
K(1)–Tm(1)–C(1)	96.0(1)	O(1)–K(1)–Cl(1A)	105.8(1)
Cnt(1)–Tm(1)–Cl(1)	105.7	Cl(1)–K(1)–Cl(2A)	84.8(1)
Cnt(1)–Tm(1)–Cl(2)	105.6	Cl(2)–K(1)–Cl(2A)	152.7(1)
Cnt(2)–Tm(1)–Cl(1)	105.4	Cl(1A)–K(1)–Cl(2A)	121.2(1)
Cnt(2)–Tm(1)–Cl(2)	106.3	Tm(1)–Cl(1)–K(1)	99.7(1)
Cnt(1)–Tm(1)–Cnt(2)	135.6	Tm(1)–Cl(1)–K(1B)	153.6(1)
Cl(1)–K(1)–Cl(2)	71.1(1)	K(1)–Cl(1)–K(1B)	91.4(1)
Cl(1)–K(1)–O(1)	122.6(1)	Tm(1)–Cl(2)–K(1)	99.1(1)
Cl(2)–K(1)–O(1)	97.8(1)	Tm(1)–Cl(2)–K(1A)	142.8(1)
Cl(1)–K(1)–Cl(1A)	127.6(1)	K(1)–Cl(2)–K(1A)	90.9(1)

coordination sphere of potassium by oligomerization.<sup>19</sup> Identification of the isostructural thulium analog, **1**, shows that this oligomeric structure does not necessarily require a large lanthanide, like cerium, and can result with smaller metals at the end of the series as well. Hence, the data obtained to date suggest that the formation of monomeric disolvates versus polymeric monosolvates is more dependent on the alkali metal than on the lanthanide.

## CONCLUSION

$[(C_5Me_5)_2Tm(\mu_3-Cl)_2K(THF)]_n$ , **1**, has been prepared in good yields from the  $TmCl_3$  and  $KC_5Me_5$  and is isostructural with a cerium analog. This bis(pentamethylcyclopentadienyl) thulium complex represents the first structurally characterized pentamethylcyclopentadienyl complex of thulium.

## Supplementary Material

X-ray diffraction and structure factor tables available from W.J.E.

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