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# Achiral and chiral, C<sub>2</sub>-symmetric bicyclic guanidines based on 1,5,7-triazabicyclo[4.4.0.]dec-5-ene as ligands in high- and mid-valent early transition metal chemistry

Adil Mohammad  
*University of Iowa*

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ACHIRAL AND CHIRAL,  $C_2$ -SYMMETRIC BICYCLIC GUANIDINATES BASED  
ON 1,5,7-TRIAZABICYCLO[4.4.0.]DEC-5-ENE AS LIGANDS IN HIGH- AND  
MID- VALENT EARLY TRANSITION METAL CHEMISTRY

by

Adil Mohammad

An Abstract

Of a thesis submitted in partial fulfillment  
of the requirements for the Doctor of  
Philosophy degree in Chemistry  
in the Graduate College of  
The University of Iowa

July 2010

Thesis Supervisor: Associate Professor Louis Messerle

## ABSTRACT

The syntheses of achiral and chiral,  $C_2$ -symmetric bicyclic guanidine derivatives of 1,5,7-triazabicyclo[4.4.0]dec-5-ene (hppH) are described.

Quantum mechanical MO calculations based on Spartan were performed in order to study the effects (electronic and steric) of substituents on the basicity of hppH derivatives. These calculations showed that substituting only one methyl group on each carbon next to the bridgehead nitrogen of hppH gives high basicity. Based on these calculations, syntheses of achiral (hpp\*H, with four methyl groups) and chiral  $C_2$ -symmetric (hpp'H, with two methyl groups) derivatives of hppH were developed based on cyclization of 1,5,9-triaminononanes with C1 reagents.

The synthesis of hpp\*H started from commercially available ethyl cyanoacetate and was completed in six steps with 2.5% overall yield. The synthesis of the chiral,  $C_2$ -symmetric hpp'H was carried out using L-alanine as the chiral precursor. The key step was the diastereoselective coupling of L-alaninol (derived from L-alanine) with commercially available hydroxyacetone via a reductive amination approach. An important step was successful diastereoselective resolution of N-benzyl-protected meso and  $C_2$ -symmetric aminodinitriles. Chiral hpp'H was synthesized in six steps starting from L-alaninol in 9% overall yield and was isolated as hpp'H<sub>2</sub><sup>+</sup>I<sup>-</sup>. Both hpp\*H and hpp'H<sub>2</sub><sup>+</sup>I<sup>-</sup> were characterized by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy and high resolution mass spectrometry. hpp'H<sub>2</sub><sup>+</sup>I<sup>-</sup> was also characterized by single-crystal X-ray diffractometry.

The coordination chemistry of non-methylated hpp<sup>-</sup> with tantalum is described. The new tantalum complexes Ta<sup>V</sup>(hpp)Cl<sub>4</sub>, Ta<sup>V</sup>(hpp)<sub>2</sub>Cl<sub>3</sub>, Cp\*Ta<sup>V</sup>(hpp)Cl<sub>3</sub> (Cp\* = C<sub>5</sub>Me<sub>5</sub>) and Cp''Ta<sup>V</sup>(hpp)Cl<sub>3</sub> (Cp'' = C<sub>5</sub>Me<sub>4</sub>Et) were synthesized. The first mid-valent hpp<sup>-</sup> complexes of tantalum, Cp\*Ta<sup>IV</sup>(hpp)Cl<sub>2</sub> and Cp''Ta<sup>IV</sup>(hpp)Cl<sub>2</sub>, were obtained by reduction of (C<sub>5</sub>Me<sub>4</sub>R)Ta(hpp)Cl<sub>3</sub> (R = Me, Et) or reaction of (C<sub>5</sub>Me<sub>4</sub>R)<sub>2</sub>Ta<sub>2</sub>(μ-Cl)<sub>4</sub> with (hpp)SiMe<sub>3</sub>. <sup>1</sup>H NMR spectroscopy and comparative single-crystal X-ray diffractometry of these complexes is discussed.

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Thesis Supervisor: Associate Professor Louis Messerle

Graduate College  
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Iowa City, Iowa

CERTIFICATE OF APPROVAL

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PH.D. THESIS

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This is to certify that the Ph.D. thesis of

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has been approved by the Examining Committee  
for the thesis requirement for the Doctor of Philosophy  
degree in Chemistry at the July 2010 graduation.

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Dedicated to my Late Father Mr. Hanif Mohammad

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## CHAPTER 1

## INTRODUCTION

Since the discovery of the first dinuclear species containing a quadruple bond,  $\text{Re}_2\text{Cl}_8^{2-}$  in 1965,<sup>1</sup> followed by the first compound with a metal-metal triple bond,<sup>2</sup> the chemistry of metal-metal bonded species has grown at a rapid pace, and a plethora of knowledge has accumulated on metal-metal bonded complexes.<sup>3</sup> The chemistry of metal-metal multiple bonds can be divided into metal-metal quadruple-, triple-, and double-bonded complexes. Complexes with metal-metal quadruple bonds are structurally interesting, but their reactivity has been less systematically studied as they are susceptible to cleavage. On the other hand, the chemistry of complexes with triple and double metal-metal bonds is very interesting particularly for small molecule activation.<sup>4</sup> Much of the progress in the field of metal-metal multiple bonds has been associated with the development of new types of metal-to-metal bridging ligands, which we term dinucleating ligands. A schematic dinucleating ligand (**I**) is shown in Figure 1.

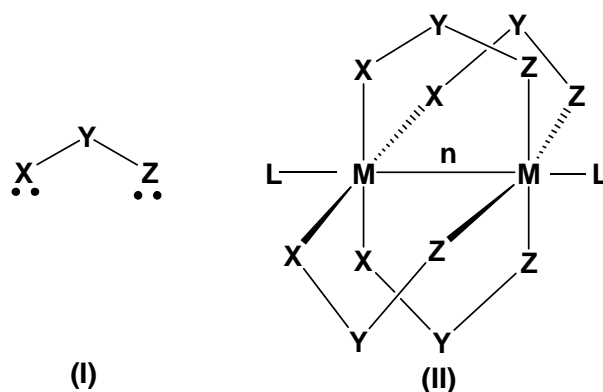


Figure 1: **I** Dinucleating ligands, and **II**, typical paddlewheel or lantern structures.

These are uninegative, bent, trinuclear (in the ligand backbone) anions with X-Z distances similar to the M-M distances across which they form a pair of approximately parallel X-M and Z-M bonds. Acetates are the now classic example of X-Y-Z

dinucleating ligand. Numerous compounds having two metal atoms (bond order ranging from  $\frac{1}{2}$  to 4) bridged by four monoanions have been prepared and are known as *paddlewheel* (structure II, Figure 1) or *tetragonal lantern* (without the axial ligands L) complexes.

## An Overview of Electronic Structure of Metal-Metal

### Multiple Bonds

The body of literature on M-M multiple bonds is immense. In this thesis the focus is on compounds that have each of two metal atoms forming a square or square pyramidal  $MX_4$  arrangement. Transition metals can form double or higher bonds from overlap of orbitals of angular momentum quantum number 2 (*d*-orbitals). A qualitative picture of quadruple bond formation can be obtained by considering *d*-orbital overlaps. When two metal atoms approach, symmetry allows five non-zero overlaps between pairs of *d*-orbitals on the two atoms. These five non-zero overlaps are those between the corresponding pairs, i.e.  $d_{z^2}$  with  $d_{z^2}$ ,  $d_{xz}$  with  $d_{xz}$ ,  $d_{xy}$  with  $d_{xy}$ ,  $d_{zx}$  with  $d_{zx}$  and  $d_{x^2-y^2}$  with  $d_{x^2-y^2}$ . The positive overlap of the two  $d_{z^2}$  orbitals,  $d_{z^2}^{(\text{atom 1})} + d_{z^2}^{(\text{atom 1})}$ , gives rise to a  $\sigma$ -bonding orbital (Figure 2). The corresponding antibonding  $\sigma^*$  molecular orbital is formed by negative overlap,  $d_{z^2}^{(\text{atom 1})} - d_{z^2}^{(\text{atom 1})}$ . The  $d_{xz}^{(\text{atom 1})} + d_{xz}^{(\text{atom 2})}$  and  $d_{yz}^{(\text{atom 1})} + d_{yz}^{(\text{atom 2})}$  (degenerate and orthogonal) atomic orbitals overlap to form molecular  $\pi$  bonds. Similarly, antibonding  $\pi^*$  molecular orbitals are formed by the negative overlap of each of these pairs of atomic orbitals. The combination of  $d_{xy}$  orbitals leads to the formation of bonding and antibonding  $\delta$  and  $\delta^*$  molecular orbitals. The remaining atomic orbital  $d_{x^2-y^2}$  on each metal atom interacts primarily with the ligand orbitals, as shown by calculations. Thus, they make a strong contribution to metal-ligand bonding but very little to M-M bonding. According to Hückel theory, MO energies are proportional to overlap integrals for similar types of atomic orbitals, and atomic orbital overlap increases in the order  $\sigma < \pi < \delta$ , so the molecular orbitals are ordered in energy as follows, beginning with the most stable:

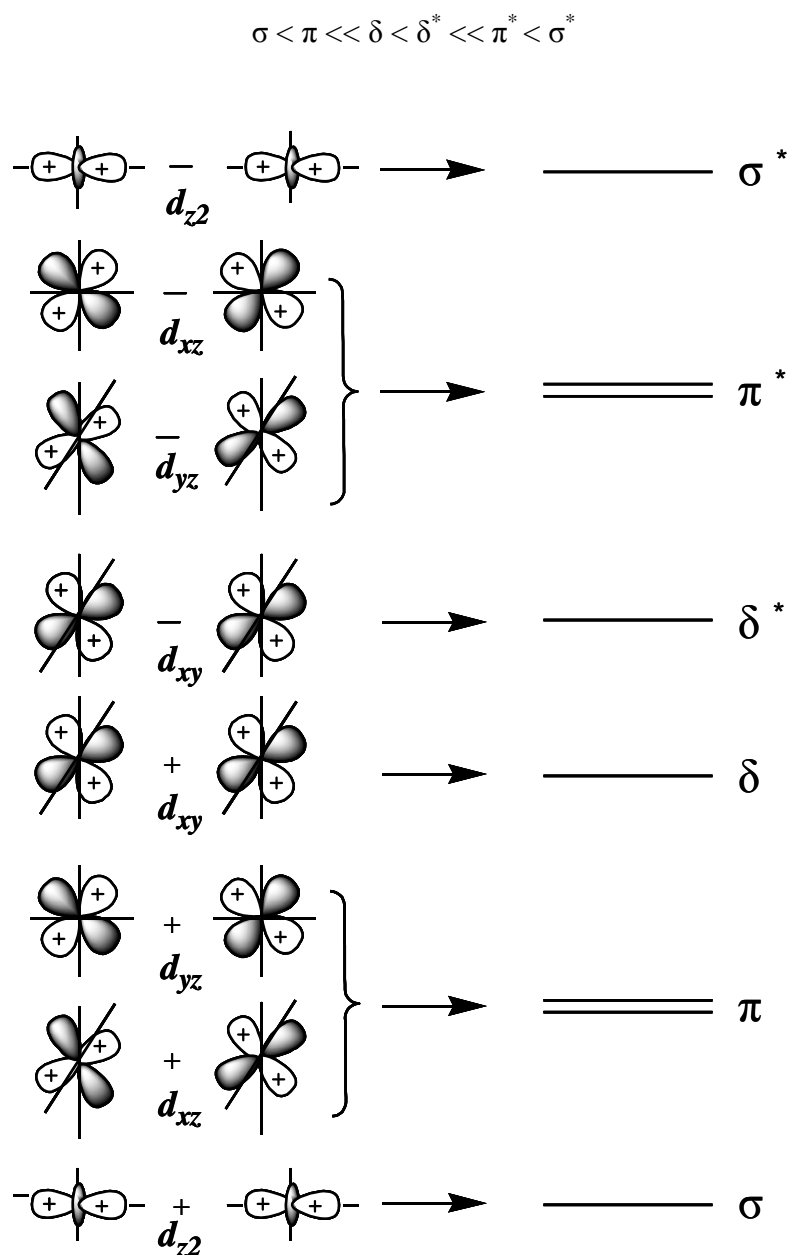


Figure 2: Diagram of overlaps of  $d$ -orbitals and resulting energy levels in the formation of M-M multiple bonds in  $X_4M-MX_4$  structure.

For the  $[\text{Re}_2\text{Cl}_8]^{2-}$  ion (the first recognized example of a compound with a quadruple bond), there are eight electrons (the Re atoms are in a formal oxidation state III and are thus each  $d^4$ ) to be placed in these molecular orbitals. These eight electrons fill

the bonding molecular orbitals, giving the configuration  $\sigma^2 \pi^4 \delta^2$ . There are four pairs of bonding and no antibonding electrons. According to MO theory, the definition of bond order is

$$\text{Bond order} = \frac{n_b - n_a}{2}$$

where  $n_b$  and  $n_a$  designate the number of electrons occupying bonding and antibonding orbitals, respectively. For  $[\text{Re}_2\text{Cl}_8]^{2-}$  the bond order is therefore 4, i.e. a quadruple bond. The quadruple bond accounts for two prominent molecular features: the extreme shortness of the Re-Re bond and the bond's tendency to impose an eclipsed configuration for the ligands. The  $\sigma$ -bond is cylindrically symmetric. Hence, the  $\sigma^2 \pi^4$  part is insensitive to the angle of internal rotation. The  $\delta$  component of the bond is markedly angle sensitive. The  $d_{xy}^{(\text{atom 1})} + d_{xy}^{(\text{atom 2})}$  overlap has its maximum value when the two  $\text{ReCl}_4$  units are precisely eclipsed, and it has a value of zero when the rotational conformation is precisely staggered. Therefore, any rotation from the eclipsed conformation causes a loss of  $\delta$  bond energy and, when carried to the limit of precise staggering, causes complete disappearance of the  $\delta$  bond. It is this dependence of the  $\delta$  bond on rotation angle that opposes any staggered conformation of ligands in these types of complexes.

Complexes with metal-metal bonds can be divided into two broad categories based on the oxidation state of the metal: (1) Those complexes with the metal atoms in a formal oxidation state of zero or close to it, including negative ones. Metal carbonyls are a prominent class of such complexes. These compounds have M-M bonds which are typically long, weak, and of bond order one. (2) Compounds with metal atoms in low to medium positive oxidation states. Many ground state electronic configurations are possible for the  $\text{MX}_4$  type paddlewheel complexes as shown in the energy level diagram. (Figure 2). Bond orders can vary, in steps of  $\frac{1}{2}$ , from  $\frac{1}{2}$  to 4. Bond orders can result in two ways: "electron poor" are those which have electrons in bonding molecular orbitals, while "electron rich" are those with electrons in both bonding and antibonding molecular orbitals.

### Ligands used in Paddlewheel Complexes

Much of the evolution in this field has come from advancements in the development of new types of dinucleating ligands, primarily (1) to extend the range of  $M_2^{n+}$  chemistry to other elements and other oxidation states, (2) to give a greater ease of preparation and greater stability, and (3) to obtain compounds with a range of properties such as redox potentials. The first dinucleating ligand to be widely used was acetate (**1**) (Figure 3), and trifluoroacetate anions led to the synthesis of paddlewheel complexes of Cr, Mo, W, Tc, Re, Ru, Os, Rh and Pt.<sup>1</sup>

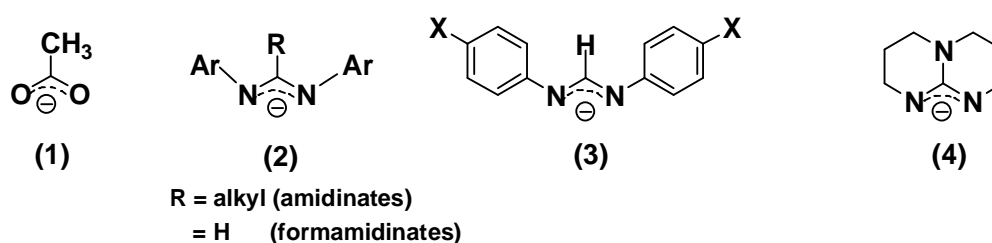


Figure 3: Various ligands used in paddlewheel complexes.

The further extension of metal-metal multiple bonding to other elements as paddlewheel complexes came with the incorporation of amidinate-type ligands (**2**), such as formamidinates (**3**). These ligands were postulated to be better than carboxylates as they are more basic (having nitrogens instead of oxygens, as in carboxylates). Complexes with formamidinates were easy to prepare, and variation of the *p*-substituents, X, permitted control of redox potential<sup>5</sup> and solubility. Significant developments using formamidinate ligands were the synthesis of the first  $V \equiv V$  (triple) bond<sup>6</sup>, the first  $Ni_2^{5+}$  complex with bond order 1/2,<sup>7</sup> and the first  $Fe_2^{3+,4+}$ ,<sup>8,9</sup>  $Co_2^{3+,4+,5+}$ ,<sup>10,11</sup>  $Ir_2^{4+}$ ,<sup>12</sup> and  $Pt_2^{4+,5+,6+}$  complexes.<sup>13</sup> Formamidinate ligands, mainly with Ar = phenyl or *p*-tolyl, were thought to be “perfect” ligand for these type of complexes. However, it was discovered that formamidinates are cleaved under the strong reducing conditions used to synthesize

$\text{Nb}_2^{2+}$  and  $\text{Ta}_2^{2+}$  complexes.<sup>14,15</sup> It then became critical to find a more robust ligand. The ideal ligand would retain the N-C-N core of formamidinates and resist cleavage. A ligand which has these desirable characteristics is the anion of 1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidine (also known as 1,5,7-triazabicyclo[4.4.0]dec-5-ene (**4**)). Because of its long chemical name, it is abbreviated as hppH, and the anionic ligand obtainable from it by deprotonation is  $\text{hpp}^-$ . The first dinuclear complex,  $\text{Ru}_2(\text{hpp})_4\text{Cl}_2$ , using  $\text{hpp}^-$  as a ligand was made by Bear *et. al* in 1996.<sup>16</sup> Cotton and co-workers at the same time were looking for a more robust ligand. After Bear's success, Cotton and co-workers started exploring the capabilities of  $\text{hpp}^-$  as a ligand to stabilize  $\text{M}_2^{n+}$  units. The bicyclic nature of  $\text{hpp}^-$  was thought to stabilize the ligand towards cleavage by reduced metal ions. Cotton and co-workers were able to synthesize the first triply-bonded  $\text{Nb}_2^{4+}$  complex<sup>17</sup> with  $\text{hpp}^-$  ligands. Complexes<sup>18</sup> analogous to those already synthesized with formamidinates, such as  $\text{V}_2(\text{hpp})_4$ ,  $\text{Cr}_2(\text{hpp})_4$  and  $\text{Mo}_2(\text{hpp})_4$  were easily prepared.  $\text{Hpp}^-$  also stabilizes dinuclear frameworks,  $\text{M}_2^{n+}$ , with high oxidation states such as  $\text{Mo}_2^{4+,5+,6+}$ ,<sup>19</sup>  $\text{W}_2^{4+,5+,6+}$ ,<sup>20</sup>  $\text{Re}_2^{6+,7+}$ ,<sup>21,22</sup>  $\text{Ru}_2^{6+}$ ,<sup>16</sup>  $\text{Os}_2^{6+,7+}$ ,<sup>23</sup>  $\text{Ni}_2^{5+}$ ,<sup>24</sup>  $\text{Pd}_2^{6+}$ ,<sup>25</sup> and  $\text{Pt}_2^{6+}$ .<sup>26</sup>

The  $\text{hpp}^-$  ligand has a stronger electron donating ability than carboxylates and formamidinates and can stabilize dimetal units with high oxidation numbers, as shown in electrochemical studies<sup>19</sup> of several complexes of Mo and W. Figure 4 depicts electrode potentials (relative to Ag/AgCl) on the y-axis and various ligands (carboxylates, formamidinates, acyclic guanidates, and  $\text{hpp}^-$ ) for the complexes of Mo and W on the x-axis. It can be inferred from Figure 4 that oxidation of comparable quadruply-bonded  $\text{W}_2^{4+}$  species is significantly easier (as expected by periodic trends) than corresponding  $\text{Mo}_2^{4+}$  species. Also, oxidation of complexes containing carboxylates is not much different than for complexes with formamidinates. Oxidation of complexes with acyclic guanidates is less than complexes with carboxylate or formamidinate as ligands. However, the most significant difference is that it is easier to oxidize complexes when the ligand is  $\text{hpp}^-$  as compared to carboxylates and formamidinates ligands. The oxidation of  $\text{Mo}_2(\text{hpp})_4^+$  is far easier than the neutral tetraformamidinate or carboxylate analog species. Further evidence that  $\text{hpp}^-$  is an extremely basic ligand for lantern and paddlewheel complexes was the observation that the  $\text{W}_2(\text{hpp})_4$  molecule is the most easily oxidized known molecule,<sup>27</sup> as it has an onset ionization potential of 3.51 eV even

lower than that of 3.89 eV for Cs in the gas phase. In these  $M_2(\text{hpp})_4$  compounds, it is observed that there is a strong interaction of the ligand  $\pi$  orbitals and the electrons in the  $\delta$  orbital of the quadruply bonded  $M_2^{4+}$  units.<sup>28</sup> This strong destabilization of the dimetal  $\delta$  orbitals favors oxidation. Thus,  $W_2(\text{hpp})_4$  could be used as an organic-soluble reducing agent by synthetic chemists.

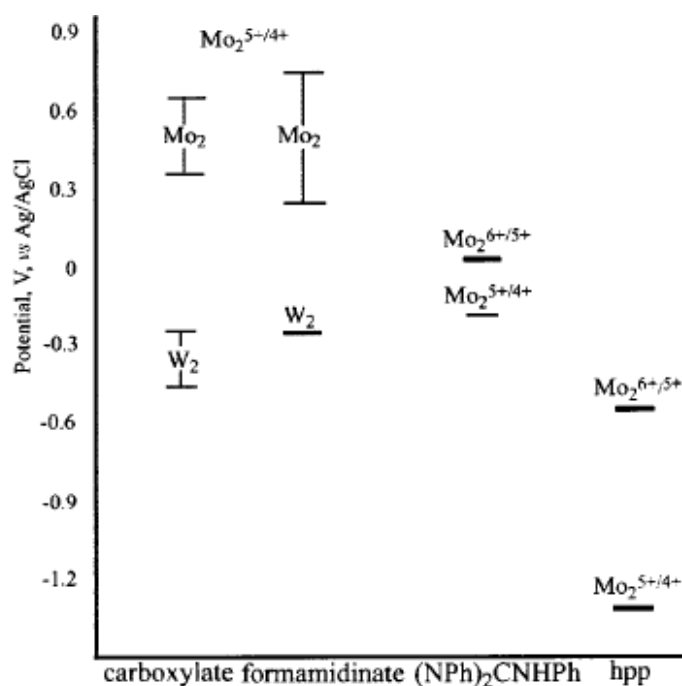


Figure 4: Variation in potentials as a function of ligand (carboxylates, formamidinates, acyclic guanidinate, and cyclic guanidinate hpp<sup>-</sup>) for paddlewheel complexes of the type  $M_2L_4$ ,  $M = \text{Mo}, \text{W}$ .



### Consequences of the Bicyclic Framework

It is clear from the work of many researchers that the  $\text{hpp}^-$  ligand is not only more robust than carboxylates and amidinates but also exhibits significant electronic differences. This can be attributed to two main reasons:

1. Guanidates can delocalize (Figure 5a) the negative charge throughout the molecule. In other words, confining the substituents of the non-amidine nitrogen atom  $\text{N}_3$  into

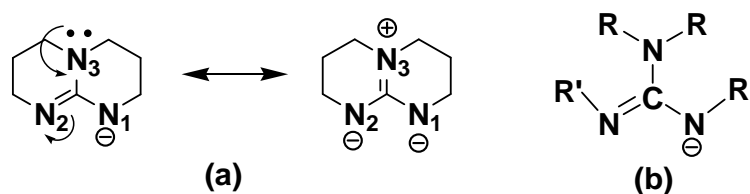


Figure 5: (a) Delocalized forms of bicyclic guanidinate anion  $\text{hpp}^-$  and (b) structure of an acyclic guanidinate ligand.

- the ring generates a favorable alignment for the lone-pair of this atom to be included in delocalization. In contrast, acyclic guanidates (Figure 5b) have an orthogonal displacement of the  $\text{N}_3$  substituents, from steric interactions with respect to the  $\text{CN}_2$  amidine unit. Also electronic structure calculations<sup>29</sup> have shown that  $\text{hpp}^-$  is a very strong Brønsted base, being 100 times more basic than tetramethylguanidinate.
2. A rigid framework is formed from constraining the nitrogen substituents into the ring, leading to decreased rotational freedom about C-N bonds and no isomerization of the C=N double bond.

### Guanidines as Catalysts in Organic Synthesis

Ionic bases have been known for centuries and are extensively used in organic synthesis.<sup>30,31</sup> However non-ionic nitrogen bases have not achieved the same degree of use. The simplicity of handling and mildness of reaction conditions render non-ionic bases excellent tools for generating carbanions for interesting and useful organic transformations. A large variety of non-ionic nitrogen bases with low to very high steric hindrance and weak to medium base strengths are available (Figure 6). Various sterically hindered amines and amidines acts as weak nucleophile and are widely used as proton acceptors in organic synthesis.<sup>32</sup>

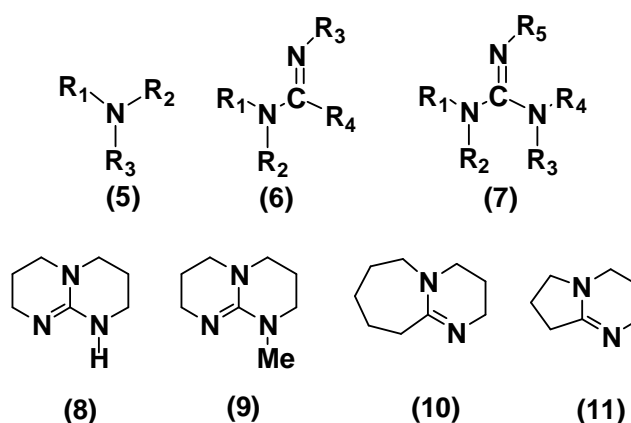


Figure 6: Various Organobases: (5) Amines, (6) Amidines, (7) Guanidines, (8) hppH, (9) Me-hpp, (10) DBU, and (11) DBN.

Stronger amidine base such as DBN are extensively used in dehydrohalogenation reactions<sup>33</sup>. The bicyclic guanidine bases hppH and Me-hpp are known as superbases because of their high pK<sub>a</sub> values<sup>34</sup>. The bicyclic hppH has been extensively used as a catalyst in several organic reactions such as Michael additions<sup>35</sup>, nitroaldol (Henry

reactions)<sup>36</sup>, Horner-Wadsworth-Emmons<sup>37</sup>, ring-opening polymerization of cyclic esters<sup>38</sup>, and transesterification reactions.<sup>39</sup> Various chiral bicyclic guanidines are also used as catalysts in various asymmetric reactions such as the diastereoselective Henry reaction<sup>40</sup>, enantioselective synthesis of  $\alpha$ -amino nitriles<sup>41</sup>, and asymmetric trimethylsilylcyanation of carbonyl compounds.<sup>42</sup>

### Bicyclic Guanidine Units in Natural Products

The guanidine unit is very important to life on earth as it forms the core of the DNA base guanine (Figure 7). Bicyclic guanidines are very important as natural guanidine derivatives are abundant in natural products. The guanidine unit is present in several structurally novel marine alkaloids within complex polycyclic architectures such

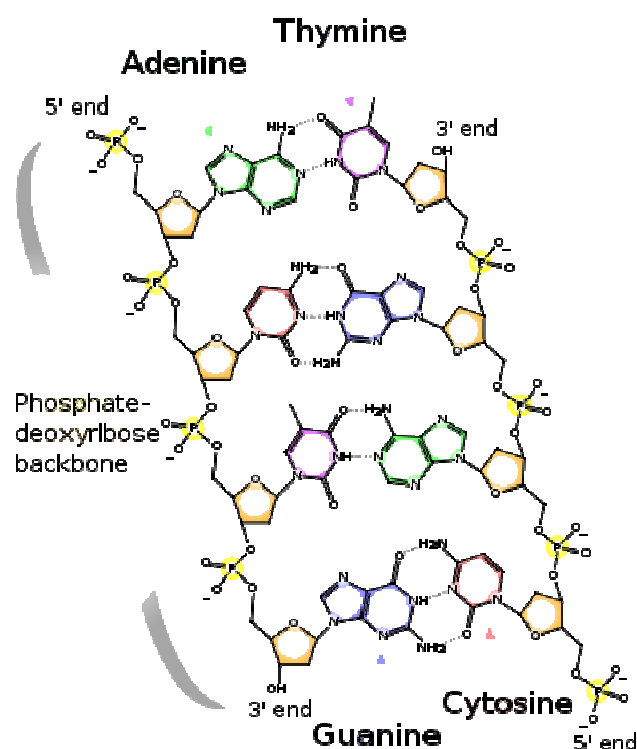
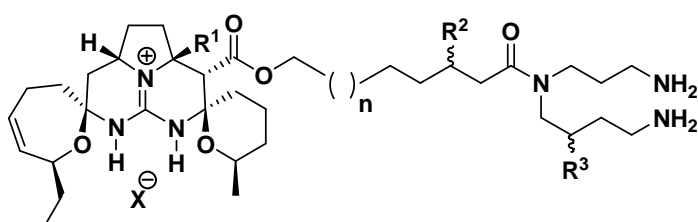


Figure 7: Base pairs in DNA double-helix structure

as the crambescidin/ptilomyalin and batzelladin families (Figure 8).<sup>43</sup> These guanidine alkaloids exhibit promising anti-cancer and anti-viral activities.<sup>44,45</sup> As shown in Figure 6, all of these natural products incorporate the hppH core. The complex nature of crambescidins and batzelladins and their compelling biological activities have sparked intense effort toward their total chemical synthesis.



- Ptilomyalin A** ( $R^1 = R^2 = R^3 = H$ ;  $n = 10$ )  
**Crambescidin 800** ( $R^1 = R^2 = H$ ,  $R^3 = \alpha\text{-OH}$ ;  $n = 10$ )  
**Crambescidin 816** ( $R^1 = OH$ ,  $R^2 = H$ ,  $R^3 = \alpha\text{-OH}$ ;  $n = 10$ )  
**Crambescidin 844** ( $R^1 = R^3 = OH$ ,  $R^2 = H$ ;  $n = 13$ )  
**Celeromyalin** ( $R^1 = R^3 = H$ ,  $R^2 = \beta\text{-OH}$ ;  $n = 10$ )

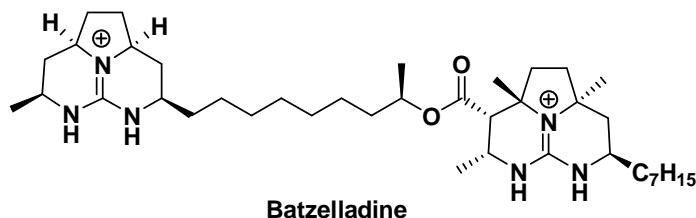


Figure 8: Guanidine alkaloids from marine sponges.

### Applications of Paddlewheel Complexes

Even though complexes with metal-metal multiple bonds were discovered in 1965, the potential applications of these complexes have not been investigated in detail. Paddlewheel complexes with metal-metal multiple bonds have been used in a variety of diverse applications such as:

1. Several complexes of Mo with foramidinate ligands are connected with several one-dimensional linkers for applications in material science and supramolecular structures.<sup>46</sup>
2. Complexes such as  $\text{Rh}_2(\text{O}_2\text{CR}_4)\text{L}_2$  (R = Me, Et, Pr; L = solvent) have potential medicinal applications as they bind to base pairs in DNA.<sup>47</sup>
3. Chiral dirhodium complexes with orthometallated aryl phosphine ligands act as catalysts in enantioselective C-H insertions of  $\alpha$ -diazoketones.<sup>48</sup>
4. Several paddlewheel complexes of Mo carboxylates exhibit liquid crystal phases and have interesting optical, magnetic, and electronic properties.<sup>49</sup>

#### Disadvantages and Challenges of $\text{hpp}^-$ Ligand:

As discussed above,  $\text{hpp}^-$  ligand has been a very robust and versatile ligand for metal-metal multiple bonded complexes. But, a major disadvantage is that these complexes are not very soluble in common organic solvents,<sup>18,28</sup> thereby limiting their solution characterization and use in organic synthesis as catalysts. Also attempts to make paddlewheel complexes of Ti, Zr, Hf and Ta have so far been unsuccessful.<sup>50</sup>

#### Thesis Overview

The main aim of this dissertation is to design and synthesize derivatives (both achiral and chiral) of  $\text{hppH}$  which not only make the complexes more soluble but also make them more basic. An obvious question is what would be the best position on the  $\text{hpp}^-$  ring (C1, C2 or C3; Figure 9) which would not only make them more soluble but also render the ligand more basic? Chapter 2 deals with quantum mechanical calculations to determine the electronic and steric effects of various substituents on the basicity of  $\text{hpp}^-$  derivatives. These calculations also show that substituting only one methyl group at C1 position makes the ligand most basic. A derivative with four methyl substituents at the C2 positions was also chosen in order to test synthetic methodology.

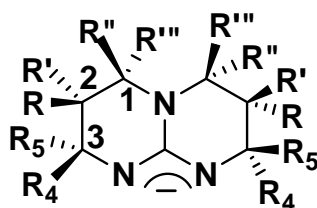


Figure 9: Substituents of various carbons on the  $\text{hpp}^-$  ring system

Chapter 3 deals with the synthesis of the achiral tetramethyl derivative of  $\text{hppH}$  (3,3,9,9-tetramethyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene; denoted as  $\text{hpp}^*\text{H}$ ). The  $C_2$  symmetric bicyclic guanidine (2-(S),10-(S)-2,10-dimethyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (denoted as  $\text{hpp}'\text{H}$ ) should eliminate the formation of several isomers during the formation of paddlewheel complexes (Chapter 4). The beauty of the  $\text{hpp}'\text{H}$  ligand is that it yields only one isomer when four ligands react to form an  $\text{M}_2\text{L}_4$  complex. Derivatives of  $\text{hppH}$  with substituents at C3 might lead to metallation during the formation of dinuclear complexes. Chapter 4 discusses the synthesis and characterization of this chiral  $C_2$  symmetric  $\text{hpp}'\text{H}$  ligand. Chapter 5 describes the coordination chemistry of  $\text{hpp}^-$  with Ta.

## CHAPTER 2

## COMPUTATIONAL STUDIES

As discussed in the last part of Chapter 1, complexes of hppH are often sparingly soluble, impacting the study of their reactivity in solution. Attempts to make complexes of  $\text{hpp}^-$  with a Ta-Ta triple bond have so far been unsuccessful. Paddlewheel  $\text{hpp}^-$  complexes of Zr, Ti and Hf are unknown. Paddlewheel complexes of Zr with a metal-metal multiple bonds could be highly reactive because of the larger HOMO and LUMO gap for the lower oxidation states.

Derivatives of  $\text{hpp}^-$  with aliphatic side groups could increase the solubility and could also be favorable electronically for stabilizing metals in middle oxidation states. These factors led to the question: where to place substituents on the ring (C1, C2 or C3, Figure 10a) and which substituents would be the best for ligand basicity. To answer this question, we turned to computational methods. This chapter deals with calculating the basicities of various substituted bicyclic guanidinate (Figure 10a).

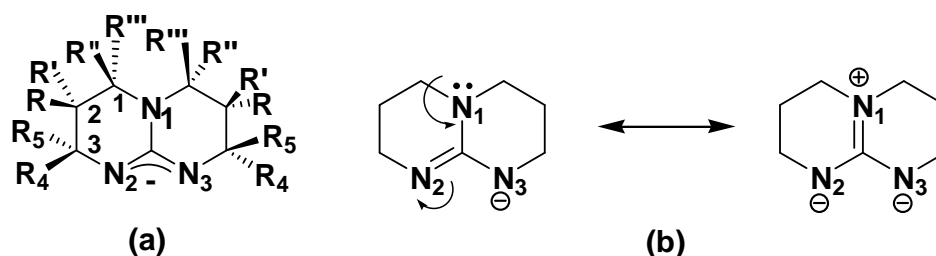
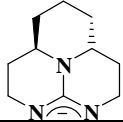


Figure 10: (a) Substituted bicyclic guanidinate ligand and (b) electronic delocalization in guanidinate core of  $\text{hpp}^-$  ligand.

The following table (Table 1) shows the various groups (substituents at R, R', R'', R''', R<sub>4</sub> and R<sub>5</sub> positions) used to study steric and electronic effects on the basicities.

Table 1: Various substituents on hppH used to calculate steric and electronic effects.

	<b>R</b>	<b>R'</b>	<b>R''</b>	<b>R'''</b>	<b>R<sub>4</sub></b>	<b>R<sub>5</sub></b>
1.	H	H	H	H	H	H
2.	Me	Me	H	H	H	H
3.	H	H	Me	Me	H	H
4.	<i>iso-Pr</i>	H	H	H	H	H
5.	<i>tert-Bu</i>	H	H	H	H	H
6.	H	H	<i>iso-Pr</i>	H	H	H
7.	H	H	<i>tert-Bu</i>	H	H	H
8.	H	H	Me	H	H	H
9.	Me	H	H	H	H	H
10.			PhCH <sub>2</sub>	H	H	H
11.	H	H	CH <sub>2</sub> CH(CH <sub>3</sub> ) <sub>2</sub>	H	H	H
12.	H	H	CH(CH <sub>3</sub> )CH <sub>2</sub> CH <sub>3</sub>	H	H	H
13.	H	H	OMe	H	H	H
14.	H	H	SiMe <sub>3</sub>	H	H	H
15.	H	H	SMe	H	H	H
16.	H	H	F	H	H	H
17.	H	H	CF <sub>3</sub>	H	H	H
18.	N	←→	P		H	H
19.						
20.	H	H	H	H	Me	H
21.	H	H	H	H	Me	Me
22.	H	H	H	H	<i>iso-Pr</i>	H
23.	H	H	H	H	<i>tert-Bu</i>	H
24.	Me	H	Me	H	H	H
25.	H	Me	Me	H	H	H
26.	H	H	Me	H	Me	H
27.	H	H	Me	H	H	Me
28.	N	←→	P	Me	H	H
29.	H	H	H	H	OMe	H
30.	H	H	H	H	SiMe <sub>3</sub>	H
31.	H	H	H	H	SMe	H
32.	H	H	H	H	F	H
33.	H	H	H	H	CF <sub>3</sub>	H

Thus calculations will give us some idea about the position of substituents on the ring (both in terms of sterics and electronics), the effect of P and N substitution, and help us to select the appropriate ligand for synthesis.



### Computation Background

There are several computational tools available to chemists to understand molecular geometry and conformation. The simplest and most commonly used is molecular mechanics<sup>51</sup>. Molecular mechanics uses Newtonian/classical mechanics to model molecular systems. The potential function computes the molecular potential energy as a sum of energy terms that describe the deviation of bond lengths, bond angles and torsion angles away from equilibrium values, plus terms for non-bonded pairs of atoms describing van der Waals and electrostatic interactions. As a consequence, molecular mechanics may not be used to calculate the relative energies of different (isomeric) molecules or reaction energies, except in cases where the bonding is identical. The molecular mechanics approach is simple and often predicts a molecule's equilibrium geometry with very little computational effort. Its major drawback is the need for explicit parameterization. Molecular mechanics cannot be used to study reaction pathways connecting stable structures, particularly in locating energy maxima (transition states) along these pathways. In these situations, molecular orbital (or quantum mechanical) calculations are used explicitly and for other calculations as well.

### Ab Initio Molecular Orbital Models

The majority of molecular orbital methods (or quantum mechanical methods) solve an approximate solution to the Schrödinger equation.

$$\hat{H}\psi = E\psi$$

In this equation, the Hamiltonian operator,  $\hat{H}$  describes both the kinetic energies of the particles (i.e. electrons and nuclei) of the molecule, and the electrostatic interactions (nuclei, which are positively charged, repel other nuclei, and electrons, which are negatively charged, repel other electrons, but nuclei attract electrons) experienced between individual particles. The quantity  $E$  in the Schrödinger equation is the energy of

the system, and  $\psi$  is the wave function. The square of the wave function corresponds to the probability of finding the particles at a particular set of coordinates. The Schrödinger equation has been solved exactly for the hydrogen atom (a one-electron system). Although the Schrödinger equation can be written for many-electron systems, it cannot be solved exactly. Approximations must therefore be made in order to solve it for more complex systems. Most of the “*ab initio*” (Latin word for ‘from the beginning’) molecular orbital methods start from the Schrödinger equation and then make three approximations:

1. The separation of nuclear and electron motions, or the Born-Oppenheimer approximation. This assumes that nuclei are stationary as compared to electrons.
2. The separation of electron motions, the Hartree-Fock approximation. This assumes many-electron wave functions as a sum of products of one-electron wave functions.
3. The representation of the individual molecular orbitals in terms of linear combinations of atom-centered basis functions or atomic orbitals (also known as the LCAO approximation).

Practical *ab initio* methods differ in the number and kind of atomic basis functions, and their computational cost increases as the fourth power of the number of basis functions (compared to a square power dependence on the number of atoms for molecular mechanics techniques). The simplest *ab initio* method uses a “minimal basis set” of atomic orbitals, which includes only those functions required to represent all electrons on an atom and to maintain spherical symmetry. The most commonly used basis sets in *ab initio* methods are “split-valence basis sets”<sup>52</sup>. The split-valence basis sets include multiple basis functions corresponding to each valence atomic orbital, and are called valence double, triple, or quadruple-zeta basis sets. These basis sets are designated by ‘Pople’ notation, typically as  $X\text{-}YZg$ . In this case,  $X$  represents the number of primitive Gaussians comprising each core atomic orbital basis function. The two numbers after the hyphens specifies that the basis set is a *split-valence double-zeta* basis set. The  $Y$  and  $Z$  indicate that the valence orbitals are composed of two basis functions each, the first one composed of a linear combination of  $Y$  primitive Gaussian functions, the other composed of a linear combination of  $Z$  primitive Gaussian functions. The most common Pople notations which will be used here are 3-21G and 6-31G\*, where \* designates polarized basis sets. 3-21G means a basis set in which inner-shell atomic orbitals are represented in

terms of three Gaussian functions, and each valence-shell atomic orbital is split into two parts, written in terms of two and one Gaussians, respectively. 6-31G\* represents a basis set in which inner-shell atomic orbitals are represented in terms of six Gaussian functions, and each valence-shell atomic orbital is split into two parts represented in terms of three and one Gaussians, respectively. *Ab initio* molecular methods using split-valence or polarization basis sets have now been commonly used to describe structures, stabilities and other properties of organic molecules. They have also been applied with considerable success to describe reaction pathways and to elucidate product distributions.

### Semi-Empirical Molecular Orbital Models

The principal disadvantage of *ab initio* methods is their computational cost and time. It is possible to introduce further approximations in order to reduce these costs significantly while still retaining the quantum mechanical formalism. Semi-empirical quantum mechanical methods are based on the Hartree-Fock formalism but make some approximations and obtain some parameters from empirical data. Semi-empirical methods of calculation use the following key approximations:

1. The elimination of overlap between functions of different atoms. This drastically reduces the computation effort by more than an order of magnitude over *ab-initio* methods.
2. Inner-shell (core) functions are not included explicitly. The cost of calculation involving a second-row element, e.g., silicon, is no more than that incurred for the corresponding first-row element, e.g., carbon.
3. Approximations to further simplify the calculations, varying among the different semi-empirical methods. The choice of parameters is the key to the success of semi-empirical methods.

Semi-empirical calculations are much faster than their *ab initio* counterparts. However, their results can be different if the molecule being computed is not similar enough to the molecules used to parameterize the method. Semi-empirical calculations have been most

successful in the description of organic molecules with only a few elements and molecules with moderate size.

### Which Models are Most Suitable?

Which of the available computational tools mentioned above are the most appropriate? The answer depends on: 1) the problem at hand, 2) the level of confidence required in the results, and 3) available computational resources. The following four methods were used for calculations in this chapter: AM1 and AM1-SM2 semi-empirical models and *ab initio* models using 3-21G (split-valence) and 6-31G\* (polarization basis sets). All four of these methods have become standards for calculations on organic systems. Molecular mechanics have been used rarely. Molecular orbital methods are preferred because they are more general and reliable. Because of its low cost, molecular mechanics is used particularly for preliminary refinement of geometry and, most importantly, for conformational searching.

**AM1 (Austin Model 1):** This is the most popular semi-empirical method for application to organic systems. It provides a good account of equilibrium structures and usually reproduces transition-state geometries obtained from higher-level *ab initio* calculations. However, it is less reliable than *ab-initio* methods in dealing with the geometries of molecules with second-row (and heavier) elements. AM1, though, does not provide an acceptable account of reaction thermochemistry, however, they are used to provide geometries used for higher-level calculations of energies.

**AM1-SM2:** This semi-empirical method is based on AM1 and is used to calculate aqueous heats of solvation. It is parameterized to reproduce experimental heats of (aqueous) solvation. The method works well for neutral organic molecules but often leads to errors for charged species and heavier atoms.

**3-21G:** This is the simplest *ab initio* model that affords equilibrium and transition state geometries as well as energetics of reactions that do not involve bond forming and bond breaking.

**6-31G\*:** This polarization basis, like 3-21G, is more successful in predicting equilibrium and transition state geometries. It is more accurate than the superior account 3-21G method.

### Computation Methodology

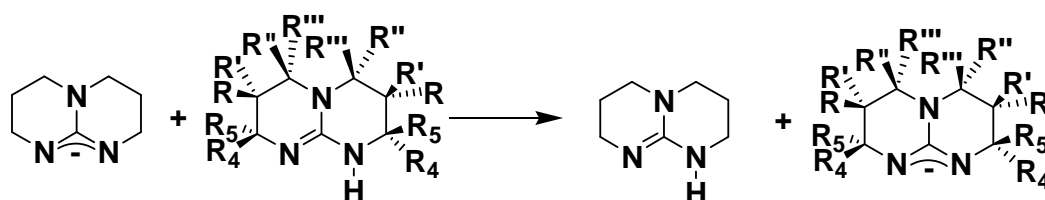
#### Spartan

Spartan'02 was used to calculate the basicities of various substituted bicyclic guanidines. The neutral substituted guanidines as well as their deprotonated forms were first optimized using AM1 semi-empirical calculations (starting from molecular mechanics optimized geometries). A better estimate of gas phase basicities was made by doing single-point energy calculations using 3-21G and 6-31G\* *ab-initio* methods (using the AM1 geometries). No other constraints were applied and no other calculations were done.

### Results and Discussion

The geometries of the various substituted bicyclic guanidines were optimized using the semi-empirical AM1 calculations. A better estimate of the basicities was obtained by doing single-point 3-21G and 6-31G\* calculations. In order to determine the effect of solvent on the basicities, AM1-SM2 calculations were also done.

The basicities of various substituted bicyclic guanidines were calculated by using the following isodesmic reaction (reaction in which the reactants and products have exactly the same numbers of each kind of formal chemical bond and each kind of formal nonbonded lone pair, Equation 1).



Equation 1: Isodesmic reaction for calculating the effect of substituents on the basicities of various substituted hpp<sup>-</sup> derivatives

$\Delta H$  of the above reaction was computed by using the equation:

$$\Delta H_m = \sum H_f(\text{products}) - \sum H_f(\text{reactants})$$

For the above isodesmic reaction, it can be assumed that the entropy differences are very small, and that the solvation effects will mostly cancel for different bicyclic guanidines. Thus, it can be assumed that the experimental relative free energies of protonation ( $\Delta G$ ) in solution closely approximate the relative enthalpies of protonation ( $\Delta H$ ) in the gas phase. The energy values therefore represent the  $\text{pK}_a$  values of the bicyclic guanidines. The more positive the value, the more basic is the bicyclic guanidine, and the more negative value means more acidic nature of the bicyclic guanidine. The energies computed by *ab initio* methods (3-21G and 6-31G\*) were obtained in Hartree units and were converted to kcal/mol by multiplying by 627.509; the resulting number was rounded off to two decimal places for simplicity.

The relative basicities of various substituted guanidines were calculated from different levels of calculations and are tabulated in Table 2 below. The key observations are:

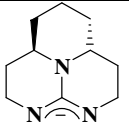
1. There is some trend in the relative basicities obtained from AM1 calculations. For example, the basicities decrease as bulky groups are substituted (entries 4, 5, 9 and 20, 22 and 23 in Table 2). The basicity decreases when bulky groups are placed on the rings (this is expected because bulky groups lead to more puckering of the ring, thus affecting the delocalization (Figure 10b)). However, the reversal trend in case of entries 8, 6 and 7 could not be explained. Therefore single-point 3-21G and 6-31G\* energies (optimized at the AM1 level) were calculated in order to get more accurate and credible numbers. The results from 3-21G follow the same trend as seen for 6-31G\* calculations. All subsequent discussions will be based on the results of 6-31G\* calculations. Table 3 lists the basicities of various substituted ligands as calculated from the 6-31G\*//AM1 calculations.

Table 2:  $\Delta H_m$  (enthalpy) calculated for Equation 1 from different levels of calculations.

	<b>AM1 (kcal/mol)</b>	<b>AM1-SM2 (kcal/mol)</b>	<b>3-21G//AM1 (kcal/mol)</b>	<b>6-31G*//AM1 (kcal/mol)</b>
1				
2	-0.58	2.55	-1.62	-1.61
3	0.16	2.96	2.70	2.87
4	-0.97	2.98	-628.38	-0.50
5	-1.25	3.59	-1.85	-1.38
6	-1.99	5.36	-4.52	-4.92
7	-0.66	6.42	-5.54	-5.29
8	0.72	1.38	0.11	1.03
9	-0.19	1.36	0.90	1.17
10	-26.79	6.2	1.96	1.62
11	1.14	2.92	2.10	1.00
12	24.39	4.73	1.24	0.50
13	-4.21	5.72	-3.08	-2.40
14	-1.22	-	1.09	1.94
15	-10.84	0	-11.79	-6.92
16	-7.36	5.31	-10.12	-9.37
17	-15.56	13.06	-12.61	-10.41
18	-1.16	-	-0.41	2.17
19	0.91	0.97	1.75	1.68
20	0.79	4.53	-1.34	-0.79
21	1.26	8.81	-3.23	-2.63
22	-0.46	8.83	-5.77	-5.01
23	-1.45	24.52	-642.22	-4.97
24	0.32	1.43	0.50	0.90
25	0.24	3.05	0.09	0.16
26	1.18	4.96	-1.16	-0.66
27	1.91	-	-1.88	-2.69
28	-3.3	-	1.35	2.17
29	-7.61	10.51	-13.65	-11.79
30	-3.75	-	-10.84	-8.12
31	-20.41	16.33	-24.92	-16.47
32	-10.34	4.5	-17.16	-15.74
33	-24.12	16.17	-22.10	-19.98



Table 3:  $\Delta H_{rn}$  (enthalpy) calculated for Equation 1 from 6-31G\*\*//AM1 calculations.

	<b>R</b>	<b>R'</b>	<b>R''</b>	<b>R'''</b>	<b>R<sub>4</sub></b>	<b>R<sub>5</sub></b>	<b>6-31G**//AM1 (kcal/mol)</b>
1	H	H	H	H	H	H	
2	Me	Me	H	H	H	H	-1.61
3	H	H	Me	Me	H	H	2.87
4	<i>iso-Pr</i>	H	H	H	H	H	-0.50
5	<i>tert.-Bu</i>	H	H	H	H	H	-1.38
6	H	H	<i>iso-Pr</i>	H	H	H	-4.92
7	H	H	<i>tert.-Bu</i>	H	H	H	-5.29
8	H	H	Me	H	H	H	1.03
9	Me	H	H	H	H	H	1.17
10			PhCH <sub>2</sub>	H	H	H	1.62
11	H	H	CH <sub>2</sub> CH(CH <sub>3</sub> ) <sub>2</sub>	H	H	H	1.00
12	H	H	CH(CH <sub>3</sub> )CH <sub>2</sub> CH <sub>3</sub>	H	H	H	0.50
13	H	H	OMe	H	H	H	-2.40
14	H	H	SiMe <sub>3</sub>	H	H	H	1.94
15	H	H	SMe	H	H	H	-6.92
16	H	H	F	H	H	H	-9.37
17	H	H	CF <sub>3</sub>	H	H	H	-10.41
18	N ← → P				H	H	2.17
19							1.68
20	H	H	H	H	Me	H	-0.79
21	H	H	H	H	Me	Me	-2.63
22	H	H	H	H	<i>iso-Pr</i>	H	-5.01
23	H	H	H	H	<i>tert.-Bu</i>	H	-4.97
24	Me	H	Me	H	H	H	0.90
25	H	Me	Me	H	H	H	0.16
26	H	H	Me	H	Me	H	-0.66
27	H	H	Me	H	H	Me	-2.69
28	N ← → P		Me	H	H	H	2.17
29	H	H	H	H	OMe	H	-11.79
30	H	H	H	H	SiMe <sub>3</sub>	H	-8.12
31	H	H	H	H	SMe	H	-16.47
32	H	H	H	H	F	H	-15.74
33	H	H	H	H	CF <sub>3</sub>	H	-19.98

2. The effect of solvation on the basicities was determined from AM1-SM2 calculations. Spartan could not calculate the solvation energies when the atoms Si, P and S were present in the system (because the AM1-SM2 model does not work for second row elements). From AM1-SM2 calculations, it could be inferred that all of the bicyclic guanidines are more basic than hppH. However, calculations do not follow normal trends. For example, substituting bulky alkyl groups or electron withdrawing groups should decrease the basicity (electron withdrawing groups makes N more electron deficient, while bulky groups pucker the ring, both affecting the delocalization, Figure 10b) but the opposite trend is observed. The reason for this could be that the solvent model is not very good and therefore could not give better estimates of basicities in water.
3. From 6-31G\*\*/AM1 calculations, it can be inferred that some bicyclic guanidines are more basic than hppH (entries 3, 8-12, 24, 25 and 28), while in some cases they are less basic than hppH.
4. 6-31G\*\*/AM1 calculations showed that substituting groups at C1 (Figure 10a) made the guanidines more basic than substituting groups at C2 and C3. C2 is better for substitution than C3, perhaps because C1 is much closer to the N1 nitrogen involved in delocalization into the guanidine core (Figure 10b).
5. Entries 2-12 and 20-23 were the result of calculations to determine steric effects on the basicities of the ligand. Bulky alkyl groups should decrease the basicity of the guanidine because of puckering of the ring system that affects the delocalization of the amine lone pair into the core of the ligand (Figure 10b, entries 9, 4, 5, 6, 7 and 20, 22 and 23). This can be explained by the puckering of the rings as shown by a comparison of the dihedral angles (Tables A3 and A4, Appendix A) vs. hppH. For example, substituting *iso*-propyl and *tert*-butyl groups (entries 4 and 5) changes the dihedral angles C1(N1)C3(C5) from -22.8 (entry 1; hppH) to 42.5 (entry 4) and to 43.3 (entry 5). Similarly, dihedral angle C3(C5)C7(N3) changes from -45.2 (entry 1; hppH) to 41 (entry 4) and to 40.9 (entry 5). In these cases, the change in dihedral angle is more for *tert*-butyl group, as would be expected based on sterics.
6. When bulky groups are substituted at the C2 position, the decrease in basicity is less as compared to when they are substituted at C1. This trend is also expected because

when groups are substituted at the C2 position, they are further from each other. This argument is supported by comparing dihedral angles. The dihedral angle N2(C1)N1(C3) changes from -2.4 (entry 1) to -14.7 and -15.3 (entries 4 and 5). For entries 6 and 7, the dihedral angle changes more (23.6 and 30.2). The dihedral angle C5(C7)N3(C1) changes from 23.2 (entry 1) to -15.9 for entries 4 and 5 and then to -33.7 and 0.6 (entries 6 and 7). Similar trends are observed when substituents are placed on C3.

7. Placing two methyl groups at C1 (entry 3) makes the bicyclic guanidine more basic than hppH, as compared to substituting at C2 (entry 2). This could be the result of the positive inductive effect of a methyl group, which makes the nitrogen next to it more electron rich. This is confirmed by the electrostatic charges at the three nitrogens N1, N2 and N3 (Figure 10a). The electrostatic charge (Table A5, Appendix A) decreases from -0.59 Coulombs (entry 1) to -0.51 Coulombs for entry 2 but it increases to -1.15 Coulombs for entry 3. The charge on N1 for entry 3 is the largest amongst all the other bicyclic guanidines, and the derivative represented by entry 3 is therefore the most basic bicyclic guanidine. The greater the charge on N1, the more charge it can delocalize into the bicyclic guanidine core.
8. Placing only one methyl group at C1 or C2 (entries 8 and 9) does not make much difference in the basicity. This guanidine is more basic than hppH but less basic than a guanidine with two methyl groups at C1 (entry 3). For entry 8 (one Me group at C1), the electrostatic charge on N1 increases from -0.59 to -0.8, thus rendering the bicyclic guanidine more basic.
9. Placing other substituents at C1 effects the basicity of the bicyclic guanidines. Bulky groups (e.g. *iso*-propyl, entry 6; *tert*-butyl, entry 7) decrease the basicity because of puckering of the bicyclic rings. A benzyl group (entry 10) increases the basicity. The electrostatic charge for entry 10; increases from -0.59 in hppH to -0.83 supporting the idea of greater basicity.
10. Substituting N1 of the bicyclic guanidine core by phosphorus (more electropositive than N, entries 18 and 28), increases the basicity as expected.

11. Entries 13-18 and 29-33 were studied in order to determine electronic effects on the basicity of bicyclic guanidines. The electrostatic charges on N1, N2 and N3 (Figure A1, Appendix A) are given in Table A5 (Appendix A). The major effect of the charge is seen on N1. As expected, when electron withdrawing groups are placed (entries 13, 16 17; 29, 32 and 33) the basicity should decrease, and it follows the trend. This trend is supported by the fact that the electrostatic charge on N1 also decreases. For example, electrostatic charge decreases from -0.59 (entry 1, H) to -0.47 (entry 13; OMe), -0.54 (entry 16; F) and to -0.3 (entry 17; CF<sub>3</sub>). The electron donating substituent SiMe<sub>3</sub> (entry 14) increases the charge on N1 from -0.59 in hppH to -0.72, making the derivative more basic than hppH. A similar trend is observed for entries 29 (OMe), 30 (SiMe<sub>3</sub>), 32 (F) and 33 (CF<sub>3</sub>).
12. Entries 20-23 show the effects of substituents at the C3 position. Entry 20 (Me at C3) is similar to entry 8 (Me at C1) except that substituents are closer to N2 and N3. The charges on N2 and N3 are -0.98 and -0.97 Coulombs in entry 20, are higher than in entries 8 and 1. Thus, if electrostatics is considered, this guanidine (Me at C3) should be more basic than both entry 8 (Me at C1) and 1 (H, hppH). But instead basicity values suggest that it is weaker base than both entry 8 and entry 1. The explanation for this may lie in the dihedral angles. Dihedral angle N2(C1)N1(C3) changes from -11.4 in entry 8 (Me at C1) to -3 in entry 20 (Me at C3), whereas dihedral angle C1(N1)C3(C5) changes from 38.2 to -22.3. Dihedral angle C5(C7)N3(C1) changes from -16.2 in entry 8 (Me at C1) to 23.5 in entry 20 (Me at C3). Because of greater puckering of the rings, delocalization is affected and the basicity decreases.
13. Calculations on entries 24-27 were used to evaluate the additive effect of substituents at C1 (and C3) and C1 (and C2). On comparing the numbers for entry 24 (energy of entry 8 + energy of 9 = 2.12 kcal/mol; calculated energy is 0.84 kcal/mol) and entry 26 (energy of entry 8 + energy of entry 20 = 0.24 kcal/mol; calculated energy is only -0.66 kcal/mol), it can be inferred that the effects are not additive.

### Conclusions

1. The basicity of bicyclic guanidines varies with substituents on the carbon backbone. The effect is greatest if the substituents are at C1 (i.e., carbon next to quaternary nitrogen N1).
2. The change in the basicity depends on the nature of the substituents. Bulky substituents lead to less basicity (i.e., sterics plays an important role). This is supported by the change in dihedral angles of the rings upon adding substituents.
3. Electrostatics also effect the basicity as seen in the case of electron donating and electron withdrawing groups. This is buttressed by the electrostatic charges calculated on the nitrogens of the bicyclic guanidinate core. Electron withdrawing groups decrease the basicity, whereas electron donating groups increase the basicity, as compared to hppH.
4. The effect of substituents on basicity is not additive as seen in the examples where two substituents are placed on separate carbons.
5. Based on these calculations, placing two Me groups at C1 (entry 3) makes the bicyclic guanidine more basic than hppH.
6. Placing only one Me group at C1 makes the bicyclic guanidine more basic than hppH (and is similar to substituting at C2). These calculations informed our synthetic targets. These calculations also show that placing a benzyl group at C1 is better than placing a Me group at C1 in terms of higher basicity.

## CHAPTER 3

SYNTHESIS OF 3,3,9,9-TETRAMETHYL-1,5,7-  
TRIAZABICYCLO[4.4.0]DEC-5-ENE

Bicyclic guanidines are a class of substituted guanidines in which the carbon of the central  $CN_3$  unit is incorporated into a bicyclic framework fused along one C-N bond,

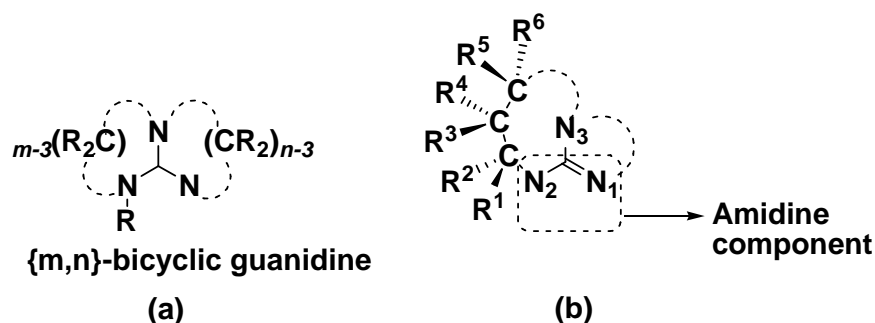


Figure 11: General structure of bicyclic guanidines.

as shown in Figure 11<sup>53</sup>. They exhibit distinctive physical, electronic and chemical characteristics compared to acyclic analogues. The nomenclature of these bicyclic guanidines is very complicated, so it is convenient to describe these compounds as  $\{m,n\}$ -bicyclic guanidines [Figure 11 (a)], with  $m$  and  $n$  defining the size of the component heterocyclic rings.

HppH (Figure 12) is the only bicyclic guanidine available commercially. Syntheses of non-functionalized bicyclic guanidines are based on either multi-step procedures<sup>54</sup> or use expensive starting materials.<sup>55</sup> A simple one-pot procedure to the  $\{6,6\}$  and  $\{5,5\}$  guanidines, Hhpp and Htbo (2,3,5,6-tetrahydro-1*H*-imidazole[1,2*a*]imidazole, Figure 12) was published by A'Court in a patent<sup>56</sup> in 1986.

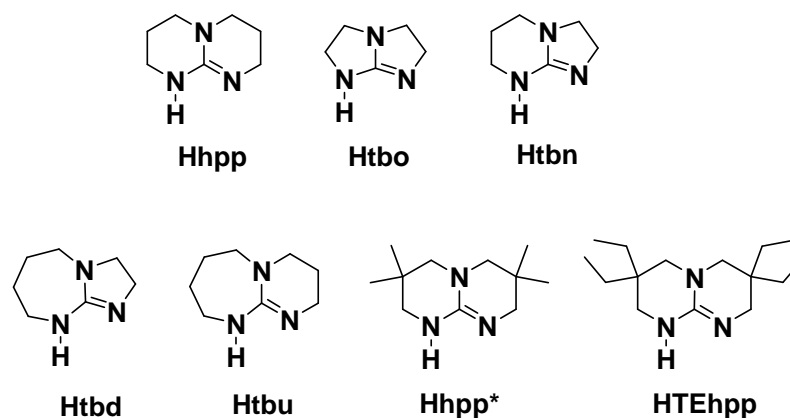
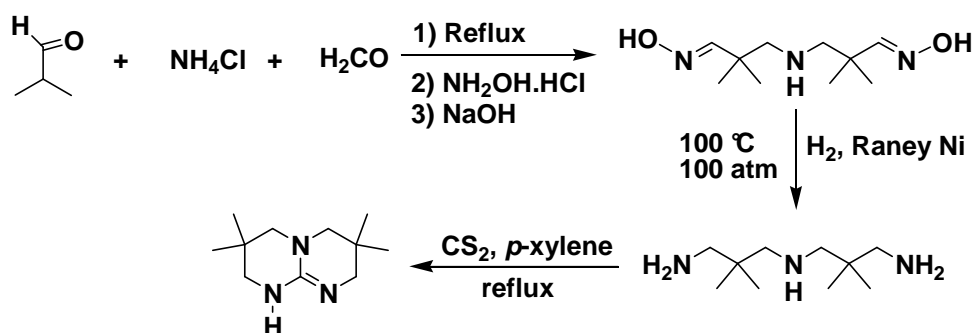


Figure 12: Derivatives and abbreviations for bicyclic guanidines.

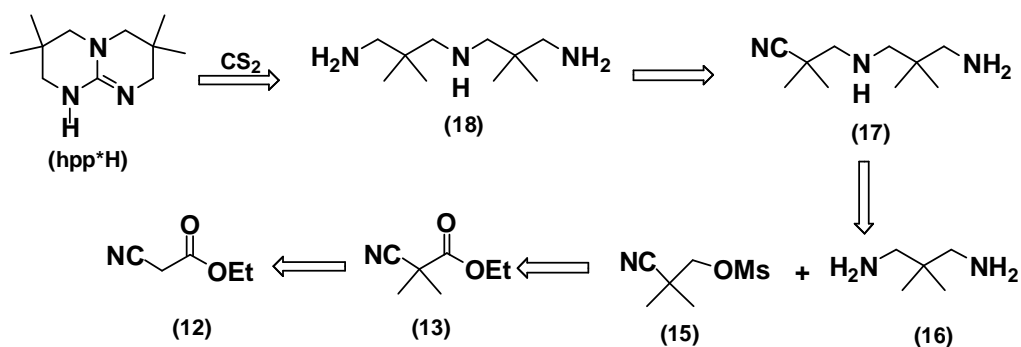
This synthetic procedure was extended to the non-symmetric {6,5}-bicyclic H-tbn (2,3,5,6,7,8-hexahydroimidazo[1,2-*a*]pyrimidine by Cotton and co-workers.<sup>57</sup> A multi-step synthetic procedure to non-functionalized guanidines containing seven-membered rings, mainly Htbd (2,5,6,7,8,9-hexahydro-3H-imidazo[1,2-*a*][1,3]diazepine and H-tbu (2,3,4,6,7,8,9,10-octahydropyrimido[1,2-*a*]-[1,3]diazepine was published at the same time.<sup>58</sup> The study of metal complexes of anions of Htbo, Htbn, Htbd and Htbu<sup>57,58</sup> has showed that the ring sizes in the ligands effect the oxidation potential of the corresponding dimolybdenum complexes. From this study it was apparent that complexes with hpp<sup>-</sup> ligands are the most easily oxidized in solution. Thus the hpp<sup>-</sup> ligand (with two fused six membered rings) is the most basic ligand among the various non-functionalized bicyclic guanidines. Unfortunately, hpp<sup>-</sup> complexes have low solubility in common organic solvents. Therefore, derivatives of hppH with aliphatic side chains should enhance the solubility without altering the basicity.<sup>59</sup> This chapter discusses the synthesis of tetramethyl hppH (3,3,9,9-tetramethyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene; termed as hpp\*H in this chapter). The synthesis of hpp\*H (and also tetraethyl hppH named as TEhpp, Figure 12) was published by Cotton and co-workers<sup>59</sup> during the course of our research. Cotton's synthesis of hpp\*H is shown in Scheme 1 below.



Scheme 1: Synthesis of hppH\* as reported by Cotton and co-workers.

### Retrosynthetic Analysis

This chapter discusses an alternate route to hpp\*H. A retrosynthetic analysis of hpp\*H is given in Scheme 2 below.



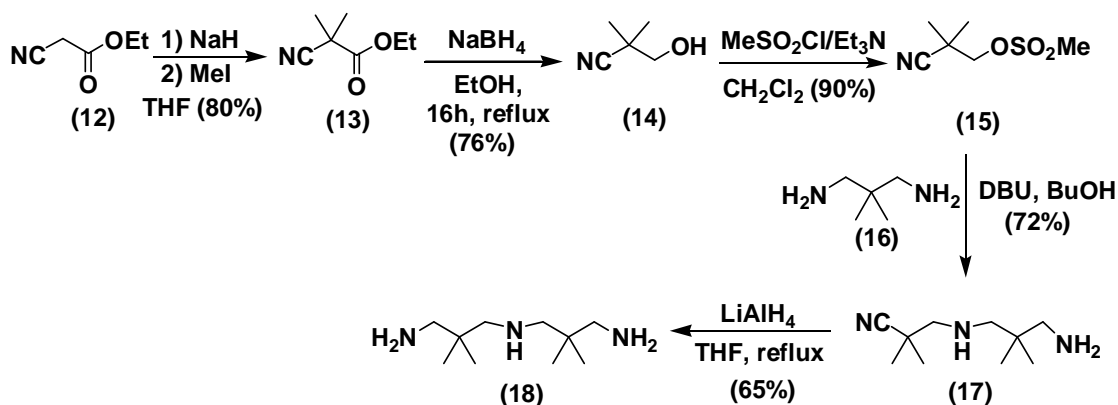
Scheme 2: Retrosynthetic analysis of hppH\*.



Retrosynthetically, hpp\*H can be synthesized from the substituted triamine. The key challenge in the synthesis of hpp\*H was the synthesis of tetrasubstituted triamine **18**. As described in this chapter, triamine **18** was obtained from the diaminonitrile **17**, which was obtained from the coupling of commercially available diamine **16** with mesylate **15**. Mesylate **15** was derived from the corresponding ester **13**, which in turn was obtained from the commercially available ethyl cyanoacetate **12**.

### Results and Discussion

As discussed in the retrosynthetic analysis of hpp\*H, the main target for synthesis of hpp\*H is the tetrasubstituted triamine **18**. There is only one report for the synthesis of triamine **18** by Cotton and co-workers, as given in Scheme 1. Our synthesis of the triamine **18** is given in Scheme 3 below and is different from that of Cotton and co-workers.

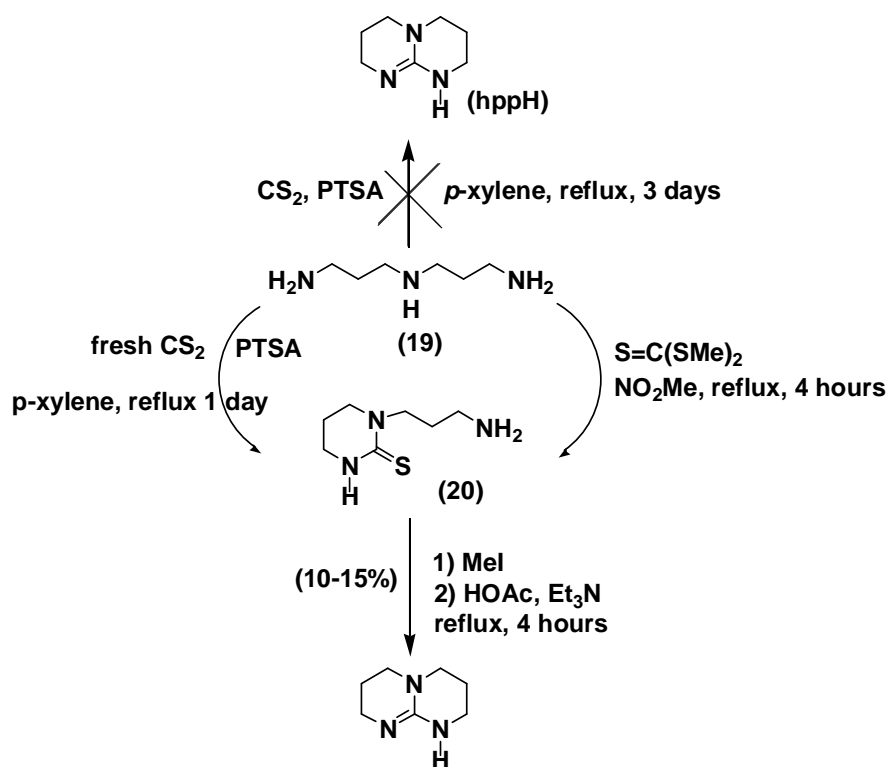


Scheme 3: Synthesis of tetramethyl triamine **18**.

The synthesis of triamine **18** starts with commercially available ethyl cyanoacetate **12**, which was dimethylated by treating with sodium hydride followed by iodomethane.<sup>60</sup>

The next step was to selectively<sup>61</sup> reduce the ester to the primary alcohol without reducing the nitrile group in **13**. This was successfully achieved by refluxing ester **13** with NaBH<sub>4</sub> to obtain pure alcohol **14** in appreciable yields. The alcohol was converted to the better leaving group methanesulfonate (or mesylate) **15**. An S<sub>N</sub>2 displacement reaction<sup>62</sup> on **15** with commercially available 2,2-dimethyl-propane-1,3-diamine **16** yielded diaminonitrile **17**. Compound **17** is a highly polar molecule and was difficult to purify on silica gel during column chromatography. Instead we used Florisil (magnesium silicate; 84% SiO<sub>2</sub>, 15.5% MgO and 0.5% Na<sub>2</sub>SO<sub>4</sub>) to effect separation. The separation is easier and quicker than with silica gel. The triamine **18** was finally obtained from **17** by reducing the nitrile group to a primary amine using LiAlH<sub>4</sub>.

The next step was to cyclize the triamine **18** to hpp\*H using a C1 reagent. The conditions for the final cyclization step were first optimized on non-methylated triamine [**19**, N-(3-aminopropyl)-1,3-propanediamine] which is available commercially. In the literature, the triamine can be cyclized to the corresponding bicyclic guanidine using a C1 reagent (a C1 reagent provides the carbon atom of the guanidine core). The various C1 reagents which have been used to cyclize the triamine to the bicyclic guanidine hppH are carbon disulfide,<sup>56</sup> thiophosgene,<sup>63</sup> dimethyltrithio carbonate<sup>64</sup> 1,1'(thiocarbonyl)dimidazole,<sup>65</sup> and tetramethyl orthocarbonate.<sup>65</sup> However, the most common C1 reagent used is carbon disulfide (CS<sub>2</sub>) because it is easy to handle and inexpensive as compared to the other reagents. Cotton and co-workers reported the synthesis of hpp\*H via cyclizing triamine **18** using CS<sub>2</sub>. Their CS<sub>2</sub> method was based on the A'Court patent reported in 1986.<sup>56</sup> When we tried the same reaction on the regular triamine **19**, the desired product hppH was not obtained. Instead we were able to isolate moncyclized thiourea **20** as the only product, as shown in Scheme 4 below, in appreciable yields.



Scheme 4: Cyclization of non-methylated triamine to hppH.

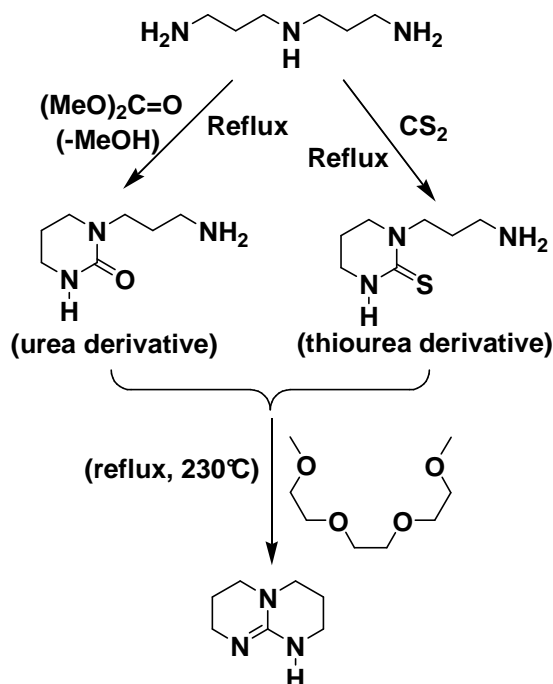
This cyclization reaction of the non-alkylated triamine was studied extensively in order to optimize the reaction conditions for this last step. Our results are summarized in Table 4.

Table 4: Cyclization of non-methylated triamine using CS<sub>2</sub> as the C1 reagent.

	Amount triamine <b>8</b> (mmoles)	Amount CS <sub>2</sub> (mmoles)	Triamine:CS <sub>2</sub>	Amount PTSA (g)	Amount Solvent, <i>p</i> -xylene (ml)	Reflux time (hours)	% Yield of <b>20</b>
1	7.15	8.58	0.84	0.05	40	24	34
2	7.15	8.58	0.84	0.05	40	72	23
3	7.15	6.43	1.1	0.05	40	24	47
4	7.15	6.43	1.1	0.05	20	24	13
5	7.15	6.43	1.1	0.05	80	24	77
6	7.15	6.43	1.1	0.05 (fresh)	40	24	60
7	7.15	6.43	1.1	0.05	160	24	82

From the studies summarized in Table 4, it can be inferred that the cyclization is sensitive to dilution (higher dilution leads to higher yields). We also noted that fresh PTSA was needed. The cyclization required only 24 hours, yielding the monocyclized thiourea derivative **20** in appreciable yields. However the double cyclized product, hppH was not obtained under any conditions.

Two very recent patents discuss an alternate cyclization route to hppH involving CS<sub>2</sub><sup>66</sup> and dimethyl carbonate<sup>67</sup> [O=C(OMe)<sub>2</sub>] as C1 reagents with non-methylated triamine **19**. These patents report that with CS<sub>2</sub> as the C1 reagent, the monocyclized thiourea is formed. When dimethyl carbonate is used, the corresponding monocyclic urea derivative is formed (Scheme 5). According to these patents, either of the monocyclized derivatives (thiourea or urea) can be cyclized by refluxing them in the high boiling point polar solvent triethyleneglycol dimethyl ether, yielding hppH in 50% yield. We found that the second cyclization of the monocyclized thiourea precursor to hppH was achieved by refluxing in this high boiling point polar solvent for 3 days. The isolation of hppH by this method was very simple: adding hexanes to the cooled reaction mixture and cooling to -10°C overnight gave crystalline hppH in ~50% yields.



Scheme 5: Synthesis of hppH using  $\text{CS}_2$  and dimethyl carbonate as C1 reagents.

Double cyclization to hppH was also achieved using the method of Davis and co-workers<sup>64</sup> by treating **20** with acetic acid and MeI (Scheme 4). However the yield for this double cyclization was very low (10-15%).

The same procedure was used for the synthesis of hpp\*H by cyclizing the tetramethyl triamine **18**. The bicyclic guanidine hpp\*H was also obtained in very low yield (10%). Sublimation of the crude solid yielded pure hpp\*H. hpp\*H was characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy and mass spectrometry. The  $^1\text{H}$  NMR spectrum (Figure 13) consisted of 3 peaks at  $\delta$  1.01 ( $\text{CH}_3$ , 12H), 2.81 ( $\text{CH}_2$ , 4H) and 2.91 ( $\text{CH}_2$ , 4H) consistent with the structure. We cannot definitively differentiate the  $\text{CH}_2$  resonances on C1 v/s C3 carbons on the ring.

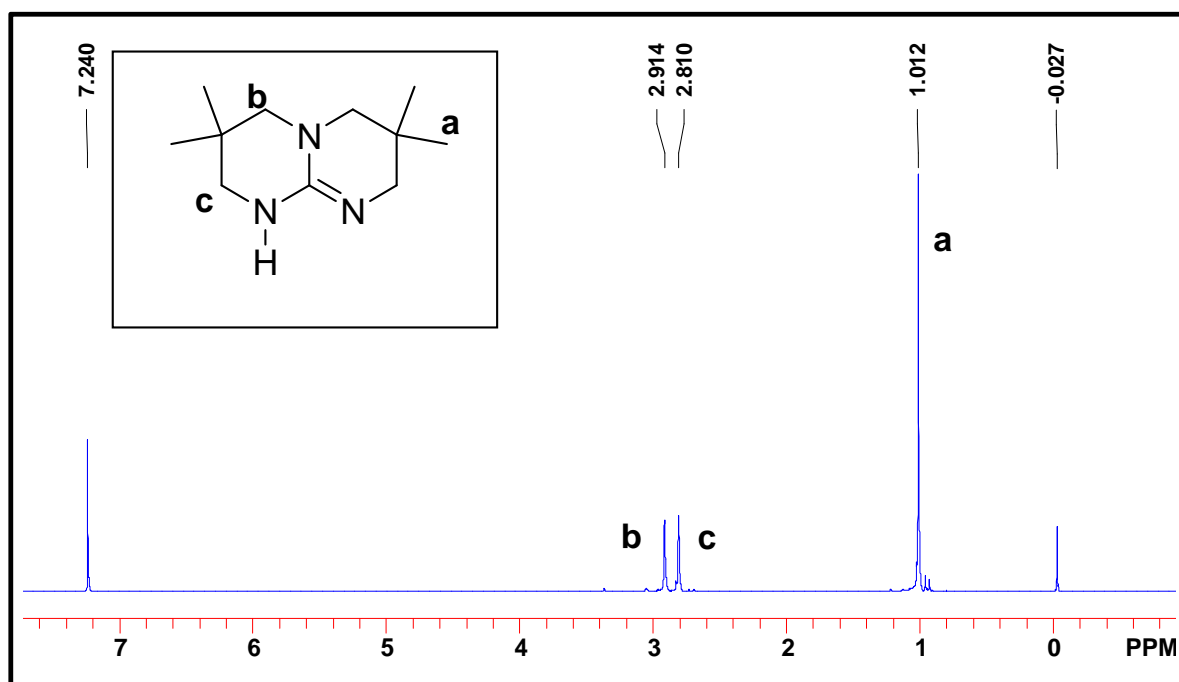


Figure 13: 300 MHz  $^1\text{H}$  NMR spectrum of sublimed hpp\*H in  $\text{CDCl}_3$ .

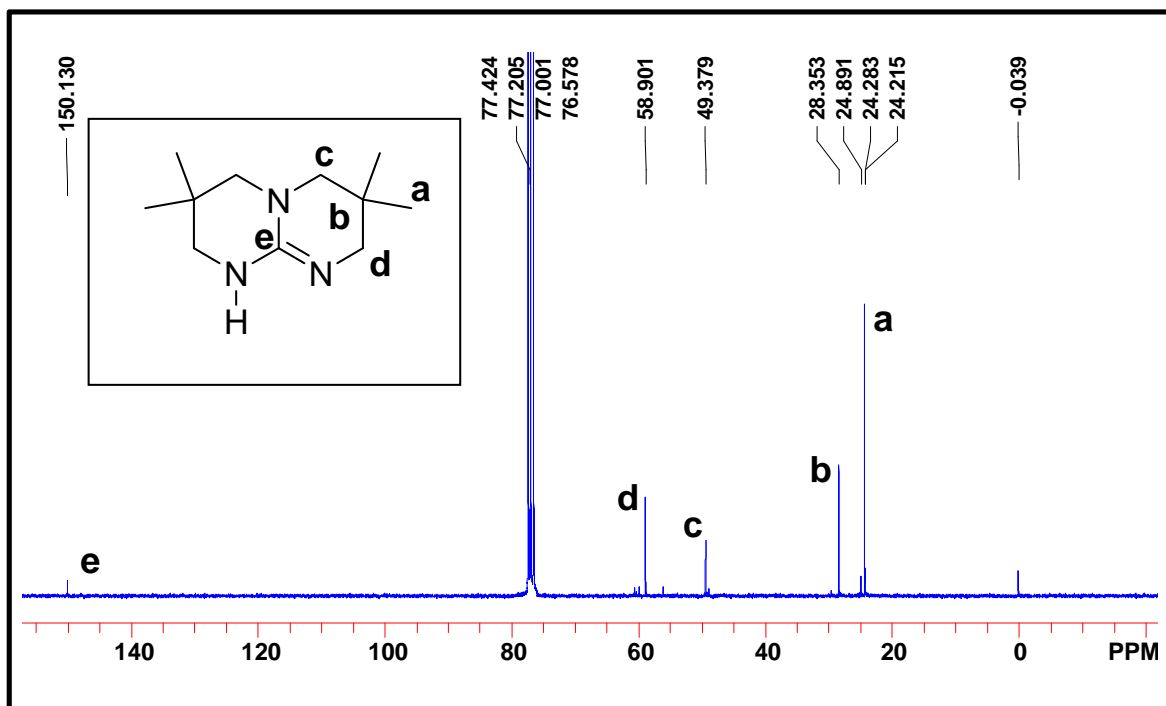


Figure 14: 75 MHz  $^{13}\text{C}$  NMR of sublimed hpp\*H in  $\text{CDCl}_3$ .

The  $^{13}\text{C}$  NMR spectrum of sublimed hpp\*H (Figure 14) was also consistent with the structure and consisted of 5 peaks. The HRMS (EI) also corresponds to the molecular mass within the error limit.

In summary we have developed an alternate route to hpp\*H that, while in low overall yield (3.9%, unoptimized, v/s 39% as reported by Cotton), and having more steps (6 v/s 3 reported by Cotton) is reproducible and does not require high pressure (1500 psi) methods. In privileged discussions with a co-worker of Cotton, their second step, the hydrogen reduction of the hydroxyimines, is not easily reproducible. Our method allows

for easier changes in the substituents on C2, since these groups are introduced in the first step using an alkyl iodide.

### Conclusions

Cyclization of non-methylated triamine  $\text{NH}((\text{CH}_2)_3\text{NH}_2)_2$  using  $\text{CS}_2$  as a C1 reagent yielded the monocyclized thiourea derivative **20** instead of the doubly cyclized product, hppH, unlike literature reports. Synthesis of hppH was carried out by heating the monocyclized thiourea **20** in a high boiling point solvent (triethylene glycol dimethyl ether) for three days. The isolation of hppH from triethylene glycol dimethyl ether was easier than from the *in situ* double cyclization route, starting with dimethyl trithiocarbonate as C1 reagent, in nitromethane.

The tetramethyl triamine  $\text{NH}(\text{CH}_2\text{C}(\text{CH}_3)_2\text{CH}_2\text{NH}_2)_2$  was derived in five steps starting from commercially available ethyl cyanoacetate in 26% overall yield. The key step was the  $\text{S}_{\text{N}}2$  reaction of commercially available 2,2-dimethyl-1,3-diaminopropane with the mesylate derivative of the synthesized alcohol ( $\text{HOCH}_2\text{C}(\text{CH}_3)_2\text{CN}$ ). Purification of the diaminonitrile product  $\text{NCC}(\text{CH}_3)_2\text{CH}_2\text{NHCH}_2\text{C}(\text{CH}_3)_2\text{CH}_2\text{NH}_2$  **17** was achieved, using Florisil instead of silica gel, via column chromatography. Hpp\*H was obtained by cyclizing the tetramethyl triamine  $\text{NH}(\text{CH}_2\text{C}(\text{CH}_3)_2\text{CH}_2\text{NH}_2)_2$  using dimethyl trithiocarbonate as a C1 reagent. The yield of this double cyclization (10-15%) needs further optimization.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy and high resolution mass spectral analyses of the doubly cyclized product was consistent with the structure of hpp\*H and matches literature data from the Cotton and co-workers group.<sup>59</sup>

Once the yield for the double cyclization step is optimized, the utility of this ligand for early transition metal chemistry, particularly Ta, will be studied in the future. A future study of the coordination complexes of  $\text{hpp}^*^-$  will ascertain whether the tetramethylation leads to more solubility for coordination complexes and also whether the  $\text{hpp}^*^-$  ligand is more basic than  $\text{hpp}^-$ .



## Experimental

**General Procedures.** All procedures were performed under an argon atmosphere unless noted and all glassware was oven dried before use. Ethyl cyanoacetate, sodium hydride, lithium aluminum hydride, p-toluenesulfonic acid monohydrate, iodomethane, sodium borohydride, dimethyl trithiocarbonate and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) were purchased from Aldrich and were used as received. Methanesulfonyl chloride and triethylene glycol dimethyl ether were purchased from Acros Chemicals. Lead(II) acetate strips and Florisil (100-200 mesh) were purchased from Fisher Scientific. The evolution of H<sub>2</sub>S gas in the synthesis of hppH and hpp\*H was monitored by exposing lead(II) acetate strips to gases evolved during the reaction. Flash column chromatography was performed employing 230-400 mesh silica gel. Thin-layer chromatography was performed using glass plates pre-coated to a depth of 0.25 mm with 230-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Proton and carbon-13 nuclear magnetic resonance (<sup>1</sup>H NMR and <sup>13</sup>C NMR) spectra were recorded on a Bruker Avance 300 instrument; chemical shifts are expressed in parts per million (δ scale) downfield from tetramethylsilane and are referenced to the residual proton resonance in the deuterated NMR solvent (CHCl<sub>3</sub>; δ 7.26 for <sup>1</sup>H NMR) or the solvent carbon-13 chemical shift (CDCl<sub>3</sub>; δ 77.16).

**Preparation of ethyl 2-cyano-2,2-dimethylpropionate (13).** Ethyl cyanoacetate **12** (15.01 g, 0.14 mol) was dissolved in 200 mL THF and stirred at 0°C in an ice-bath for 10 minutes. NaH (13.26 g, 0.35 mol) was added slowly in four portions, with stirring and cooling, at intervals of 30 minutes. Methyl iodide (18.16 g, 0.31 mol) was added in four equal portions, one after each portion of NaH added. The resulting mixture was stirred at 25°C overnight. The reaction was quenched by addition of ice. The aqueous layer was extracted with ethyl acetate (4 x 50 mL). The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and evaporated to obtain a crude oil. The crude oil was distilled *in vacuo* (50°C/150mTorr, lit.<sup>60</sup> bp 34°C/110 mTorr) to yield **13** (14.05 g, 75% based on **12**) as colorless oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.33 (t, 3H, 7.1 Hz, CH<sub>3</sub>CH<sub>2</sub>-), 1.60 (s, 6H, -(CH<sub>3</sub>)<sub>2</sub>), 4.27 (q, 2H, 7.1 Hz, CH<sub>3</sub>CH<sub>2</sub>-).

**Preparation of 3-hydroxy-2,2-dimethylpropanenitrile (14).** A solution of **13** (10.01 g, 0.07 mol) in 10 mL absolute ethanol was added dropwise to an ice-cooled suspension of sodium borohydride (4.23 g, 0.105 mol) in 250 mL of absolute ethanol. After complete addition, the mixture was refluxed for 24 hours. The reaction mixture was cooled in an ice-bath and the solution neutralized with 2N HCl until the pH was 7. The white granulated precipitate was removed by filtration and the filtrate concentrated using a rotary evaporator to give **14** (4.9 g, 70% based on **13**) as a colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  1.26 (s, 6H,  $-(\text{CH}_3)_2$ ), 3.47 (s, 2H,  $-\text{CH}_2$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  22.6 ( $-(\text{CH}_3)_2$ ), 34.94 ( $-\text{C}(\text{CH}_3)_2$ ), 68.35 ( $-\text{CH}_2\text{OH}$ ), 124.25 ( $\text{CN}$ ).

**Preparation of 2-cyano-2-methylpropyl methanesulfonate (15).** To a stirred solution of **14** (16.01 g, 0.16 mol) and trimethylamine (45.5 mL, 0.32 mol) in  $\text{CH}_2\text{Cl}_2$  (200 mL) cooled to  $0^\circ\text{C}$  was added, dropwise, a solution of methanesulfonyl chloride (17.5 mL, 0.22 mol) in 20 mL  $\text{CH}_2\text{Cl}_2$ . The mixture was stirred overnight. The solution was washed with saturated aqueous  $\text{NaHCO}_3$  solution. The organic layer was dried over  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum to give crude as a yellow-orange oil. The oil was purified using column chromatography (hexane/ethyl acetate 1:1,  $R_f = 0.6$ ) to afford **15** (27.50 g, 96% based on **14**) as a yellow-orange oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  1.44 (s, 6H,  $-(\text{CH}_3)_2$ ), 3.31 (s, 3H,  $-\text{OSO}_2\text{CH}_3$ ), 4.12 (s, 2H,  $\text{CH}_2\text{OH}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  20.81 ( $-(\text{CH}_3)_2$ ), 32.92 ( $-\text{OSO}_2\text{CH}_3$ ), 37.53 ( $-\text{C}(\text{CH}_3)_2$ ), 72.80 ( $-\text{CH}_2\text{O}-$ ), 121.92 ( $\text{CN}$ ).

**Preparation of 3-(3-amino-2,2-dimethylpropylamino)-2,2-dimethylpropanenitrile (17).** 2,2-Dimethyl-1,3-diaminopropane **16** (13.6 mL, 0.13 mol) was added to a stirred solution of **15** (10.03 g, 56.60 mmol) in 100 mL *n*-butanol and 20 drops of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU). The mixture was refluxed for 48 h. After cooling, the mixture was concentrated under vacuum to give a viscous oil. The oil was then subjected to flash chromatography using Florisil as a solid support ( $\text{CH}_2\text{Cl}_2$ : MeOH:  $\text{NH}_4\text{OH}$  8:2:0.01,  $R_f = 0.38$ ) to obtain **17** (7.47 g, 72% based on **15**) as a colorless viscous oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  0.89 (s, 6H,  $-(\text{CH}_3)_2$ ), 1.34 (s, 6H,  $-(\text{CH}_3)_2$ ), 1.95 (broad s, 3H,  $-\text{NH}_2 + -\text{NH}$ ), 2.52 (s, 2H,  $-\text{CH}_2$ ), 2.56 (s, 2H,  $-\text{CH}_2$ ), 2.66 (s, 2H,  $-\text{CH}_2$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  23.36 ( $-(\text{CH}_3)_2$ ), 24.25 ( $-(\text{CH}_3)_2$ ), 33.84 ( $-\text{C}(\text{CH}_3)_2$ ), 35.34 ( $-\text{C}(\text{CH}_3)_2$ ), 50.72 ( $-\text{CH}_2\text{-NH}_2$ ), 58.94 ( $-\text{CH}_2\text{-N-}$ ), 59.00 ( $-\text{CH}_2\text{-N-}$ ), 124.65 ( $\text{CN}$ ).

**Preparation of bis-(3-amino-2,2-dimethylpropyl) amine (18).** The solution of **17** (15.01 g, 81.96 mmol) in 200 mL THF under argon was cooled in an ice-bath for 10 minutes. LiAlH<sub>4</sub> (8.18 g, 204.9 mmol) was added slowly, with constant stirring. After complete addition, the mixture was refluxed for 2 days. The reaction mixture was cooled in an ice-bath and the pH neutralized to 8 by slow addition of 20% aqueous NaOH. The white precipitate was filtered and the filtrate dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated using rotary evaporator to yield **18** as a colorless, viscous oil. The oil was distilled *in vacuo* (60-65°C at 150 mTorr) to yield **18** (9.98 g, 65% based on **17**) as a pure colorless oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 0.79 (s, 12H, -(CH<sub>3</sub>)<sub>2</sub>), 1.16 (broad s, 4.78, -NH<sub>2</sub> + -NH), 2.33 (s, 4H, -CH<sub>2</sub>), 2.46 (s, 4H, -CH<sub>2</sub>). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 23.76 (-(CH<sub>3</sub>)<sub>2</sub>), 35.58 (-C(CH<sub>3</sub>)<sub>2</sub>), 51.54 (-CH<sub>2</sub>-NH<sub>2</sub>), 59.87 (-CH<sub>2</sub>-N-).

**Preparation of 1-(3-aminopropyl)tetrahydropyrimidine-2-thione (20).** Distilled **19** (1.01 mL, 7.15 mmol) was dissolved in 80 mL dry *p*-xylene under argon and *p*-toluenesulfonic acid (0.05 g) was added. Carbon disulfide (0.39 mL, 6.43 mmol) was slowly added, resulting in the formation of a white precipitate. The mixture was refluxed for 24 hours. *p*-Xylene was decanted to leave behind any oily residue in the flask, and cooled to room temperature under argon. A white precipitate was filtered and dried under vacuum to yield pure **20** (0.87 g, 70% based on **19**) <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.34 (broad s, 2H, NH<sub>2</sub>), 1.77 (quintet, 2H, 7.05 Hz, -CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-), 1.99 (quintet, 2H, 5.85 Hz, -CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-), 2.73 (t, 2H, 6.6 Hz, -N-CH<sub>2</sub>-), 3.26-3.38 (m, 4H, -CH<sub>2</sub>-NH<sub>2</sub> + -CH<sub>2</sub>-N-), 3.94 (t, 2H, 7.05 Hz, -N-CH<sub>2</sub>-), 6.37 (broad s, 1H, -NH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 20.8 (-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-), 30.67 (-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-), 38.74 (-CH<sub>2</sub>-NH<sub>2</sub>), 40.62 (-CH<sub>2</sub>-NH), 45.81 (-CH<sub>2</sub>-N), 51.13 (-CH<sub>2</sub>-N), 178.12 (-C=S).

**Preparation of 1,5,7-triazabicyclo[4.4.0]dec-5-ene (hppH).** A solution of **20** (2.11 g, 12.18 mmol) in 10 mL triethylene glycol dimethyl ether was refluxed at 230°C for 3 days. Evolution of H<sub>2</sub>S was detected using lead acetate strips. The contents of the flask were cooled to room temperature and hexane (20 mL) was slowly added dropwise. The mixture was then kept in a freezer (5°C) overnight to yield white crystals of hppH (0.79 g, 47% based on **20**). The <sup>1</sup>H NMR matches that of a commercially available sample from Aldrich. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.86 (quintet, 2H, 5.91 Hz, -CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>), 3.10 (t, 2H, 6 Hz, -N-CH<sub>2</sub>-), 3.18 (t, 2H, 6 Hz -N-CH<sub>2</sub>-).

**Preparation of 3,3,9,9-tetramethyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (hpp\*H).**

Tetramethyl triamine **18** (1.01 g, 5.30 mmol) was dissolved in dry, degassed NO<sub>2</sub>Me and stirred under argon. Dimethyl trithiocarbonate (0.71 mL, 6.4 mmol) was added and the mixture refluxed for 3 hours, then allowed to cool. Acetic acid (1.25 mL, 21.3 mmol) and methyl iodide (0.68 mL, 10.6 mmol) was added. The mixture was refluxed for 3 hours and stirred at room temperature overnight. The mixture was evaporated under reduced pressure and the residue partitioned between CH<sub>2</sub>Cl<sub>2</sub> and water. The products were extracted into CH<sub>2</sub>Cl<sub>2</sub> (3 x 15 mL). Excess C1 reagent was removed by passing a CH<sub>2</sub>Cl<sub>2</sub> solution of the product through a plug of silica gel and further eluting with CH<sub>2</sub>Cl<sub>2</sub> (50 mL) and then MeOH-CH<sub>2</sub>Cl<sub>2</sub> (1:4). The combined MeOH- CH<sub>2</sub>Cl<sub>2</sub> washes were treated with charcoal, dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a brown-red solid. The solid was dissolved in 20 mL water and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 30 mL). The combined CH<sub>2</sub>Cl<sub>2</sub> extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated under reduced pressure to yield a yellow-brown solid. The crude solid was purified by sublimation to obtain hpp\*H (0.12 g, 11% based on **18**) as a white solid. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 0.96 (s, 12H, (-CH<sub>3</sub>)<sub>4</sub>), 2.78 (s, 4H, -N-CH<sub>2</sub>-), 2.91 (s, 4H, -N-CH<sub>2</sub>-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 24.29 (-CH<sub>3</sub>), 28.36 (-C(CH<sub>3</sub>)<sub>2</sub>), 49.39 (-N-CH<sub>2</sub>-), 58.91 (-N-CH<sub>2</sub>-), 150.14 (-C=N-). HRMS (EI): calculated for C<sub>11</sub>H<sub>21</sub>N<sub>3</sub> (M<sup>+</sup>): 195.1735, found 195.1739.

## CHAPTER 4

SYNTHESIS OF 2-(S),10-(S)-2,10-DIMETHYL-1,5,7-  
TRIAZABICYCLO[4.4.0]DEC-5-ENE

Compounds containing the bicyclic guanidine unit are of substantial biological interest because of hydrogen-bonded mediated interactions **21** (Figure 15) with a variety of oxoanions such as carboxylates<sup>68</sup> and phosphates.<sup>69</sup> The bicyclic guanidinium scaffold **22** (Figure 15) has been widely adopted for the construction of molecular hosts for anions.<sup>70</sup> The parent bicyclic guanidine hppH (Figure 15) is available commercially and has been used as a versatile and robust ligand to stabilize low oxidation state transition metals with metal-metal multiple bonds, as discussed in Chapter 1. Chiral  $C_2$ -symmetric derivatives of hppH at carbon C3 **23** (Figure 15, R = CH<sub>2</sub>OH<sup>71</sup>, CH<sub>2</sub>NH<sub>2</sub><sup>72</sup> and CHPh<sub>2</sub><sup>64</sup>)

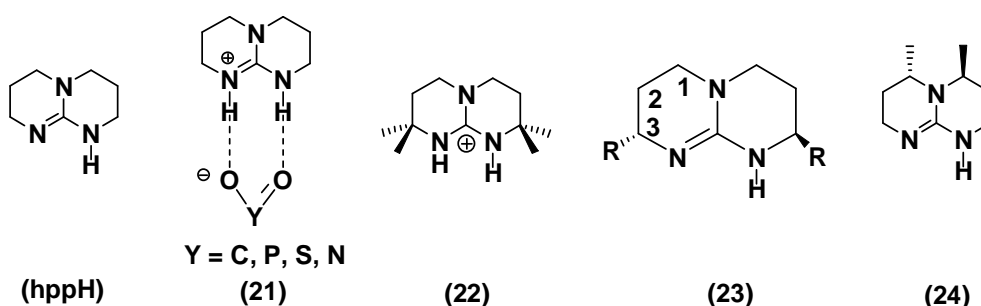


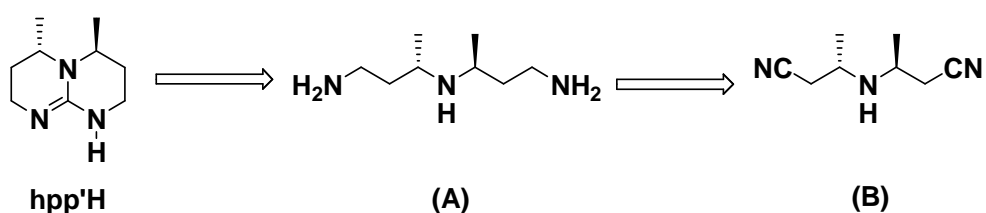
Figure 15: Bicyclic guanidinium hppH as molecular host **21** and **22**; chiral bicyclic guanidine derivatives of hppH **23** and **24**.

have been synthesized and used as chiral molecular hosts. Unfortunately, these known derivatives may not be suitable for dinuclear chemistry because they could be susceptible to ortho-metallation during the synthesis of paddlewheel complexes. Chiral  $C_2$ -symmetric derivatives at C1 and C2 (**23** Figure 15) are unknown. From MO calculations discussed in Chapter 2, a Me substituent at C1 makes the bicyclic guanidine more basic than hppH. Thus, on the basis of these calculations, we decided to synthesize a chiral  $C_2$ -symmetric

derivative of hppH (**24**, R = CH<sub>3</sub>, Figure 15). This derivative 2-(S),10-(S)-2,10-dimethyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (**24** Figure 15) will be designated as hpp'H.

### Retrosynthetic Analysis

As discussed in Chapter 2, bicyclic guanidines can be synthesized from the



Scheme 6: Retrosynthetic analysis of chiral C<sub>2</sub>-symmetric hpp'H.

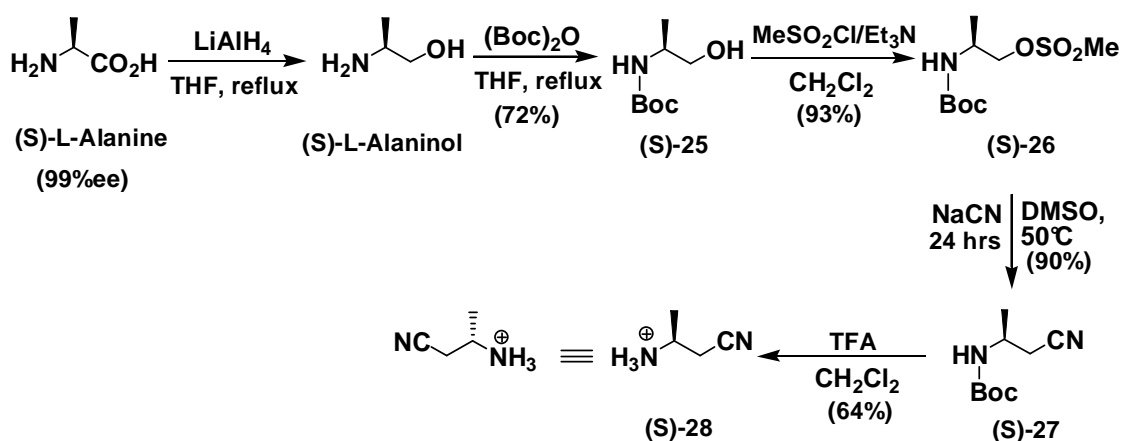
corresponding triaminonanes. Thus chiral C<sub>2</sub>-symmetric hpp'H could be synthesized from chiral C<sub>2</sub>-symmetric triamine **A** (Scheme 6). This chiral triamine **A** can be synthesized from the chiral dinitrile **B** using a reducing agent.

### Results and Discussion

#### Attempts to synthesize chiral dinitrile **B**

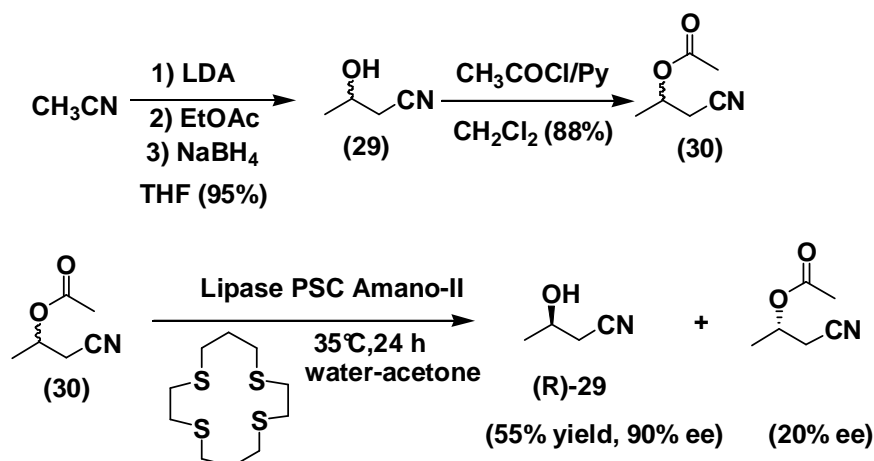
Several routes were employed in attempts to synthesize chiral dinitrile **B**. The chiral hpp'H (or the chiral dinitrile **B**) has a methyl group next to the tertiary nitrogen. The synthesis of chiral C<sub>2</sub>-symmetric hpp'H was designed to use a chiral source that was inexpensive and easily available. L-Alanine is the simplest chiral amino acid which has one methyl group adjacent to the amino group. L-Alanine therefore was chosen as a chiral source for the synthesis of chiral hpp'H.

The first step in synthesis of chiral dinitrile **B** was the derivatization of L-alanine (99% ee) as shown in Scheme 7 below. Reduction of L-alanine to L-alaninol via  $\text{LiAlH}_4$  was followed by protection of the amino group with  $(\text{Boc})_2\text{O}$  in a single pot to yield (S)-**25**.<sup>73</sup> The next step was to convert the alcohol to a nitrile. This was achieved by converting the OH group to a better leaving group, mesylate, by treating with



Scheme 7: Derivatization of L-alanine to synthesize chiral aminonitrile.

methanesulfonyl chloride/ $\text{Et}_3\text{N}$ <sup>62</sup> to form (S)-**26**.  $\text{S}_{\text{N}}2$  displacement of the mesylate group by NaCN lead to the N-Boc-protected chiral nitrile (S)-**27**. The last step was the removal of the Boc group with trifluoroacetic acid in  $\text{CH}_2\text{Cl}_2$  as solvent.<sup>74</sup> (S)-**28** was characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopies and mass spectrometry and is consistent with the ammonium salt.

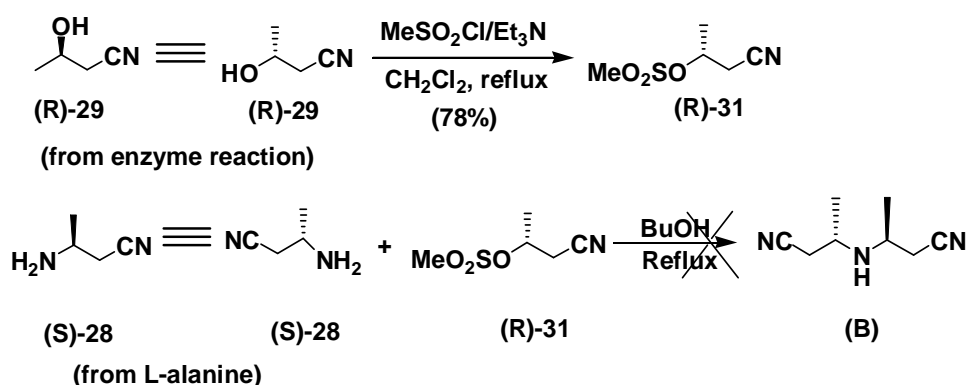


Scheme 8: Synthesis of chiral  $\beta$ -hydroxynitrile.

The next target in the synthesis of chiral dinitrile **B** was the chiral  $\beta$ -hydroxynitrile (**R**)-**29**, as shown in Scheme 8 above. The synthesis of chiral  $\beta$ -hydroxynitrile (**R**)-**29** began with the synthesis of racemic  $\beta$ -hydroxynitrile **29**. Deprotonation of acetonitrile using the base lithium diisopropylamide, LDA, followed by treatment with EtOAc and *in situ* reduction of the resulting ketone gave racemic  $\beta$ -hydroxynitrile **29**<sup>75</sup> in almost quantitative yields. The alcohol was then converted to ester acetate **30** by treatment with acetyl chloride/pyridine. The resulting racemic ester **30** was hydrolyzed<sup>76</sup> by Lipase PSC Amano-II enzyme in the presence of a 1,4,8,11-tetrathiacyclotetradecane (thiacrown ether) to yield the chiral  $\beta$ -hydroxynitrile (**R**)-**29** in appreciable yield and very high optical purity (90% ee by chiral GC).

The next step was to couple (Scheme 9) chiral amino nitrile (**S**)-**28** with the mesylate ((**R**)-**31**) of the chiral  $\beta$ -hydroxynitrile (**R**)-**29** by refluxing in butanol in the presence of DBU (1,8-diazabicyclo[5.4.0]undec-7-ene) as base.

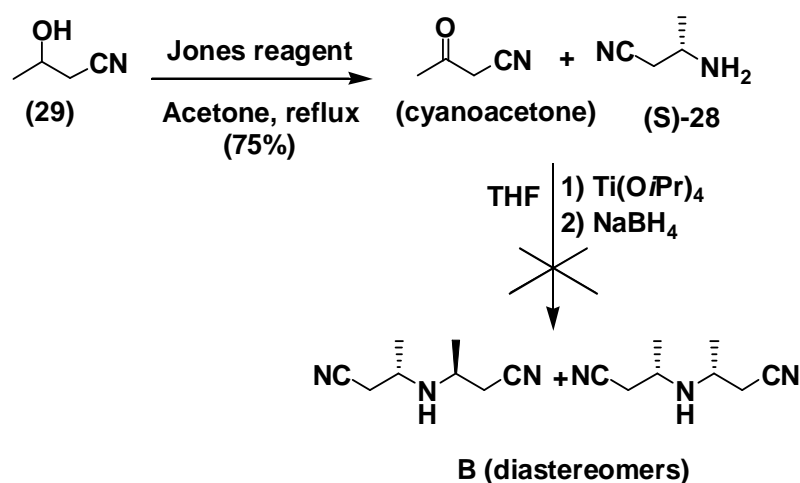




Scheme 9: Attempt to synthesize dinitrile **B**.

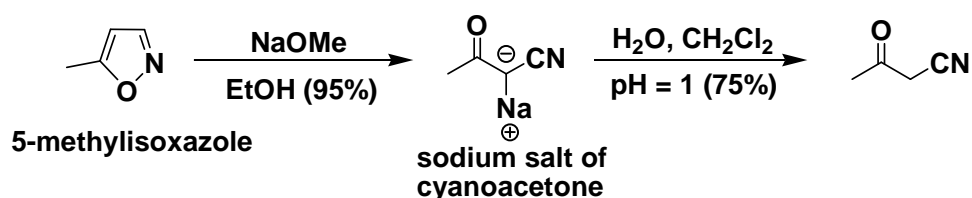
Unfortunately, the desired chiral dinitrile **B** was not obtained. The  $^1\text{H}$  NMR of the obtained product was too complicated to interpret. The reason for this could be due to steric hindrance.

After this unsuccessful result, a reductive amination approach<sup>77</sup> was used as an alternate route to chiral dinitrile **B** as shown in Scheme 10 below. The key step is the coupling of chiral amino nitrile (**S**)-**28** (obtained from derivatization of L-alanine) and cyanoacetone (obtained from oxidation of **29** using Jones reagent) in the presence of  $\text{Ti}(\text{O}i\text{Pr})_4$  (as acid) and sodium borohydride (as reducing agent). In this case, the desired diastereomers were not obtained. Instead, the reduced secondary alcohol of cyanoacetone and pure chiral amine (**S**)-**28** was obtained.



Scheme 10: Reductive amination approach to chiral dinitrile **B**.

We assumed that the unidentified impurities in the cyanoacetone was the reason for the lack of success, so we sought to synthesize cyanoacetone by another synthetic route. Cyanoacetone was then prepared from the sodium salt of cyanoacetone (Scheme 11 below).

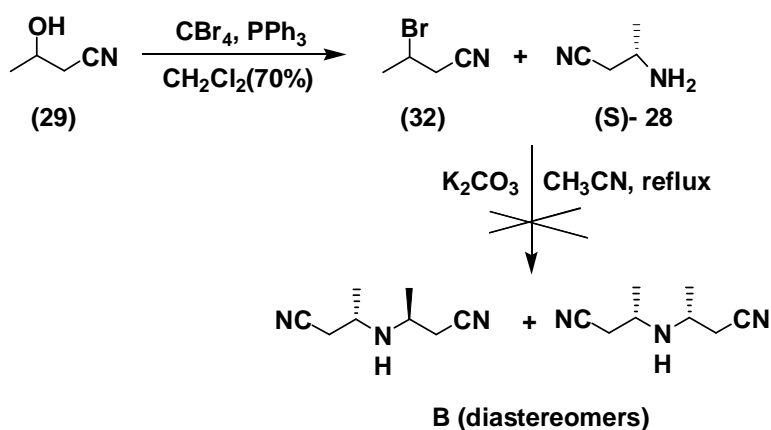


Scheme 11: Alternate synthesis of cyanoacetone.

Cyanoacetone was synthesized by treating 5-methylisoxazole<sup>78</sup> with sodium methoxide (Scheme 11) to yield the sodium salt of cyanoacetone. The salt is a stable white solid and can be stored at room temperature. Treatment of the sodium salt of cyanoacetone<sup>79</sup> with HCl until the pH decreased to 1, and extracting into organic phase yielded cyanoacetone in appreciable yields. However, the coupling of cyanoacetone with chiral amine (**S**)-**28**

(as in Scheme 10) did not form the desired diastereomers of **B**. Instead, the chiral amine and reduced secondary alcohol of cyanoacetone were obtained as major products.

Another approach to form the diastereomers of **B** was to convert the  $\beta$ -hydroxynitrile (**29**) to the corresponding bromide by treatment with  $\text{CBr}_4/\text{PPh}_3$ , followed by reacting the bromide with chiral amine **S-28** as shown in Scheme 12 below.



Scheme 12: Approach to diastereomers of dinitrile **B**.

Again, the desired diastereomers of **B** were not obtained. The reason could be that **32** is a low boiling liquid and could have evaporated during the reflux reaction conditions (temperature around  $80^\circ\text{C}$ ).

We postulated several reasons for this lack of success, possibly involving the chiral amine (**S**)-**28** and also cyanoacetone. The chiral amine (**S**)-**28** is unreported in the literature. We synthesized this chiral (**S**)-**28** amine as the trifluoroacetate ammonium salt. However, when we tried neutralization we were unable to isolate the neutral chiral amine, as shown by  $^1\text{H}$  NMR spectroscopy. Also, cyanoacetone is an unstable compound, forming an orange polymer (insoluble in any organic solvent) on standing for 10 minutes in air. It is also reported in the literature that is an unstable compound.<sup>79</sup> We surmised that if we could add bulky substituents on these two compounds, we might be able to stabilize and isolate these precursors and then continue the synthesis. To our surprise, we found

that the corresponding compounds with a phenyl group are stable and reported in the literature. The corresponding chiral amino nitrile<sup>80</sup> (**S-36**, Figure 16) can be isolated as a neutral amine as a colorless viscous oil, and the corresponding ketone was a yellow-orange stable solid<sup>81,82</sup> (**37**, Figure 16).

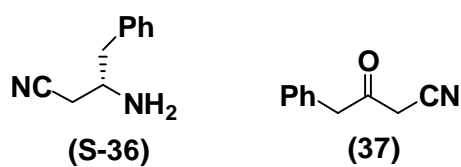
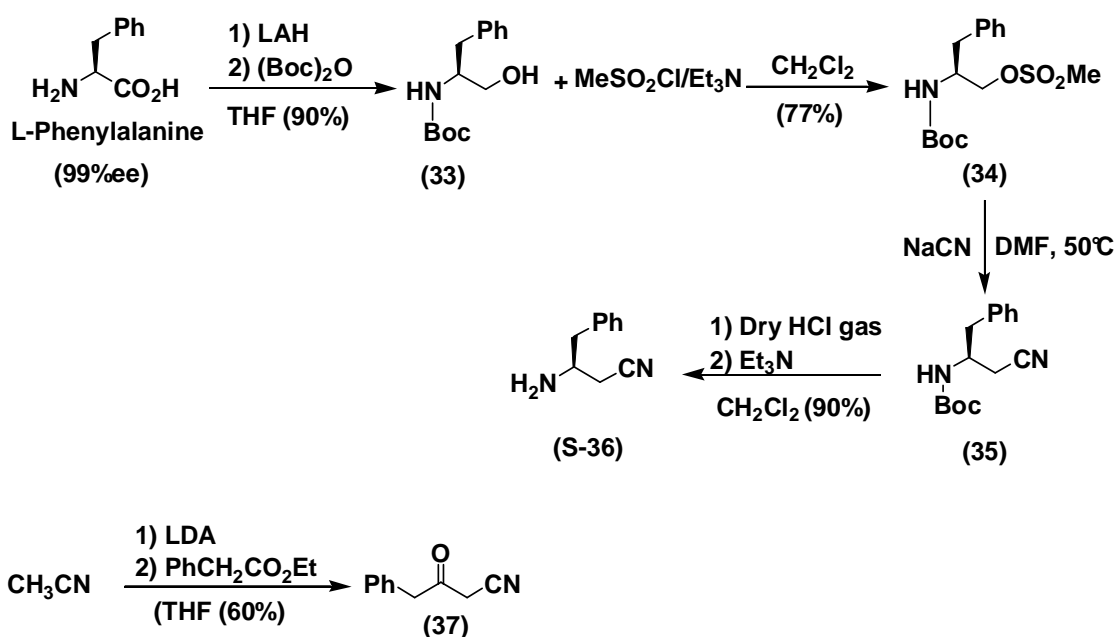


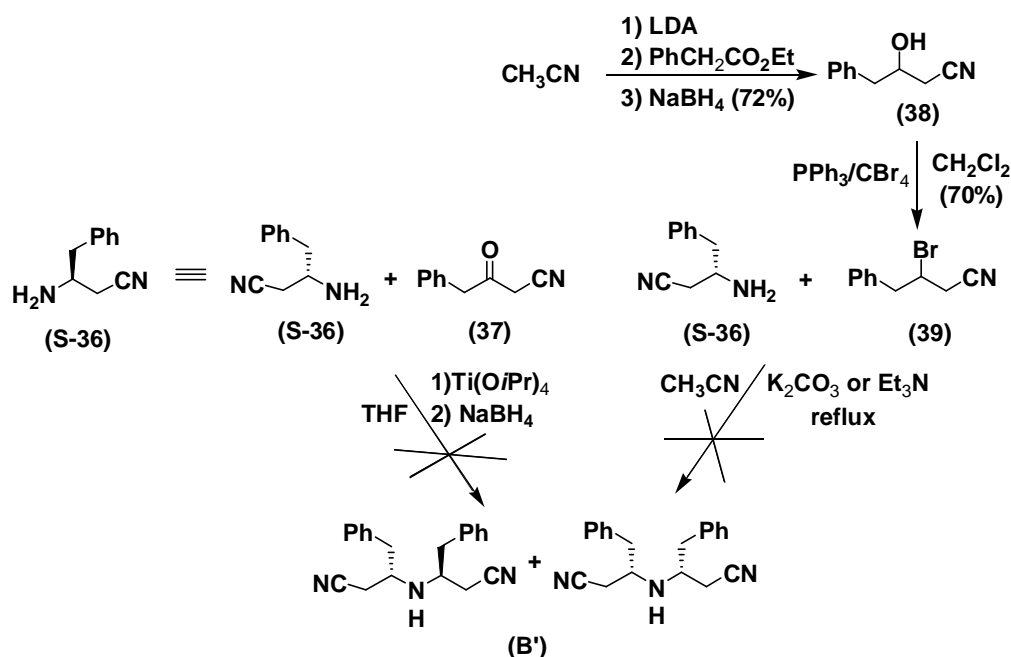
Figure 16: Chiral aminonitrile and β-ketonitrile with phenyl group as substituent.

The synthesis of (**S**)-**36** and **37** is shown in Scheme 13 below.



Scheme 13: Synthesis of (**S**)-**36** and **37**.

The synthesis of chiral (**S**)-**36** is similar to chiral (**S**)-**28**, but starts with L-phenylalanine as the chiral source. The only difference was that the neutral chiral amine (**S**)-**36** was isolated and was a stable, colorless, viscous oil unlike the chiral amine (**S**)-**28** derived from L-alanine. The  $\beta$ -ketonitrile **37** was synthesized from acetonitrile, using LDA as base and then treating with the corresponding ester, ethyl phenylacetate, to yield **37** in appreciable yields.

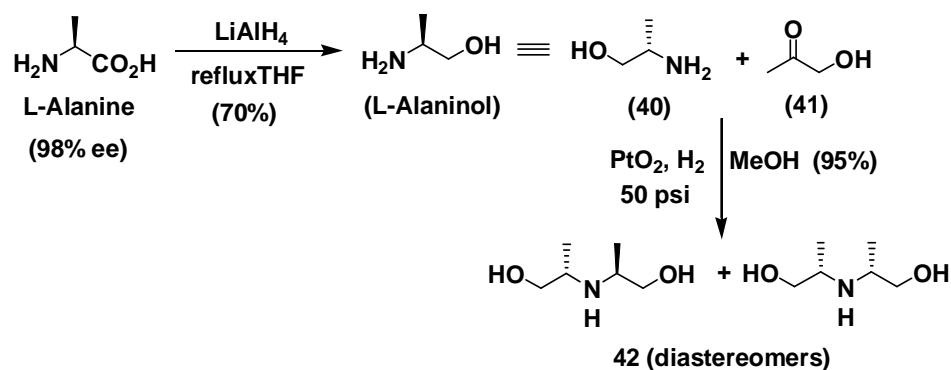


Scheme 14: Attempts to synthesize chiral dibenzylnitrite.

The next step was to couple the chiral amino nitrile (**S**)-**36** with ketone **37** via reductive amination using  $\text{Ti(OiPr)}_4$  and  $\text{NaBH}_4$ , as shown in Scheme 14. The desired diastereomers **B'** were not obtained; instead, the chiral amine (**S**)-**36** and the corresponding reduced secondary alcohol (of the ketone) was obtained. This was confirmed by gas-chromatography-mass spectrometry (GC-MS) on the two products isolated. In another attempt, the secondary alcohol **38** was synthesized from the corresponding ketone using  $\text{NaBH}_4$  as reducing agent. The secondary alcohol **38** was converted to the corresponding bromide **39** using  $\text{CB}_4/\text{PPh}_3$ . The next step was an  $\text{S}_{\text{N}}2$  reaction with chiral amine (**S**)-**36** to yield the desired diastereomers **B'**. However, the desired diastereomers **B'** were not obtained. In this case, the bromide **39** did not react, as its spot on TLC was visible even after 24 hour reflux, and no new spot on TLC was observed.

After these unsuccessful attempts, we altered our strategy. If we could couple the chiral amino alcohol with the prochiral ketoalcohol to get the chiral aminoalcohol, then the CN group can be introduced using  $\text{NaCN}$ . This first step of coupling the chiral L-alaninol with the corresponding prochiral ketoalcohol (hydroxyacetone, available

commercially) is also reported in the literature.<sup>83</sup> When we tried the same reaction, we were able to synthesize the chiral aminoalcohol **42** as a mixture of diastereomers (meso compound and the chiral  $C_2$ -symmetric diastereomer) in almost quantitative yields as shown in Scheme 15 below.



Scheme 15: Synthesis of diastereomers **42** starting from chiral L-alanine.

The next step was to protect the secondary amino group in **42**. The reaction with  $(\text{Boc})_2\text{O}$  was unsuccessful. Refluxing **42** with benzyl bromide in the presence of  $\text{NaHCO}_3$  yielded the benzyl-protected chiral aminoalcohol **43** as shown in Scheme 16 below. The next step was to convert the hydroxyl groups in **43** to better leaving groups so that an  $\text{S}_{\text{N}}2$  reaction by  $\text{NaCN}$  would yield the N-benzyl-protected chiral dinitrile **45**. The first attempts involved conversion of the hydroxyl groups to bromides. Only  $\text{PBr}_3$  worked of the several reagents ( $\text{PPh}_3/\text{CBr}_4$ ,  $\text{HBr}$ ,  $\text{PPh}_3/\text{Br}_2$ , Vilsmeier's reagent) tried. Refluxing **43** in benzene in presence of  $\text{PBr}_3$  yielded **44** in appreciable yields as a mixture of diastereomers. The  $\text{S}_{\text{N}}2$  displacement reaction of bromide in **44** with sodium cyanide ( $\text{NaCN}$ ) yielded the N-benzyl-protected chiral dinitrile **45** (as a mixture of diastereomers) in very good yields as determined by  $^1\text{H}$  NMR.







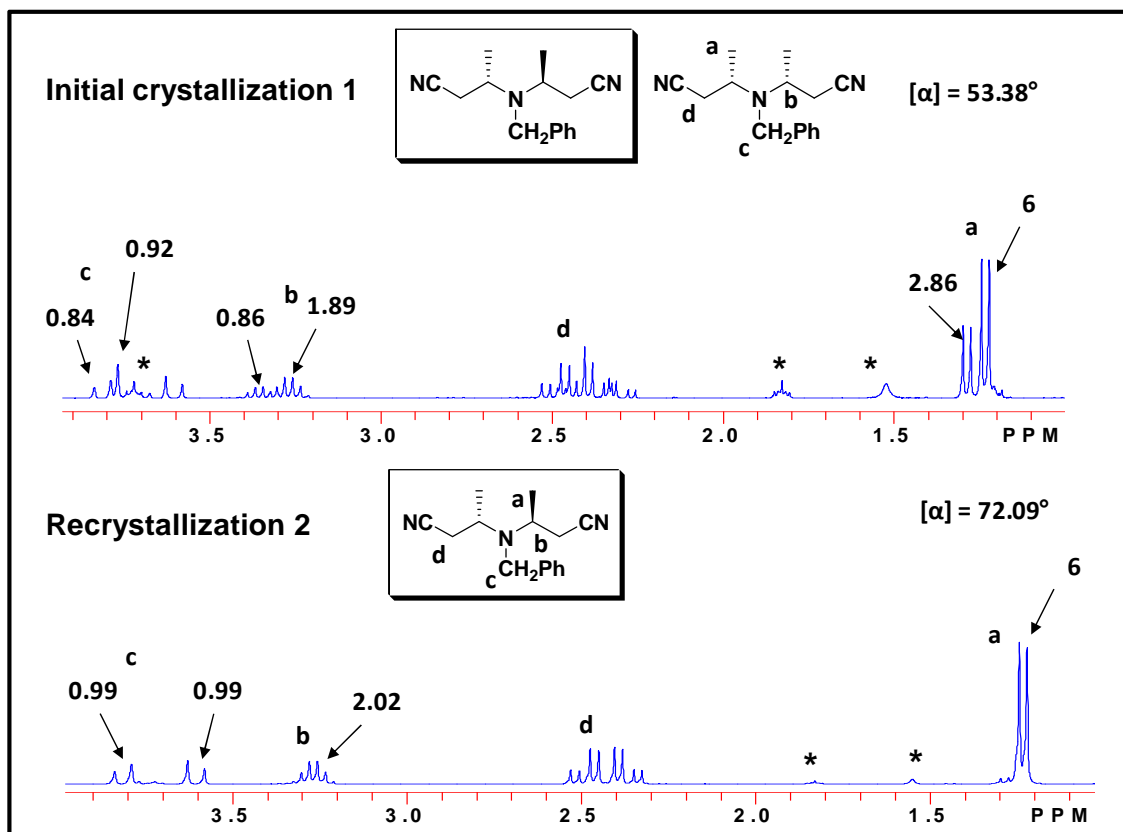
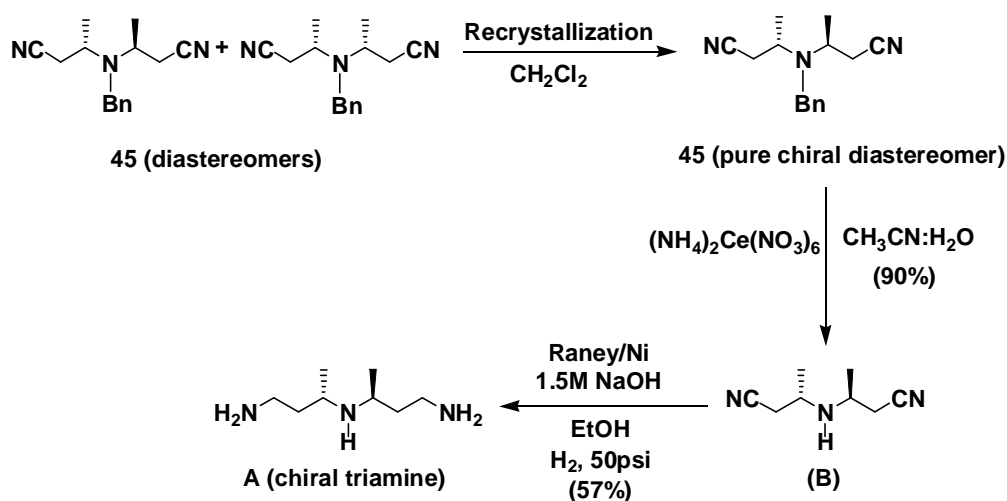


Figure 17: 300 MHz  $^1\text{H}$  NMR in  $\text{CDCl}_3$  depicting the separation of diastereomers **30**.

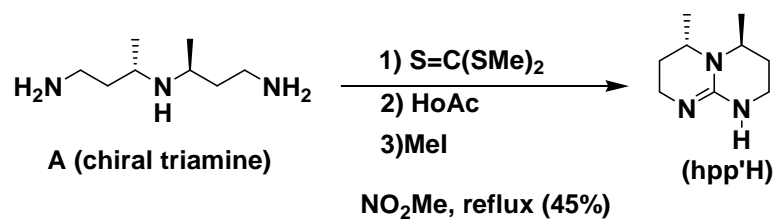
The upper spectrum displays the spectrum of the first crop of mixed diastereomers after recrystallization 1 and the bottom spectrum displays the spectrum of recrystallized product with only a single diastereomer (recrystallization 2). Numerical values above spectral features represent integration of resonances compared to the resonance at  $\delta$  1.22 set to a value of 6 protons,  $[\alpha]$  values are the specific rotation values of crystallized and recrystallized products, \* denotes resonances of impurities.



Scheme 18: Synthesis of chiral  $C_2$  symmetric triamine.

The next step was to remove the N-benzyl group from pure **45**. This was achieved using ceric ammonium nitrate<sup>84</sup> as the debenzylating reagent in acetonitrile:H<sub>2</sub>O as the solvent system. The desired chiral diastereomer **B** was obtained in 90% yields (Scheme 18). The last step in the synthesis of the desired chiral triamine **A**, reduction of the nitrile groups, was achieved by hydrogenation under dihydrogen pressure (50 psi) using Raney-Nickel<sup>85</sup> as catalyst. The chiral triamine **A** was obtained in appreciable yields after purification of crude product by vacuum distillation. A specific rotation of +27.67° indicated no racemization, and <sup>13</sup>C NMR spectra also show no peaks of diastereomers (in case racemization had occurred).

The last step in the synthesis of hpp'H was cyclization using a C1 reagent. The cyclization was achieved using the method of Davis and co-workers<sup>64</sup> with dimethyl trithiocarbonate, S=C(SMe)<sub>2</sub>, as a C1 reagent, giving chiral,  $C_2$ -symmetric hpp'H (Scheme 19). The specific rotation of the cyclized product was determined to be +8.73°, which indicated that there was no racemization during the cyclization.



Scheme 19: Cyclization of chiral triamine **A** to synthesize hpp'H.

### Spectroscopic Characterization of hpp'H

The cyclized product, either the guanidine hpp'H or the guanidinium hpp'H<sub>2</sub><sup>+</sup>I<sup>-</sup>, was characterized by <sup>1</sup>H (600 MHz) and <sup>13</sup>C (75 MHz) NMR spectroscopies. The <sup>1</sup>H (600 MHz) spectrum consists of a doublet at δ 1.235 with coupling constant of 6 Hz and corresponds to the methyl group. There are multiplets at δ 1.85 and 1.94 corresponding to 2 protons (by integration) each in the <sup>1</sup>H NMR spectrum. Another multiplet exists at δ 3.35 corresponds to 4 protons (by integration). A multiplet at δ 3.58 corresponds to the CH attached to the methyl group. High field 2D NMR experiments (<sup>1</sup>H COSY) will be needed in future studies in order to interpret the <sup>1</sup>H NMR spectrum more conclusively. The <sup>13</sup>C NMR (75 MHz) of hpp'H (if proton exchange between the amidine N's of the guanidine is rapid on the NMR time scale) has 5 peaks that are consistent with the structure of hpp'H or hpp'H<sub>2</sub><sup>+</sup>I<sup>-</sup>. The key resonance at δ 150.23 corresponds to the guanidine core carbon atom.

### Solid-State Structure of hppH<sub>2</sub><sup>+</sup>I<sup>-</sup> via Single-Crystal X-ray Diffractometry

What was thought to be hpp'H crystallized in a chiral space group *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>. Surprisingly, the compound studied was the HI salt hpp'H<sub>2</sub><sup>+</sup>I<sup>-</sup>, as shown by single-crystal X-ray diffractometry. The iodide presumably was derived from the last step in the synthesis that used MeI. Therefore, the reaction mixture was not sufficiently neutralized by NaOH. However, the presence of the adventitious I<sup>-</sup> proved fortuitous as it allowed for

direct assignment of the chiral centres. Dempsey's method<sup>64</sup> also generated the iodide salt before neutralization.

The solid state structure of  $\text{hpp}'\text{H}_2^+\text{I}^-$  compares closely to that of  $\text{hpp}'\text{H}$  as synthesized by Cotton and co-workers;<sup>59</sup> they crystallized  $\text{hpp}'\text{H}$  as the  $\text{HCO}_3^-$  salt  $\text{hpp}'\text{H}_2^+(\text{HCO}_3)^-$ . The crystal structure of  $\text{hpp}'\text{H}_2^+\text{I}^-$  (Figure 18) shows the expected

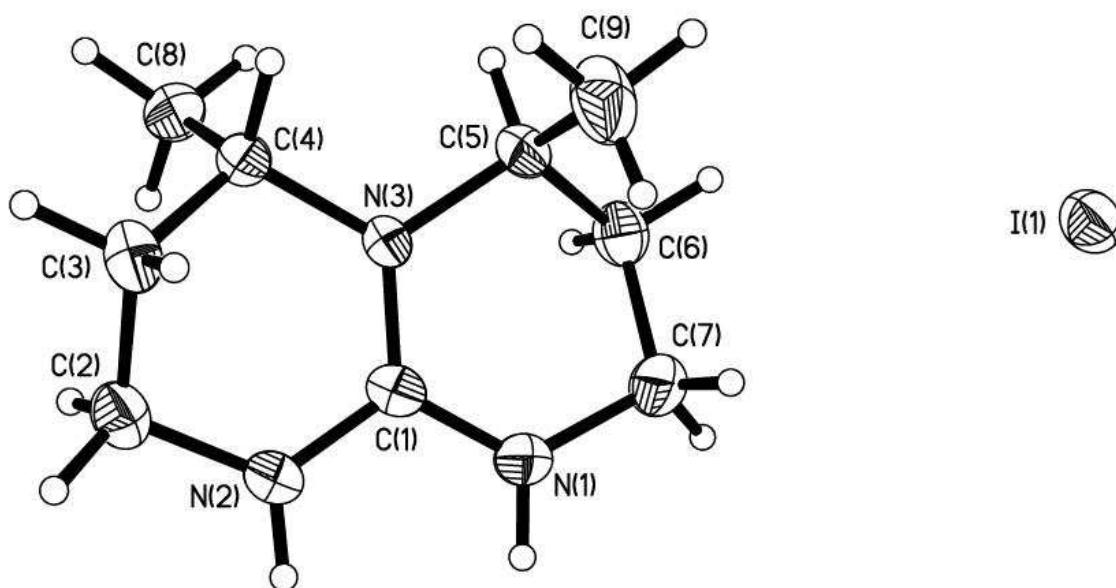


Figure 18: View of solid-state structure of  $\text{hpp}'\text{H}_2^+\text{I}^-$  approximately perpendicular to the bicyclic structure. Thermal ellipsoids are shown at the 50% probability level.

stereochemistry of the methyl groups at the carbons attached to the bridge-head nitrogen (N3), which suggests no racemization and no isomerization during the course of chemical synthesis, though on the basis of the one crystal examined. The C-N bond lengths of the guanidinium core of  $\text{hpp}'\text{H}_2^+\text{I}^-$  ( $\text{C}(1)\text{-N}(3) = 1.323(6) \text{ \AA}$ ,  $\text{C}(1)\text{-N}(1) = 1.337(7) \text{ \AA}$  and  $\text{C}(1)\text{-N}(2) = 1.333(7) \text{ \AA}$ ) are consistent with delocalization of the lone pair of the bridge-head nitrogen (N3) into the guanidinium core. The bond lengths  $\text{C}(1)\text{-N}(1)$  and  $\text{C}(1)\text{-N}(2)$  are similar, consistent with a guanidinium structure. The C-N bond lengths are also

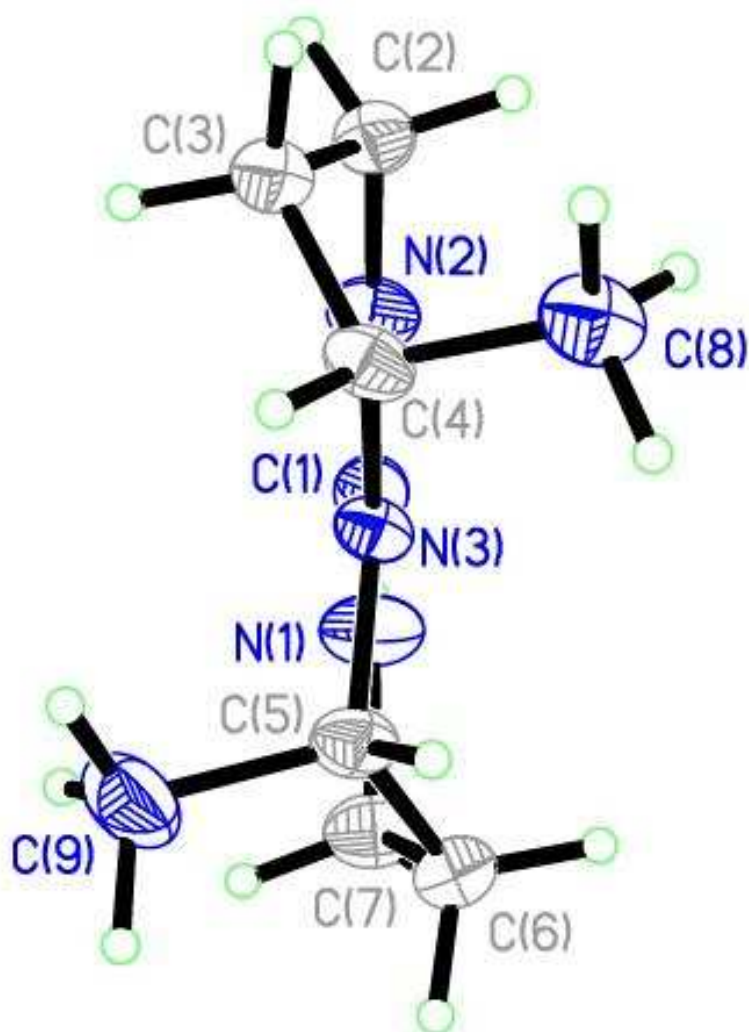


Figure 19: View of solid-state structure of hpp'H<sub>2</sub><sup>+</sup>I down the N(3)-C(1) bond. Thermal ellipsoids are shown at the 50% probability level.

similar to those in Cotton's<sup>59</sup>  $\text{hpp}^*\text{H}_2^+(\text{HCO}_3^-)$  ( $\text{C}(1)\text{-N}(3) = 1.333(2) \text{ \AA}$ ,  $\text{C}(1)\text{-N}(1) = 1.335(2) \text{ \AA}$  and  $\text{C}(1)\text{-N}(2) = 1.334(2) \text{ \AA}$ ). The bond angles around  $\text{C}(1)$  ( $\text{N}(3)\text{-C}(1)\text{-N}(2) = 121.1(5)^\circ$ ,  $\text{N}(3)\text{-C}(1)\text{-N}(1) = 121.6(5)^\circ$  and  $\text{N}(1)\text{-C}(1)\text{-N}(2) = 117.2(5)^\circ$ ) are also very similar to those found in Cotton's  $\text{hpp}^*\text{H}_2^+(\text{HCO}_3^-)$  ( $\text{N}(3)\text{-C}(1)\text{-N}(2) = 121.0(2)^\circ$ ,  $\text{N}(3)\text{-C}(1)\text{-N}(1) = 120.6(2)^\circ$  and  $\text{N}(1)\text{-C}(1)\text{-N}(2) = 118.3(2)^\circ$ ). The bond angles around  $\text{N}(3)$  in  $\text{hppH}_2^+\text{I}$  are  $\text{C}(1)\text{-N}(3)\text{-C}(4) = 122.1(4)^\circ$ ,  $\text{C}(1)\text{-N}(3)\text{-C}(5) = 120.3(4)^\circ$  and  $\text{C}(4)\text{-N}(3)\text{-C}(5) = 117.2(4)^\circ$ . The sum of the angles at both  $\text{C}(1)$  ( $359.9^\circ$ ) and  $\text{N}(3)$  ( $359.6^\circ$ ) show that these atoms are planar, consistent with delocalization of electron density from the bridge-head nitrogen  $\text{N}(3)$  into the core of the ring. Figure 19 shows the planarity of the guanidinium  $\text{N}_2\text{CN}$  core of the ring and also the orientation of the two methyl groups.

### Conclusions

The synthesis of  $\text{hpp}'\text{H}$ , the precursor to the  $C_2$ -symmetric guanidinate  $\text{hpp}'^-$  was based on L-alanine as the chiral precursor. The key step was the coupling of L-alaninol (derived from L-alanine) with commercially available hydroxyacetone via reductive amination. An important step was the diastereoselective resolution of N-benzyl-protected meso and  $C_2$ -symmetric aminodinitriles  $\text{PhCH}_2\text{N}(\text{CH}(\text{CH}_3)\text{CH}_2\text{CN})_2$ . Cyclization of the chiral,  $C_2$ -symmetric triamine  $\text{HN}(\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{NH}_2)_2$ , derived from reduction of the debenzylated aminodinitrile, using dimethyl trithiocarbonate yielded  $\text{hpp}'\text{H}_2^+\text{I}$  in 26% yield (assuming the product obtained was only  $\text{hpp}'\text{H}_2^+\text{I}$  and not neutral  $\text{hpp}'\text{H}$  or a mixture of the two).  $\text{Hpp}'\text{H}$  was isolated as  $\text{hpp}'\text{H}_2^+\text{I}$ , presumably from incomplete neutralization by NaOH, as determined by single-crystal X-ray diffractometry.

Chiral  $\text{hpp}'\text{H}_2^+\text{I}$  was synthesized in six steps, starting from L-alaninol, in 9% overall yield. Chiral  $\text{hpp}'\text{H}_2^+\text{I}$  was characterized by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopies, single-crystal X-ray diffractometry, and high resolution mass spectrometry.

An alternate route to synthesize neutral  $\text{hpp}'\text{H}$ , using carbon disulfide or dimethyl carbonate as C1 reagents, will be explored in the near future.

The potential use of chiral  $\text{hpp}'\text{H}$  as a catalyst in the diastereoselective Henry reaction and enantioselective synthesis of  $\alpha$ -amino acids will be studied in the future.

Also, the coordination chemistry of  $\text{hpp}'^-$  with Ta will be studied. The complexes of  $\text{hpp}'^-$  will be chiral and are postulated to be more soluble than  $\text{hpp}^-$  complexes. Also, the  $C_2$ -symmetry of  $\text{hpp}'^-$  will lead to only a single isomer during the formation of paddlewheel complexes. These future studies will determine whether  $\text{hpp}'^-$  is more basic than  $\text{hpp}^-$  upon coordination to a metal ion.

### Experimental

**General Procedures.** All procedures were performed under an argon atmosphere unless noted and all glassware was oven dried before use. Sodium cyanide, di-tert-butylidicarbonate  $[(\text{Boc})_2\text{O}]$ , carbon tetrabromide, triphenyl phosphine, benzyl bromide, ceric ammonium nitrate, hydroxyacetone, Raney-Nickel (Raney 2800 nickel slurry in water), carbon disulfide (anhydrous) and dimethyl trithiocarbonate were purchased from Aldrich and were used as received. Lithium aluminum hydride and 5-methylisoxazole were purchased from TCI America. Chromium oxide, L-alanine (99% ee), L-phenylalanine, sodium hydride (60% dispersion in mineral oil), and iodomethane were purchased from Acros Organics. L-Alaninol (98% ee) was purchased from Chem-Impex International. Lipase PSC Amano II was a gift from Amano Pharmaceuticals, Japan. Optical rotation was recorded on Rudolph Research Analytical Autopol III polarimeter at 25°C and 589nm.

**Preparation of (S)-L-alaninol.** Lithium aluminum hydride (8.96 g, 0.22 mol) was suspended in dry THF, stirred under argon, and cooled in an ice-bath for 15 minutes. After cooling, L-alanine (10.0 g, 0.11 mol) was added to the mixture using a solid addition funnel, slowly and with constant stirring. L-alanine was completely added over a period of 30 minutes. After complete addition, the ice-bath was removed and the mixture refluxed overnight. The reaction mixture was removed from the oil bath and cooled to room temperature and then in an ice-bath for 15 minutes. To this cooled reaction mixture was added 9 mL of water (very slowly, dropwise as the reaction is very exothermic, especially in the beginning), followed by 9 mL of 15% NaOH solution and finally 27 mL water. This resulted in formation of a white granular solid, which was removed by



filtration and washed with EtOAc (20 mL x 3 times). The resulting filtrate was dried over NaSO<sub>4</sub> and concentrated using a rotary evaporator to yield L-alaninol as a colorless, viscous oil (7.59 g) used in the next step without purification.

**Preparation of 2-hydroxy-1 (S)-methylethyl carbamic acid *tert*-butyl ester ((S)-25) .<sup>62</sup>**

The crude L-alaninol (7.59 g, 0.11 mmol) obtained above was dissolved in 60 mL THF, stirred under argon, and cooled in ice-bath for 15 minutes. (Boc)<sub>2</sub>O (20.95 g, 0.09 mol) dissolved in 20 mL THF was slowly added to the cooled solution of L-alaninol. After complete addition, the mixture was stirred overnight at room temperature. The solvent was removed using a rotary evaporator and the resulting oil dissolved in water and then extracted using CH<sub>2</sub>Cl<sub>2</sub> (50 mL x 3 times). The combined organic layers were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to an oil. The resulting oil solidified to a white solid on cooling to 5°C. The white solid was recrystallized using heptane to yield (S)-25 (12.68 g, 72% overall yield starting from L-alanine) as white crystals. Melting point 62-64°C (lit<sup>62</sup> 60°C). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.1 (d, 3H, 6.6 Hz, -CH-CH<sub>3</sub>), 1.41 (s, 9H, -(CH<sub>3</sub>)<sub>3</sub>), 2.6 (broad s, 1H, -OH), 3.44-3.62 (m, 2H, -CH-CH<sub>2</sub>-), 3.7 (m, 1H, -CH-CH<sub>3</sub>), 4.67 (broad s, 1H, -NH-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 17.19 (-CH<sub>3</sub>), 28.26 (-(CH<sub>3</sub>)<sub>3</sub>), 48.53 (-CH-CH<sub>2</sub>-), 67.21 (-CH-CH<sub>2</sub>-), 79.58 (-O-C(CH<sub>3</sub>)<sub>3</sub>), 156.26 (-NH-CO<sub>2</sub>-).

**Preparation of 2-methylsulfonyloxy-1 (S)-methylethyl carbamic acid *tert*-butyl ester ((S)-26)<sup>62</sup>.** The recrystallized (S)-25 (16.01 g, 0.09 mol) obtained from the above reaction was dissolved in 100 mL CH<sub>2</sub>Cl<sub>2</sub>, cooled in ice bath, and stirred under argon. After stirring for 15 minutes, triethylamine (25.68 mL, 0.18 mol) was added. A solution of methanesulfonyl chloride (9.94 mL, 0.13 mol) in 20 mL CH<sub>2</sub>Cl<sub>2</sub> was slowly added, with constant stirring, to the cooled mixture. After complete addition, the reaction mixture was stirred overnight at room temperature. The reaction mixture was added to 100 mL distilled water and extracted into CH<sub>2</sub>Cl<sub>2</sub> (75 x 3mL). The combined organic layers were washed with 0.1N aqueous HCl and then aqueous NaHCO<sub>3</sub> solution. The organic layers were combined, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated to yield a yellow-white solid. The crude product was used, without purification, in the next step. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.57 (d, 3H, 6.3 Hz, -CH-CH<sub>3</sub>), 1.43 (s, 9H, -(CH<sub>3</sub>)<sub>3</sub>), 2.71-2.89 (m, 2H, -CH-CH<sub>2</sub>-), 3.11 (s, 3H, -OSO<sub>2</sub>CH<sub>3</sub>), 4.96-5.06 (m, 1H, -CH-CH<sub>2</sub>-).

**Preparation of 2-cyano-1 (S)-methylethyl carbamic acid *tert*-butyl ester ((S)-27).**<sup>62</sup>

To a stirred solution of (S)-26 (18.63 g, 0.07 mol) in 140 mL DMSO was added NaCN (10.81 g, 0.22 mol). The mixture was stirred at 45°C for 24 hours. After cooling to room temperature, water (50 mL) was added and the product was extracted into ethyl acetate (4 x 40 mL). The combined organic layers were dried using Na<sub>2</sub>SO<sub>4</sub> and evaporated to yield crude oil. The oil was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.70) to yield (S)-27 as a white solid (12.19 g, 90% based on (S)-25). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.28 (d, 3H, 6.8 Hz, -CH-CH<sub>3</sub>), 1.41 (s, 9H, -(CH<sub>3</sub>)<sub>3</sub>), 2.49-2.52 (m, 1H, -CH-CH<sub>2</sub>-), 2.71-2.74 (m, 1H, -CH-CH<sub>2</sub>-), 3.87-3.95 (m, 1H, -CH-CH<sub>3</sub>), 4.67 (broad s, 1H, -NH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 19.44 (-CH-CH<sub>3</sub>), 25.13 (-(CH<sub>3</sub>)<sub>3</sub>), 28.25 (-CH-CH<sub>2</sub>-), 43.19 (-CH-CH<sub>2</sub>), 80.00 [-OC(CH<sub>3</sub>)<sub>3</sub>], 117.27 (CN), 154.78 (-NH-CO<sub>2</sub>-).

**Preparation of 2-cyano-1-(S)-methylethylammonium trifluoroacetate ((S)-28).** A solution of (S)-27 (14.29 g, 0.08 mol) in 60 mL CH<sub>2</sub>Cl<sub>2</sub> was stirred under argon and cooled in an ice-bath for 10 minutes. A solution of trifluoroacetic acid (29.81 mL, 0.39 mol) in 20 mL CH<sub>2</sub>Cl<sub>2</sub> was slowly added to the reaction mixture, with constant stirring. The mixture was stirred at room temperature for 4 hours. The solvent was then removed using a rotary evaporator. The excess TFA was removed by adding diethyl ether and removing bath with the rotary evaporator. This cycle of adding ether and removing volatiles was continued (6 x 20 mL) until the end product was a white solid, a very fine crystalline powder (9.83 g, 64% based on (S)-27). <sup>1</sup>H NMR (CDCl<sub>3</sub> + CD<sub>3</sub>OD, 300 MHz): δ 1.47-1.49 (d, 3H, 6.6 Hz, -CH-CH<sub>3</sub>), 2.76-2.94 (m, 2H, -CH-CH<sub>2</sub>-), 3.56-3.65 (m, 1H, -CH-CH<sub>2</sub>-), 3.87-3.89 (broad s, 1H, -NH<sub>3</sub><sup>+</sup>). <sup>13</sup>C NMR (CDCl<sub>3</sub> + CD<sub>3</sub>OD, 75 MHz): δ 17.64 (-(CH<sub>3</sub>)), 22.53 (-CH-CH<sub>2</sub>-), 43.90 (-CH-CH<sub>2</sub>), 115.52 (CN).

**Preparation of 3-hydroxybutyronitrile (29).**<sup>75</sup> Diisopropylamine (14.01 mL, 99.89 mmol) was dissolved in 150 mL dry THF and cooled to -20°C (dry ice-acetonitrile bath), and was stirred for 15 minutes under argon. A 2.5 M hexane solution of butyllithium (39.6 mL, 99.01 mmol) was added slowly via syringe. The mixture was stirred at -20°C for 30 minutes. The mixture was then cooled to -80°C (dry ice-acetone bath) and 5.17 mL (99.01 mmol) of acetonitrile in 20 mL THF was added dropwise with stirring for 30 minutes. Ethyl acetate (9.67 mL, 99.01 mmol) dissolved in 15 mL THF was then added dropwise and the resulting mixture stirred at -60°C (dry ice-acetonitrile bath) for 4 hours.

NaBH<sub>4</sub> (2.29 g, 59.52 mmol) was added and the mixture brought to room temperature and stirred for 3 hours. The contents were cooled in an ice-bath and the pH slowly neutralized to just acidic (pH=6) by adding aqueous 4N HCl solution. The resulting white solids were filtered and washed with EtOAc (3 x 20 mL). The organic layers were combined, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuum to yield crude product as a viscous oil with some white precipitate. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.23) to yield pure **29** (8.01 g, 95% based on acetonitrile) as a colorless, viscous oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.26 (d, 3H, 6.3 Hz, CH<sub>3</sub>CH-), 2.43, 2.50 (doublets of AB q, 2H, <sup>2</sup>J<sub>AB</sub> = 17.1 Hz, C<sub>HAHB</sub>, <sup>3</sup>J<sub>AC</sub> = 5.56 Hz, C<sub>HAHC</sub>, -CH-CH<sub>2</sub>-), 3.28 (broad s, 1H, OH), 4.04-4.01 (sextet, 1H, 6 Hz, -CH<sub>2</sub>-CH-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 22.34 (CH<sub>3</sub>), 27.17 (-CH<sub>2</sub>), 63.59 (-CH), 117.74 (CN).

**Preparation of acetic acid-2-cyano-1-methyl-ethyl ester<sup>76</sup> (30).** A solution of 3-hydroxybutyronitrile (**29**) (14.12 g, 0.17 mol) in 200 mL of CH<sub>2</sub>Cl<sub>2</sub> was stirred under argon and cooled in ice-bath. Pyridine (30 mL) was added followed by slow addition of acetyl chloride (23.6 mL, 0.33 mol) dissolved in 20 mL CH<sub>2</sub>Cl<sub>2</sub>. The reaction mixture was stirred overnight at room temperature. The contents of the flask were cooled in ice, and ice was added to quench the reaction. The product was extracted into CH<sub>2</sub>Cl<sub>2</sub> (3 x 25 mL) and washed with concentrated aqueous NaHCO<sub>3</sub> and finally with brine solution. The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield an orange-red oil. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.58) to yield **30** (18.6 g, 88.2 % based on **29**) as a yellow oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.38 (d, 3H, 6.3 Hz, CH<sub>3</sub>-CH-), 2.05 (s, 3H, CH<sub>3</sub>CO-), 2.65, 2.73 (doublets of AB q, 2H, <sup>2</sup>J<sub>AB</sub> = 16.95 Hz, C<sub>HAHB</sub>, <sup>3</sup>J<sub>AC</sub> = 5.40 Hz, C<sub>HAHC</sub>, -CH-CH<sub>2</sub>-), 5.02-5.08 (sextet, 1H, 5.4 Hz, -CH<sub>2</sub>-CH-).

**Preparation of chiral (R)-3-hydroxybutyronitrile ((R)-29): Enzyme catalyzed reaction.**<sup>76</sup> To a solution of **30** (3.01 g, 0.02 mol) in deionized water-acetone (200mL: 30 mL), was added 1,4,8,11-tetrathiacyclotetradecane (0.32 g, 1.18 mmol). Lipase PSC Amano-II (0.31 g) was added and the mixture stirred at 35°C for 24 hours. The contents were then cooled to room temperature and filtered in order to recover the enzyme. The product was extracted into ethyl acetate (3 x 35 mL). The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a viscous oil. The crude was then purified

using flash chromatography (hexane/ethyl acetate 1:1,  $R_f = 0.23$ ) to yield pure **(R)-29** as a colorless oil (1.11 g, 55% based on **30**). The enantiomeric excess of **(R)-29** was determined using chiral GC (90% ee).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  1.26 (d, 3H, 6.0 Hz,  $\text{CH}_3\text{-CH-}$ ), 2.69, 2.43, 3.50 (doublets of AB q, 2H,  $^2J_{\text{AB}} = 17.1$  Hz,  $C_{\text{HAHB}}$ ,  $^3J_{\text{AC}} = 5.56$  Hz,  $C_{\text{HAHC}}$ ,  $\text{-CH-CH}_2\text{-}$ ), 3.28 (broad s, 1H,  $\text{OH}$ ), 4.04-4.01 (sextet, 1H, 6 Hz,  $\text{-CH}_2\text{-CH-}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  22.34 ( $\text{CH}_3$ ), 27.17 ( $\text{-CH}_2\text{-}$ ), 63.59 ( $\text{-CH-}$ ), 117.74 ( $\text{CN}$ ).

**Preparation of methanesulfonic acid-2-cyano-1(R)-methylethyl ester ((R)-31).** A solution of **(R)-29** (2.01 g, 0.02 mol) in 30 mL  $\text{CH}_2\text{Cl}_2$  was stirred under argon and cooled in an ice-bath for 10 minutes. Triethylamine (6.61 mL, 0.05 mol) was added, followed by a solution of methanesulfonyl chloride (2.54 mL, 0.03 mol) in 5 mL  $\text{CH}_2\text{Cl}_2$ . The reaction mixture was refluxed for 4 hours, cooled to room temperature, and mixed with distilled water. The product was extracted with  $\text{CH}_2\text{Cl}_2$  (3 x 15 mL) and washed with 0.1 N aqueous HCl and aqueous  $\text{NaHCO}_3$  solution. The organic phase was dried with  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum to yield an oil. The crude oil was purified using flash chromatography (hexane/ethyl acetate 1:1,  $R_f = 0.4$ ), to yield **(R)-31** as orange-yellow oil (2.99 g, 78% based on **(R)-29**).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  1.46 (d, 3H, 6 Hz,  $\text{CH}_3\text{-CH-}$ ), 2.84, 2.65 (doublets of AB q, 2H,  $^2J_{\text{AB}} = 17.1$  Hz,  $C_{\text{HAHB}}$ ,  $^3J_{\text{AC}} = 5.6$  Hz,  $C_{\text{HAHC}}$ ,  $\text{-CH-CH}_2\text{-}$ ), 3.01 (s, 3H,  $\text{-O-SO}_2\text{-CH}_3$ ), 4.90 (sextet, 1H, 5.7 Hz,  $\text{-CH-CH}_2\text{-}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  20.82 ( $\text{CH}_3\text{-CH-}$ ), 25.32 ( $\text{-CH}_2\text{-CN}$ ), 38.40 ( $\text{-O-SO}_2\text{-CH}_3$ ), 72.78 ( $\text{-O-CH-}$ ), 115.78 ( $\text{CN}$ ).

**Preparation of cyanoacetone.** A solution of 3-hydroxybutyronitrile **29** (2.01 g, 0.02 mol) in 50 mL acetone was cooled in an ice-bath. Jones reagent (12 mL, prepared by adding 5.01 g of  $\text{CrO}_3$  to 5 mL conc.  $\text{H}_2\text{SO}_4$ , followed by adding to 15 mL cooled water to give an orange-red solution) was slowly added to the cooled solution until the red color of Jones reagent persisted. The mixture was then refluxed for 1 hour, followed by cooling to room temperature. The pH of the reaction mixture was neutralized to just basic by adding 20% aqueous NaOH solution. The resulting precipitate was filtered through Celite and the product extracted into EtOAc (3 x 25 mL). The organic extracts were dried over  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum to yield cyanoacetone (1.46 g, 75% based on **29**). The product obtained was pure by TLC and  $^1\text{H}$  NMR spectroscopy. Cyanoacetone

prepared above was used immediately as it decomposes to an orange-red polymer (which is not soluble in common organic solvents) in air for 10 minutes.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  2.4 (s, 3H,  $\text{CH}_3$ ), 3.68 (s, 2H,  $\text{CH}_2$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  29.27 ( $\text{CH}_3$ ), 32.86 ( $-\text{CH}_2$ ), 114.16 ( $-\text{CN}$ ), 198.12 ( $-\text{C}=\text{O}$ ).

**Coupling of cyanoacetone with (S)-28 via reductive amination.** Freshly-prepared cyanoacetone (1.46 g, 17.51 mmol) was dissolved in 30 mL dry THF and stirred under argon.  $\text{Ti}(\text{O}i\text{Pr})_4$  (6.31 mL, 21.01 mmol) was added slowly to the reaction mixture with stirring at room temperature for 10 minutes. (S)-28 (3.48 g, 17.51 mmol) was added and the mixture was stirred at room temperature for 5 hours.  $\text{NaBH}_4$  (1.38 g, 35.01 mmol) was then added very slowly and the contents stirred at room temperature overnight. The reaction was quenched with 30 mL 1N aqueous NaOH (added very slowly as the reaction was rapid and resulted in the formation of bubbles) to give a white precipitate. The white precipitate was filtered and the solvent removed under vacuum. The resulting oil was dissolved in distilled water and the products extracted into  $\text{CH}_2\text{Cl}_2$  (3 x 25 mL), dried over  $\text{Na}_2\text{SO}_4$ , and concentrated under vacuum. The crude products were analyzed by GC-MS and were determined to be the reduced secondary alcohol of cyanoacetone and the neutral chiral amine (S)-28. The desired diastereomers **B** were not obtained.

**Preparation of the sodium salt of cyanoacetone.**<sup>78</sup> Sodium methoxide (1.95 g, 36.01 mmol) was suspended in 10 mL absolute EtOH, cooled in an ice-bath and stirred under argon. After 10 minutes, a solution of 5-methylisoxazole (3.01 g, 36.01 mmol) in 10 mL EtOH was added slowly dropwise. The mixture was stirred at room temperature overnight. The solvent was removed under vacuum and the white solid (3.61 g, 95% based on 5-methylisoxazole) dried under vacuum and then used in the next step without purification.

**Preparation of cyanoacetone from sodium salt of cyanoacetone.**<sup>79</sup> The sodium salt of cyanoacetone (1.01 g, 9.51 mmol) was suspended in a 1:1 mixture of  $\text{CH}_2\text{Cl}_2$ : $\text{H}_2\text{O}$  (15 mL: 15 mL). Aqueous 2N HCl solution was added dropwise until the pH of the solution reached 1. The product was extracted into  $\text{CH}_2\text{Cl}_2$  (3 x 20 mL), dried under  $\text{Na}_2\text{SO}_4$ , and concentrated under vacuum to yield cyanoacetone (0.59 g, 75%) as a colorless oil. The product obtained was used immediately for the next step as it decomposes to an orange-red polymer in air.

**Preparation of 3-bromobutyronitrile (32).** A solution of 3-hydroxybutyronitrile (**29**) (4.01 g, 47.01 mmol) in 50 mL CH<sub>2</sub>Cl<sub>2</sub> was cooled in an ice-bath and stirred for 10 minutes. Triphenylphosphine (14.79 g, 56.41 mmol) was added, followed by dropwise addition of carbon tetrabromide (18.71 g, 56.41 mmol) in 10 mL CH<sub>2</sub>Cl<sub>2</sub> using an addition funnel over a period of 10 minutes. The reaction mixture was stirred at room temperature for 14 hours. Water (20 mL) was added and the mixture extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 15 mL). The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a crude yellow-orange solid. The solid was purified using column chromatography (hexane/ethyl acetate 6:4, R<sub>f</sub> = 0.56) to yield **32** as a colorless oil (4.81 g, 70% based on **29**). The product **32** is very volatile, so concentration on a rotary evaporator must be done very carefully in order to minimize product loss. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.84 (d, 3H, 6.6 Hz, CH<sub>3</sub>-CH-), 2.98 (d, 2H, 6 Hz, -CH-CH<sub>2</sub>-), 4.28 (m, 1H, -CH-CH<sub>2</sub>-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 25.29 (CH<sub>3</sub>-CH-), 29.54 (-CH-CH<sub>2</sub>-), 40.19 (-CH-CH<sub>2</sub>-), 116.51 (CN).

**Preparation of 1-hydroxy-3-(S)-phenyl propyl carbamic acid *tert*-butyl ester (33).** Lithium aluminum hydride (8.01 g, 0.21 moles) was suspended in 250 mL dry THF under argon and cooled in an ice-bath. L-Phenylalanine (16.59 g, 0.11 moles) was slowly added using a solid-addition funnel, keeping the temperature of the reaction flask below 40°C, over a period of 45 minutes. The ice-bath was removed and the mixture allowed to rise to room temperature. The reaction mixture was refluxed for 14 hours. The flask was cooled to room temperature, then in an ice-bath for 10 minutes. The excess lithium aluminum hydride was destroyed by slowly adding 8 mL H<sub>2</sub>O, followed by 8 mL of 15% NaOH and then finally with 24 mL of H<sub>2</sub>O. The resulting white granular solid was stirred for 5 minutes. (Boc)<sub>2</sub>O (21.91 g, 0.11 moles) dissolved in 20 mL dry THF, was added to the reaction mixture slowly, with stirring. The resulting mixture was refluxed overnight. The white granular solids were removed by filtration and washed with EtOAc (5 x 25 mL). The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield **33** (22.7 g, 90% based on L-phenylalanine) as a white solid. The solid was used in the next step without purification. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 1.40 (s, 9H, -C(CH<sub>3</sub>)<sub>3</sub>), 2.83 (d, 2H, 7.05 Hz, -CH<sub>2</sub>Ph), 3.49-3.65 (m, 2H, -CH-CH<sub>2</sub>-), 3.86 (m, 1H, -CH-CH<sub>2</sub>-), 4.92 (broad s, 1H, -NH-CO-), 7.20-7.29 (m, 5H, Ph).

**Preparation of methanysulfonyloxy-2 (S)-3-phenyl propyl carbamic acid *tert*-butyl ester (34).** A solution of **33** (22.51 g, 89.51 mmol) in 100 mL CH<sub>2</sub>Cl<sub>2</sub> under argon was cooled in an ice bath. After stirring for 15 minutes, triethylamine (25.18 mL, 0.18 mol) was added followed by solution of methanesulfonyl chloride, (8.35 mL, 0.11 mol) in 20 mL CH<sub>2</sub>Cl<sub>2</sub>. The reaction mixture was stirred overnight at room temperature, added to 100 mL distilled water, and extracted into CH<sub>2</sub>Cl<sub>2</sub> (5 x 30 mL). The combined organic layers were washed with 0.1N aqueous HCl and saturated aqueous NaHCO<sub>3</sub> solution. The organic layers were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a yellow-white solid. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.53) to give **34** as a white solid (22.72 g, 77% based on **33**). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.43 (s, 9H, -C(CH<sub>3</sub>)<sub>3</sub>), 2.88 (m, 2H, -CH<sub>2</sub>Ph), 3.02 (s, 3H, -OSO<sub>2</sub>CH<sub>3</sub>), 4.1-4.24 (m, 3H, -CH<sub>2</sub>-CH-), 4.8 (m, 1H, -NH-CO-), 7.24-7.33 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 28.23 (-C(CH<sub>3</sub>)<sub>3</sub>), 37.16 (-OSO<sub>2</sub>CH<sub>3</sub>), 49.20 (-CH-NH-), 69.76 (-CH<sub>2</sub>-O-), 84.42 [-O-C(CH<sub>3</sub>)<sub>3</sub>], 126.89, 128.70, 129.20, 136.54 (Ph), 155.86 (-NH-C=O).

**Preparation of 3-cyano-2 (S)-phenyl propyl carbamic acid *tert*-butyl ester (35).** To a stirred solution of **34** (22.68 g, 68.8 mmol) in 250 mL dry DMSO, was added NaCN (8.43 g, 172 mmol). The contents of the reaction mixture were stirred at 50°C for 24 hours. The reaction was quenched with 200 mL water and 100 mL brine solution. The product was extracted with EtOAc (4 x 60 mL) and the combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a dark-brown oil. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.75) to yield **35** as a white solid (11.06 g, 61% based on **34**). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.4 (s, 9H, -C(CH<sub>3</sub>)<sub>3</sub>), 2.35-2.42 (dd, 1H, <sup>2</sup>J<sub>AB</sub> = 16.8 Hz, C<sub>H</sub>AHB, <sup>3</sup>J<sub>AC</sub> = 4.32. C<sub>H</sub>AHC, -CH<sub>2</sub>Ph), 2.65-2.67 (m, 1H, -CH<sub>2</sub>-CH-), 2.79-2.87 (m, 1H, -CH<sub>2</sub>Ph), 2.96-2.98 (m, 1H, CH<sub>2</sub>-CH-), 4.05 (m, 1H, -CH<sub>2</sub>-CH-), 4.74-4.77 (m, 1H, -NH-CO-), 7.19-7.32 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 22.34 (-CH<sub>2</sub>-CN), 28.15 (-C(CH<sub>3</sub>)<sub>3</sub>), 39.26 (CH<sub>2</sub>Ph), 48.36 (-CH-NH-), 80.16 [-O-C(CH<sub>3</sub>)<sub>3</sub>], 117.24 (CN), 127.12, 128.82, 128.98, 136.0 (Ph), 154.74 (-NH-C=O).

**Preparation of 3-amino-4-(S)-phenyl-butyronitrile ((S)-36).** Pure **35** (10.01 g, 38.41 mmol) was dissolved in 100 mL CH<sub>2</sub>Cl<sub>2</sub> and stirred under argon. Dry HCl gas was bubbled through the solution via a needle through a septum. Immediately, white solid

precipitated. Dry HCl gas was added for 30 minutes until the spot on TLC for **35** disappeared. The resulting white solid was filtered and dried under vacuum. The white solid was suspended in 100 mL CH<sub>2</sub>Cl<sub>2</sub> and the pH of the solution increased by adding Et<sub>3</sub>N until the pH was basic (~8.0 using pH paper). Water was added and the product was extracted into CH<sub>2</sub>Cl<sub>2</sub> (3 x 50 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a viscous oil. The crude product was purified by flash chromatography (CH<sub>2</sub>Cl<sub>2</sub>/MeOH 9:1, R<sub>f</sub> = 0.47) to yield pure (**S**)-**36** (5.51 g, 90% based on **35**) as a colorless, viscous oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.38 (broad s, 2H, -NH<sub>2</sub>), 2.35, 2.45 (doublets of AB q, 2H, <sup>2</sup>J<sub>AB</sub> = 17.1 Hz, C<sub>HAHB</sub>, <sup>2</sup>J<sub>AC</sub> = 5.73 Hz, C<sub>HAHC</sub>, -CH<sub>2</sub>-Ph), 2.73, 2.83 (doublets of AB q, 2H, <sup>2</sup>J<sub>AB</sub> = 16.1 Hz, C<sub>HAHB</sub>, <sup>2</sup>J<sub>AC</sub> = 7.56 Hz, C<sub>HAHC</sub>, -CH<sub>2</sub>-CN), 3.33-3.38 (quintet, 1H, 5.78 Hz -CH<sub>2</sub>-CH-), 7.17-7.31 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 25.82 (-CH<sub>2</sub>-CN), 43.20 (CH<sub>2</sub>Ph), 49.74 (-CH-CH<sub>2</sub>-), 117.99 (CN), 126.86, 128.70, 129.04, 137.25 (Ph).

**Preparation of 3-oxo-4-phenylbutyronitrile (37).** Freshly distilled (from KOH) diisopropylamine (14.00 mL, 99.61 mmol) was dissolved in dry THF (200 mL), stirred under argon, and cooled to -20°C in a dryice-acetonitrile bath. After 15 minutes, 2.5 M BuLi (40.10 mL, 99.61 mmol) was added very slowly, with constant stirring over 10 minutes. The mixture was stirred at -20°C for 30 minutes, then cooled to -80°C (dry ice-acetone bath). A solution of CH<sub>3</sub>CN (5.00 mL, 95.71 mmol) in 10 mL THF was added slowly to yield a white, milky suspension. The mixture was stirred for 30 minutes. Ethyl phenylacetate (15.00 mL, 94.18 mmol) dissolved in 10 mL THF was added to the mixture, resulting in a clear yellow solution. The mixture was stirred at -60°C (dry ice-acetonitrile bath) for 4 hours then allowed to warm to room temperature. The mixture was cooled in an ice-bath and the pH neutralized to 7 using aqueous 1N HCl. The product was extracted into EtOAc (5 x 30 mL), dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated to yield a yellow, viscous oil. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.63) to give **37** (9.14 g, 60% based on acetonitrile) as a yellow-white solid. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 3.4 (s, 2H, -CH<sub>2</sub>-CN), 3.82 (s, 2H, -CH<sub>2</sub>-Ph), 7.17-7.35 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 31.13 (-CH<sub>2</sub>-CN), 49.03 (-CH<sub>2</sub>-Ph), 113.65 (CN), 127.82, 129.10, 129.34, 131.87 (Ph), 195.38 (-C=O).

**Preparation of 3-hydroxy-4-phenylbutyronitrile (38).** Diisopropylamine (20 mL, 142 mmol) was dissolved in dry THF (250mL) in a three-neck flask under argon and cooled



at  $-20^{\circ}\text{C}$  (dry ice-acetonitrile bath) for 15 minutes. A hexane solution of 2.5 M BuLi (57 mL, 142 mmol) was slowly added, using a syringe, with constant stirring. The contents of the flask were stirred at  $-20^{\circ}\text{C}$  for 30 minutes. The mixture was cooled to  $-70^{\circ}\text{C}$  in a dryice- acetone bath for 30 minutes, and  $\text{CH}_3\text{CN}$  (7.3 mL, 140 mmol) diluted with 10 mL THF was slowly added to the reaction mixture to give a white-turbid solution. Ethyl phenylacetate (22.29 mL, 140 mmol), diluted with 20 mL THF, was added slowly to the reaction mixture. The mixture was stirred at  $-60^{\circ}\text{C}$  (dry ice-acetonitrile bath) for 4 hours. Sodium borohydride (5.4 g, 142 mmol) was slowly added and the reaction mixture allowed to warm to room temperature and stirred overnight. The reaction mixture was cooled in an ice-bath and the pH was neutralized with 2N aqueous HCl to pH 6. The product was extracted with EtOAc (4 x 30 mL), and the extracts were dried over  $\text{Na}_2\text{SO}_4$  and then concentrated to yield a light yellow oil. The crude oil was purified using flash chromatography (hexane/ethyl acetate 1:1,  $R_f = 0.47$ ) to give **38** (16.12 g, 72% based on acetonitrile) as a colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  2.43, 2.51 (doublet of AB q, 2H,  $^2J_{\text{AB}} = 16.74$  Hz,  $\text{C}_{\text{HAHB}}$ ,  $^3J_{\text{AC}} = 5.5$  Hz,  $\text{C}_{\text{HAHC}}$ ,  $-\underline{\text{CH}}_2\text{-CN}$ ), 2.83-2.86 (m, 2H,  $\text{Ph-CH}_2$ -), 3.19-3.21 (m, 1H,  $\text{CH-OH}$ ), 4.07-4.09 (m, 1H,  $-\underline{\text{CH}}\text{-CH}_2$ -), 7.18-7.30 (m, 5H, Ph).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  24.79 ( $-\underline{\text{CH}}_2\text{-CN}$ ), 42.45 ( $\text{Ph-CH}_2$ -), 68.23 ( $-\underline{\text{CH}}\text{-OH}$ ), 117.64 ( $\underline{\text{CN}}$ ), 126.82, 128.57, 129.14, 136.36 (Ph).

**Preparation of 3-bromo-4-phenylbutyronitrile (39).** A solution of **38** (3.00 g, 18.6 mmol) in 25 mL  $\text{CH}_2\text{Cl}_2$  in a three-neck flask was cooled in an ice-bath. Triphenylphosphine (5.80 g, 22.31 mmol) was added, followed by dropwise addition of carbon tetrabromide (7.41 g, 22.31 mmol) in 10 mL  $\text{CH}_2\text{Cl}_2$  using an addition funnel. The reaction mixture was stirred at room temperature for 12 hours, then added to 10 mL water. The product was extracted into  $\text{CH}_2\text{Cl}_2$  (3 x 15 mL). The combined organic layers were dried over  $\text{Na}_2\text{SO}_4$  and concentrated to yield a solid. The crude product was purified using column chromatography (hexane/ethyl acetate 7:3,  $R_f = 0.6$ ) to yield **39** (2.9 g, 70% based on **38**) as a colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  2.83-2.87 (m, 2H,  $-\underline{\text{CH}}_2\text{-CN}$ ), 3.18-3.44 (m, 2H,  $\text{Ph-CH}_2$ -), 4.22-4.35 (quintet, 1H, 6.2 Hz,  $-\underline{\text{CH}}\text{-Br}$ ), 7.12-7.34 (m, 5H, Ph).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$ : 26.81 ( $-\underline{\text{CH}}_2\text{-CN}$ ), 44.07 ( $\text{Ph-CH}_2$ -), 45.42 ( $-\underline{\text{CH}}\text{-Br}$ ), 116.45 ( $\underline{\text{CN}}$ ), 127.49, 128.78, 128.96, 136.24 (Ph).

**Preparation of 2-(2-hydroxy-1-methyl-ethylamino)propan-1-ol (42).**<sup>83</sup> L-Alaninol (15.36 g, 204.5 mmol) was added to 350 mL MeOH, in a pressure reactor glass bottle, followed by hydroxyacetone (17.15 mL, 224.95 mmol). PtO<sub>2</sub> (0.15 g) was added to the reaction mixture and the reactor bottle was pressurized with H<sub>2</sub> gas at 50 psi. The mixture was stirred at room temperature for 15 hours. The brown catalyst (PtO<sub>2</sub>) turned black after stirring under H<sub>2</sub>. The mixture was filtered through Celite and the solids washed with 10 mL MeOH. The filtrates were combined and concentrated on a rotary evaporator to yield a viscous oil. The crude product was purified using vacuum distillation (90-95°C/120 mTorr, literature value<sup>83</sup> = 83°C/150 mTorr) to yield pure **42** (24.27 g, 89% based on L-alaninol) as a highly viscous oil (mixture of diastereomers, 1:0.9 by <sup>1</sup>H NMR integration). The product was stored at 5°C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 0.89-0.96 (d, 6H, 6.5 Hz, CH<sub>3</sub>-CH-), 2.27-2.84 (m, 2H, -CH-CH<sub>2</sub>-), 3.18-3.29 (m, 4H, -CH-CH<sub>2</sub>-), 3.44-3.50 (m, 4H, -CH-CH<sub>2</sub>-), 3.9 (broad s, 3H, -NH + -OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 16.29, 17.79 (CH<sub>3</sub>-CH-), 50.68, 51.80 (-CH-CH<sub>2</sub>-), 65.06, 66.02 (-CH-CH<sub>2</sub>-).  $[\alpha]_{589\text{nm}}^{25^\circ\text{C}} = +21.45^\circ$  (*c* = 0.1434, EtOH). HRMS (ESI) calculated for C<sub>6</sub>H<sub>16</sub>NO<sub>2</sub> (M+H): 134.1181, found 134.1185.

**Preparation of 2-[benzyl-(2-hydroxy-1-methyl-ethyl)-amino]propan-1-ol (43).** Pure **42** (18.5 g, 138.9 mmol) was dissolved in 150 mL dry CH<sub>3</sub>CN in a three-neck flask under argon. NaHCO<sub>3</sub> (17.51 g, 208.3 mmol) and dry benzyl bromide (18.17 mL, 152.79 mmol) was added and the mixture refluxed for 20 hours. The reaction mixture was cooled to room temperature and the white solid filtered and washed with 10 mL CH<sub>3</sub>CN. The filtrate was concentrated using a rotary evaporator to a viscous oil. The crude oil was purified using flash chromatography (hexane/ethyl acetate 2:8, R<sub>f</sub> = 0.31) to yield pure **43** (27.58 g, 89% based on **42**) as a white solid (mixture of diastereomers). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 0.97-0.99 (d, 5H, 6.6 Hz, CH<sub>3</sub>-CH-), 1.05-1.07 (d, 6H, 6.75 Hz, CH<sub>3</sub>-CH-), 2.62 (broad s, 4H, OH), 2.99-3.05 (m, 4H, CH<sub>3</sub>-CH-), 3.34-3.39 (m, 8H, -CH-CH<sub>2</sub>-), 3.52-3.56 (d, 1H, 13.98 Hz, -CH<sub>2</sub>-Ph), 3.72 (s, 2H, -CH<sub>2</sub>-Ph), 3.80-3.84 (d, 1H, 14 Hz, -CH<sub>2</sub>-Ph), 7.23-7.31 (m, 10H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 12.79, 14.92 (CH<sub>3</sub>-CH-), 47.64, 48.78 (-CH<sub>2</sub>-Ph), 52.46, 56.54 (CH<sub>3</sub>-CH-), 63.79, 64.17 (-CH<sub>2</sub>-OH), 126.96, 128.38, 128.46, 128.73 (Ph).  $[\alpha]_{589\text{nm}}^{25^\circ\text{C}} = +60.68^\circ$  (*c* = 0.0515, CH<sub>2</sub>Cl<sub>2</sub>). HRMS (ESI) calculated for C<sub>13</sub>H<sub>21</sub>NO<sub>2</sub> (M+H): 224.1651, found 224.1658.

**Preparation of benzylbis(2-bromo-1-methylethyl)amine (44).** A three-neck flask was charged with pure **43** (8.08 g, 36.2 mmol) and dissolved in 50 mL of dry benzene. The flask was stirred under argon and cooled in an ice-bath for 15 minutes. PBr<sub>3</sub> (10.21 mL, 108.6 mmol) was slowly added (with constant stirring), leading to the formation of a white precipitate. After complete addition, an orange precipitate formed. The mixture was refluxed for 4 hours, and then was brought to room temperature. Benzene was decanted and the orange viscous oil was washed with Et<sub>2</sub>O (10 mL). The pH of the orange viscous oil was slowly neutralized to pH = 8.0 using saturated aqueous NaHCO<sub>3</sub>. The product was extracted into EtOAc (4 x 30 mL). The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated using rotary evaporation to yield **44** (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.9) as a brown oil (8.98 g, 71% based on **43**). The product was pure by TLC and NMR spectroscopy and used in the next step without purification. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.62-1.66 (m, 6H, CH<sub>3</sub>-CH-), 2.65-2.91 (m, 4H, -CH<sub>2</sub>-CH-), 3.56-3.79 (m, 2H, Ph-CH<sub>2</sub>-), 4.02-4.04 (m, 2H, -CH<sub>2</sub>-CH-), 7.27-7.31 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 23.81 23.89 (CH<sub>3</sub>-CH-), 47.71, 47.76 (-CH<sub>2</sub>-Ph), 59.68, 59.86 (-CH-CH<sub>3</sub>), 63.70, 64.13 (-CH<sub>2</sub>-Br), 127.22, 127.26, 128.17, 128.23, 128.83, 128.94, 138, 28, 138.36 (Ph). Mass Spectrometry EI (347:349:351; 1:2:1; two Br isotope pattern in mass spectrum).

**Preparation of 3-[benzyl-(2-cyano-1-methylethyl)amino]butyronitrile (45).** Compound **44** (8.99 g, 25.75 mmol) was dissolved in 80 mL dry DMF in a three-neck flask. Sodium cyanide (5.05 g, 103.01 mmol) was added and the contents heated at 40-50°C for 20 hours. The reaction mixture was cooled to room temperature. Water (100 mL) was added, and the product extracted into CH<sub>2</sub>Cl<sub>2</sub> (3 x 30 mL). The combined organic extracts were washed with brine solution, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated under vacuum to yield an oil. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.63) to yield pure **45** (6.48 g, 97% based on **44**) as a white solid. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.13-1.20 (m, 6H, CH<sub>3</sub>-CH-), 2.23-2.40 (m, 4H, -CH<sub>2</sub>-CH-), 3.13-3.26 (m, 2H, -CH<sub>2</sub>-CH-), 3.53-3.70 (m, 2H, -CH<sub>2</sub>-Ph), 7.24-7.29 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 16.17, 18.16 (CH<sub>3</sub>-CH-), 22.7, 24.51 (-CH<sub>2</sub>-CH-), 48.56, 48.97 (-CH<sub>2</sub>-Ph), 49.84, 49.97 (-CH<sub>2</sub>-CH-), 118.48 (CN), 127.11, 127.15, 127.89, 128.01, 128.34, 128.36, 138.77, 138.80 (Ph).

**Preparation of 3-[benzyl-(2-cyano-1-methyl-ethyl)-amino]-butyronitrile (45) via a mesylate route.** A solution of **43** (15.31 g, 68.61 mmol) in dry 200 mL CH<sub>2</sub>Cl<sub>2</sub> was stirred under argon and cooled in an ice-bath. Triethylamine (24.01 mL, 171.41 mmol) was added, followed by slow addition of methanesulfonyl chloride (11.19 mL, 143.0 mmol). The resulting solution was stirred for two hours at room temperature. The solvent was removed using a rotary evaporator and the resulting oil suspended in 120 mL dry DMF. Sodium cyanide (13.44 g, 274.2 mmol) was added and the mixture was heated at 40°C for 20 hours. The solvent was removed using a rotary evaporator and the resulting oil was suspended in 100 mL water and extracted into EtOAc (4 x 50 mL). The combined organic extracts were dried using Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum to yield a deep brown viscous oil. The crude product was purified using flash chromatography (hexane/ethyl acetate 1:1, R<sub>f</sub> = 0.63) to yield pure **45** (11.55 g, 70% based on **43**) as a white solid. <sup>1</sup>H and <sup>13</sup>C NMR peaks were identical to those mentioned above.  $[\alpha]_{589\text{nm}}^{25^\circ\text{C}} = +21.87^\circ$  (*c* = 0.12, CHCl<sub>3</sub>). HRMS (ESI) calculated for C<sub>15</sub>H<sub>20</sub>N<sub>3</sub> (M+H): 242.1657, found 242.1658.

**Separation of diastereomers of 45 to yield pure, chiral C<sub>2</sub>-symmetric 45.** Purified **45** (8.69 g, as a mixture of diastereomers) was suspended in 2 mL CH<sub>2</sub>Cl<sub>2</sub> and stirred at room temperature for 4 hours. The solid was filtered, washed with cold CH<sub>2</sub>Cl<sub>2</sub>: hexane (2:8) and dried under vacuum to yield a white solid (3.37 g). This white solid was again suspended in 2 mL CH<sub>2</sub>Cl<sub>2</sub> and stirred at room temperature for 2 hours. The white solid was filtered, washed with cold CH<sub>2</sub>Cl<sub>2</sub>: hexane (2:8) and dried under vacuum to yield pure **45** (2.45 g) as a single diastereomer. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.23 (d, 6H, 6.66 Hz, CH<sub>3</sub>-CH-) δ 2.44, 2.80 (doublet of AB q, 4H, <sup>2</sup>J<sub>AB</sub> = 16.75 Hz, C<sub>H</sub>AHB, <sup>3</sup>J<sub>AC</sub> = 7.23 Hz, C<sub>H</sub>AHC, -CH<sub>2</sub>-CN), 3.27 (sextet, 2H, 6.84 Hz, CH<sub>3</sub>-CH-), 3.6 (d, 1H, 14.5 Hz, -CH<sub>2</sub>-Ph), 3.81 (d, 1H, 14.5 Hz, -CH<sub>2</sub>-Ph), 7.22-7.35 (m, 5H, Ph). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 16.54 (CH<sub>3</sub>-CH-), 24.86 (-CH<sub>2</sub>-CH-), 49.29 (CH<sub>3</sub>-CH-), 50.44 (-CH<sub>2</sub>-Ph), 118.51 (CN), 127.47, 128.21, 128.63, 138.93 (Ph).  $[\alpha]_{589\text{nm}}^{25^\circ\text{C}} = +72.09^\circ$  (*c* = 0.055, CHCl<sub>3</sub>).

**Preparation of 3-(2-cyano-1-methylethylamino)butyronitrile (B).** In a three-neck flask, pure **45** (14.79 g, 61.3 mmol) was dissolved in a CH<sub>3</sub>CN:H<sub>2</sub>O (200 mL:40 mL) solvent mixture under argon. The mixture was cooled in an ice-bath, and ceric

ammonium nitrate (70.56 g, 128.71 mmol) was slowly added with constant stirring. The mixture was stirred at room temperature for three hours, cooled in an ice-bath, and treated with aqueous saturated  $\text{NaHCO}_3$  until the pH increased to 8.0. The products were then extracted into EtOAc (5 x 40 mL). The organic extracts were dried over  $\text{Na}_2\text{SO}_4$  and concentrated using a rotary evaporator to yield an oil. The crude product was purified using flash chromatography (hexane/ethyl acetate 2:8,  $R_f = 0.44$ ) to yield pure **B** (8.79 g, 95% based on **45**) as a yellow-orange oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  1.01 (broad s, 1H,  $\text{NH}$ ), 1.18 (d, 6H, 6.4 Hz,  $\text{CH}_3\text{-CH-}$ ), 2.37, 2.43 (doublet of AB q, 4H,  $^2J_{\text{AB}} = 16.70$  Hz,  $C_{\text{HAHB}}$ ,  $^3J_{\text{AC}} = 5.46$  Hz,  $C_{\text{HAHC}}$ ,  $-\text{CH}_2\text{-CN}$ ), 3.08 (sextet, 2H, 6.0 Hz,  $\text{CH}_3\text{-CH-}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  20.86 ( $\text{CH}_3\text{-CH-}$ ), 25.43 ( $-\text{CH}_2\text{-CH-}$ ), 47.12 ( $-\text{CH}_2\text{-CH-}$ ), 117.64 ( $\text{CN}$ ).  $[\alpha]_{589\text{nm}}^{25^\circ\text{C}} = -37.52^\circ$  ( $c = 0.11$ ,  $\text{CHCl}_3$ ). HRMS (ESI) calculated for  $\text{C}_8\text{H}_{14}\text{N}_3$  (M+H): 152.1188, found 152.1192.

**Preparation of 3-(amino-1-methylpropyl)butane-1,3-diamine (A).** A glass pressure reactor bottle was charged with pure **B** (8.78 g, 58 mmol), dissolved in 70 mL EtOH and 5 mL  $\text{H}_2\text{O}$ . Sodium hydroxide (1.25 g) was added and the mixture was stirred to dissolve the NaOH. Raney nickel (1.25 g) was washed with  $\text{H}_2\text{O}$  (2 mL x 5 times) and then EtOH (2 mL x 5 times) and transferred to the reaction mixture. The pressure bottle was pressurized with dihydrogen gas (50 psi) and the mixture stirred at room temperature for 24 hours. Fresh washed (water and EtOH) Raney nickel (0.5 g) was again added and the reaction stirred for additional 12 hours under 50 psi of dihydrogen gas. The catalyst mixture was filtered through Celite (caution: catalyst was never dried and always left wet with EtOH) and the solids washed with 5 mL EtOH. The filtrate was concentrated using a rotary evaporator to yield a viscous oil. The oil was dissolved in 20 mL saturated NaOH and extracted with  $\text{CH}_2\text{Cl}_2$  (5 x 20 mL). The combined organic extracts were dried over  $\text{Na}_2\text{SO}_4$  and concentrated to yield an oil. The crude oil was purified by vacuum distillation (65-70°C/150 mTorr) to yield **A** (6.91 g, 74% based on **B**) as a colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz):  $\delta$  0.96 (d, 6H, 6.2 Hz,  $\text{CH}_3\text{-CH-}$ ), 1.01 (broad s, 5H,  $\text{NH}_2 + \text{NH}$ ), 1.4 (m, 4H,  $-\text{CH}_2\text{-CH-}$ ), 2.66-2.81 (m, 6H,  $-\text{CH}_2\text{-NH}_2 + -\text{CH-NH-}$ ).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz):  $\delta$  20.62 ( $\text{CH}_3\text{-CH-}$ ), 39.38 ( $-\text{CH}_2\text{-CH}_2\text{-CH-}$ ), 41.75 ( $-\text{CH}_2\text{-NH}_2$ ), 47.93 ( $\text{CH}_3\text{-CH-}$ ).  $[\alpha]_{589\text{nm}}^{25^\circ\text{C}} = +27.67^\circ$  ( $c = 0.11$ ,  $\text{CHCl}_3$ ). HRMS (ESI) calculated for  $\text{C}_8\text{H}_{22}\text{N}_3$  (M+H): 160.1814, found 160.1816.

**Preparation of 2-(S),10-(S)-2,10-dimethyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (hpp'H).** A three-neck flask was charged with pure **A** (1.1 g, 6.9 mmol) and dissolved in 40 mL dry, degassed NO<sub>2</sub>Me under argon. Dimethyl trithiocarbonate (0.95 mL, 8.61 mmol) was added and the mixture was refluxed for three hours. The mixture was cooled to room temperature. Acetic acid (1.58 g, 27.6 mmol) and methyl iodide (0.86 mL, 13.8 mmol) was added and the mixture refluxed for three hours and then stirred at room temperature overnight. The solvent was evaporated under reduced pressure and the resulting oil suspended in 20 mL water and extracted into CH<sub>2</sub>Cl<sub>2</sub> (5 x 20 mL). The organic extracts were passed through a plug of silica gel and eluted with 25 mL CH<sub>2</sub>Cl<sub>2</sub> and then with 30 mL MeOH-CH<sub>2</sub>Cl<sub>2</sub> (1:4). The collected MeOH-CH<sub>2</sub>Cl<sub>2</sub> layers were treated with charcoal, dried with Na<sub>2</sub>SO<sub>4</sub>, and concentrated under reduced pressure to yield a yellow oil. The oil was dissolved in aqueous 4N NaOH and extracted with CH<sub>2</sub>Cl<sub>2</sub> (5 x 15 mL). The combined organic extracts were dried over Na<sub>2</sub>SO<sub>4</sub> and concentrated using a rotary evaporator to yield hpp'H (0.53 g, 26% based on **A**) as a yellow-white solid. The product was characterized as hpp'H.HI by single-crystal X-ray diffractometry. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz): δ 1.19 (d, 6H, 6.6 Hz, CH<sub>3</sub>-CH-), 1.75-1.91 (m, 4H, -CH<sub>2</sub>-CH<sub>2</sub>-CH-), 3.28-3.33 (m, 4H, -N-CH<sub>2</sub>-), 3.51-3.57 (m, 2H, -N-CH-), <sup>1</sup>H NMR (CDCl<sub>3</sub>, 600 MHz): δ 1.235 (d, 3H, 6 Hz, CH<sub>3</sub>-CH-), 1.83-1.86 (m, 2H, -CH<sub>2</sub>-CH<sub>2</sub>-CH-), 1.92-1.96 (m, 2H, -CH<sub>2</sub>-CH<sub>2</sub>-CH-), 3.33-3.38 (m, 4H, -CH<sub>2</sub>-CH<sub>2</sub>-CH-), 3.57-3.59 (m, 2H, -CH<sub>2</sub>-CH<sub>2</sub>-CH-). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz): δ 17.98 (CH<sub>3</sub>-CH-), 26.40 (-CH<sub>2</sub>-CH-), 34.56 (-N-CH<sub>2</sub>-CH<sub>2</sub>-), 48.65 (CH<sub>3</sub>-CH-), 150.03 (-C=N-). HRMS (ESI) calculated for C<sub>9</sub>H<sub>18</sub>N<sub>3</sub> (M+H) 168.1501; found 168.1502.

**X-Ray Diffractometry : hpp'H<sub>2</sub><sup>+</sup>I.** A colorless blade with 0.22 x 0.09 x 0.02 mm<sup>3</sup> dimensions was mounted *via* grease to the tip of a glass fiber (epoxied to a brass pin) and placed on the diffractometer with the long crystal dimension (the *b* unit cell dimension) approximately parallel to the diffractometer phi axis. Data were collected on a Nonius KappaCCD diffractometer (Mo K<sub>α</sub> radiation, graphite monochromator) at 190 K (cold N<sub>2</sub> gas stream) using standard CCD data collection techniques. Lorentz and polarization corrections were applied to the 10536 data. A correction for absorption using the multi-scan technique was applied (T<sub>max</sub> = 0.9485, T<sub>min</sub> = 0.5912). Equivalent data were averaged yielding 2806 unique data (R-int = 0.0424, 2806 with F > 2σ(F)). Based on

preliminary examination of the crystal, the space group  $P 2_12_12_1$  was assigned. The computer programs from the HKLint package were used for the data reduction. Structure refinement was performed with the SHELXTL v6.1 package.

## CHAPTER 5

HIGH- AND MID- VALENT TANTALUM AND  
MONO(PERALKYLCYCLOPENTADIENYL)TANTALUM COMPLEXES OF THE  
BICYCLIC GUANIDINATE HEXAHYDROPYRIMIDOPYRIMIDINATE

Mono(cyclopentadienyl) piano stool complexes of the early transition metals and lanthanides are of considerable interest in alkene polymerization, e.g., for constrained-geometry catalyst systems,<sup>86,87,88,89,90,91</sup> and small molecule reactivity because they are less sterically encumbered than the extensively-studied bent-sandwich bis(cyclopentadienyl) complexes. Steric and electronic factors of relevance to catalytic activity, selectivity, enantioselectivity, and stability can be modified by changes in cyclopentadienyl substituents and the other ancillary ligands in the coordination sphere in these mono(cyclopentadienyl) complexes.

The anion (often abbreviated hpp) of the bicyclic guanidine 1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidine, or 1,5,7-triazabicyclo[4.4.0]dec-5-ene, abbreviated hppH or TBD, exhibits diverse mononuclear and dinuclear transition metal, main group element, and lanthanide coordination chemistries. This area has been reviewed by Coles recently.<sup>53</sup> Since the first demonstration of dinuclear bridging hpp coordination<sup>16</sup> by Bear et al. in 1996, an extensive and interesting range of dinuclear metal-metal bonded hpp paddlewheel- and lantern-style complexes have been reported by Cotton and Murillo,<sup>27,92,93,94</sup> Chisholm,<sup>95</sup> Fackler,<sup>96</sup> and co-workers predominantly for the middle and late transition metals. Notable among these reports is the remarkable  $W_2(\text{hpp})_4$  with a highly-reducing ditungsten(II) quadruple bond.

Guanidinate ligands are more basic than the common carboxylate, amidinate, and formamidinate ligands because of a canonical form with donation of the non-coordinating nitrogen's lone pair to the resonance-stabilized NCN portion of the guanidinate. Amidinate and formamidinate ligands have led to a significant early transition metal chemistry relevant to alkene polymerization and small molecule activation (recently and notably dinitrogen activation and cleavage).<sup>97</sup> Amidinates and formamidinates have also been utilized as dinucleating ligands, though they are susceptible to cleavage by strongly reducing early transition metals in their low oxidation states.<sup>98</sup> The bicyclic guanidinate



hpp<sup>-</sup> has been shown to be more robust and resistant to such cleavage, leading to low-valent complexes such as V<sub>2</sub>(hpp)<sub>4</sub>,<sup>18</sup> Nb<sub>2</sub>(hpp)<sub>4</sub>,<sup>99</sup> and [Nb<sub>2</sub>(η<sup>2</sup>-hpp)<sub>4</sub>(μ-η<sup>2</sup>,η<sup>2</sup>-hpp)](PF<sub>6</sub>).<sup>100</sup> The latter compound as well as Ti<sub>2</sub>(η<sup>2</sup>-hpp)<sub>2</sub>(μ-η<sup>2</sup>,η<sup>2</sup>-hpp)<sub>2</sub>Cl<sub>2</sub> demonstrate that the hpp ligand can chelate as well as bridge in dinuclear chemistry.

We have been developing mid-valent early transition metal hpp chemistry of tantalum with the goal of accessing new organoditantalum complexes and studying their small molecule, for example dinitrogen,<sup>101</sup> activation chemistry. Tantalum hpp coordination chemistry is poorly developed. The tantalum analog of V<sub>2</sub>(hpp)<sub>4</sub> and Nb<sub>2</sub>(hpp)<sub>4</sub>, Ta<sub>2</sub>(hpp)<sub>4</sub>, is unknown and only a mononuclear [Ta<sup>V</sup>(hpp)<sub>4</sub>]<sup>+</sup> salt was obtained during synthetic attempts.<sup>50</sup> There are no tantalum analogs of the reported Nb(hpp)Cl<sub>4</sub> or Nb(hpp)<sub>2</sub>Cl<sub>3</sub>,<sup>102</sup> as similar synthetic approaches via TaCl<sub>5</sub> yielded only [Ta(hpp)<sub>4</sub>]<sup>+</sup> [TaCl<sub>6</sub>]<sup>-</sup>.<sup>103</sup> There are also no reported mid- or low-valent tantalum or mono(cyclopentadienyl)tantalum hpp complexes.

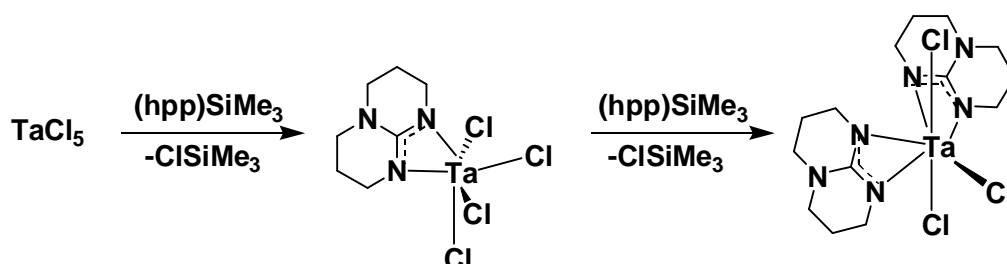
## Results and Discussion

**Synthesis and Spectroscopic Characterization of Ta(hpp)<sub>x</sub>Cl<sub>4-x</sub> (x = 1, 2).** The addition of one equivalent of distilled (i.e., not generated *in situ* from Li(hpp) and ClSiMe<sub>3</sub> in ethereal solvents) (hpp)SiMe<sub>3</sub> to TaCl<sub>5</sub> suspended in dichloromethane cleanly gives the mono(hpp) adduct Ta(hpp)Cl<sub>4</sub> in 70% yield via elimination of ClSiMe<sub>3</sub> (Scheme 20). Ta(hpp)Cl<sub>4</sub> is yellow as a recrystallized solid and air- and moisture-sensitive. It is somewhat soluble in aromatic solvents and very soluble in dichloromethane.

The <sup>1</sup>H NMR spectra in C<sub>6</sub>D<sub>6</sub> and in CD<sub>2</sub>Cl<sub>2</sub> each showed two triplets and an apparent quintet for three sets of methylene protons of the hpp ligand at room temperature. This suggests that either a mirror plane bisects the bicyclic guanidinate in the solution structure or that the hpp ligand is undergoing a dynamic process on the proton NMR time scale that averages the two halves of the hpp ligand.

The bis(hpp) compound Ta(hpp)<sub>2</sub>Cl<sub>3</sub> cannot be prepared directly from TaCl<sub>5</sub> and (hpp)SiMe<sub>3</sub> under our reaction conditions (room temperature, dichloromethane solvent);

instead, the reported compound  $[\text{Ta}(\text{hpp})_4]^+[\text{TaCl}_6]^-$  was obtained. This may be the result of the relative insolubility of  $\text{TaCl}_5$  in dichloromethane, such that  $\text{hpp}/\text{Cl}$  metathesis proceeds too rapidly compared to dissolution/reaction of  $\text{TaCl}_5$ . However,  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  can be prepared in nearly quantitative yield by addition of a second equivalent of  $(\text{hpp})\text{SiMe}_3$  to a dichloromethane solution of  $\text{Ta}(\text{hpp})\text{Cl}_4$  at  $25^\circ\text{C}$  (Scheme 20).



Scheme 20: Synthesis of  $\text{Ta}(\text{hpp})_x\text{Cl}_{4-x}$  ( $x = 1, 2$ ).

$\text{Ta}(\text{hpp})_2\text{Cl}_3$  exhibited a fast-exchange limit  $^1\text{H}$  NMR spectrum at room temperature in  $\text{CD}_2\text{Cl}_2$ , with two triplets and an apparent quintet at chemical shifts slightly upfield of those for  $\text{Ta}(\text{hpp})\text{Cl}_4$  in the same solvent. Given the solid-state pentagonal bipyramidal structure (*vide infra*) of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$ , there should be six sets of methylene resonances, so  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  is clearly fluxional in solution. Interestingly, the  $^1\text{H}$  NMR spectrum of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  in  $\text{C}_6\text{D}_6$  at room temperature shows a broad, poorly resolved doublet, one just past coalescence, for the most downfield methylene resonance. The middle resonance is a slightly broadened triplet, while the upfield quintet is instead a broad apparent singlet. Variable-temperature  $^1\text{H}$  NMR spectra in  $\text{CD}_2\text{Cl}_2$  are shown in stacked form in Figure 20. The spectra show clear trending toward a slow-exchange limit

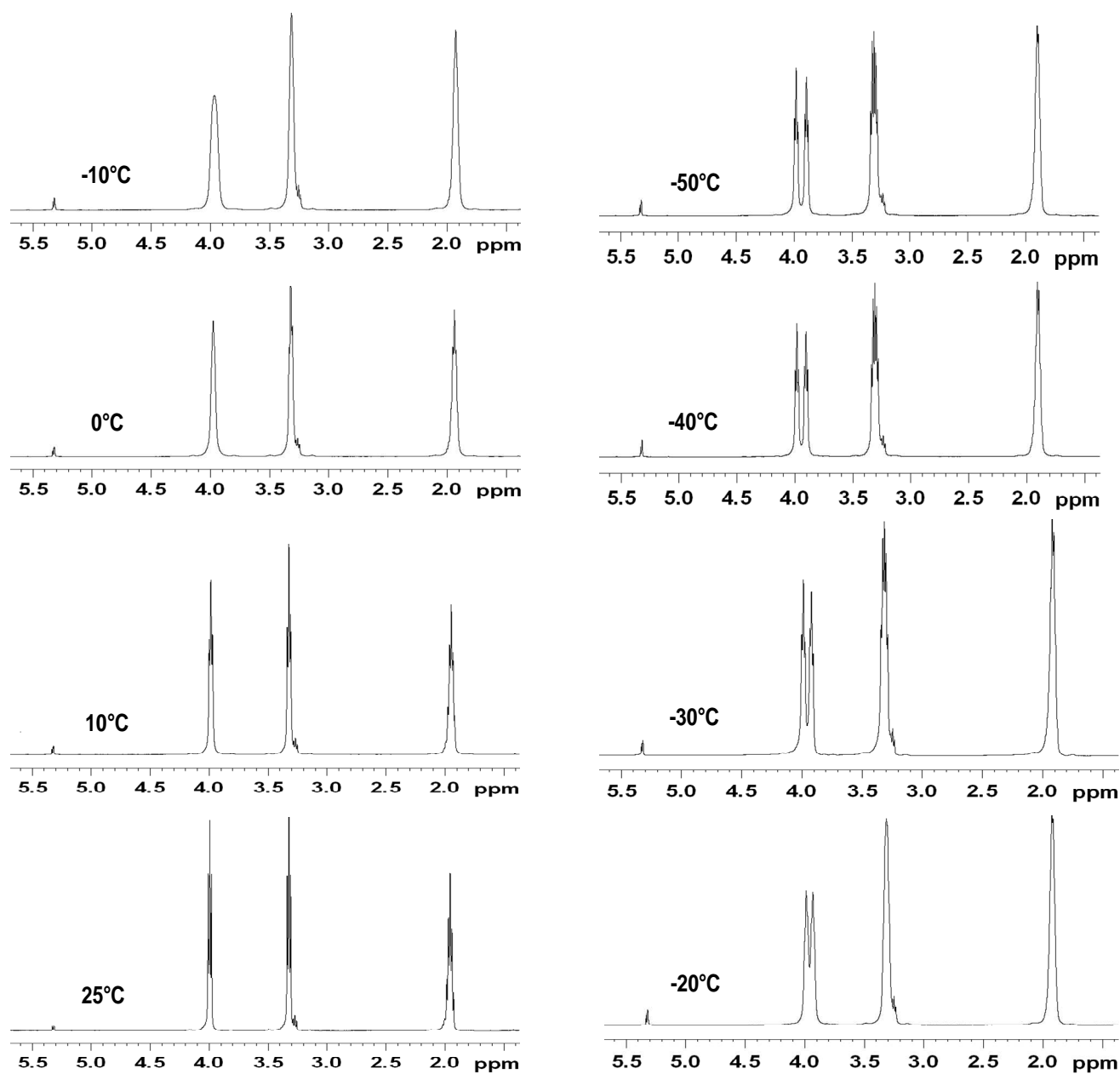


Figure 20: Variable-temperature  $^1\text{H}$  NMR spectra of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  (in  $\text{CD}_2\text{Cl}_2$ ) as a function of temperature.

spectrum at  $-60^\circ\text{C}$  with six different methylene resonances, consistent with the solid-state structure (*vide infra*).

### Solid-State Structures of Ta(hpp)<sub>x</sub>Cl<sub>4-x</sub> (x = 1, 2) via Single-Crystal X-ray

#### Diffractionometry.

Ta(hpp)Cl<sub>4</sub> has a distorted octahedral structure in the solid-state (Figure 21) in terms of directly-bonded atoms, with one independent molecule in the unit cell. Tables 5 and 6 compare selected bond distances, bond angles, and dihedral angles for all new tantalum hpp complexes that were characterized structurally. For Ta(hpp)Cl<sub>4</sub>, the Ta(1)–N(1) and Ta(1)–N(2) distances of 2.04(1) and 2.06(1) Å, respectively, are nominally the shortest of all the new tantalum hpp complexes reported here, and are the same statistically. The error limits are large for the poorer-quality data set for the Ta(hpp)Cl<sub>4</sub> crystal that was examined because the guanidinate carbon C(1) refined to a negative isotropic thermal parameter and a non-positive anisotropic thermal parameter; its isotropic thermal parameter was then set to that of N(1) and constrained during refinement. The Cl(3)–Ta(1)–N(1) and Cl(1)–Ta(1)–N(2) angles are 86.8(5)° and 90.5(5)°, respectively. Because of the acute chelate bite angle of the hpp ligand (N(1)–Ta–N(2), 62.5(7)°), Cl(1) and Cl(3) are bent up from the idealized octahedral positions to give an obtuse Cl(1)–Ta–Cl(3) angle of 119.4(1)°, or nearly 120°. Thus, an alternate description of the coordination geometry would be that of a trigonal bipyramid with Cl(1), Cl(3), and the non-tantalum-bonded bridgehead C(1) (Ta(1)···C(1), 2.56(2)Å) being the equatorial positions. The Cl(1)–Ta(1)···C(1) and Cl(3)–Ta(1)···C(1) angles are 121.1(4)° and 119.5(4)°, respectively, for a total combined with the 119.4(1)° value for Cl(1)–Ta(1)–Cl(3) of 360° within experimental error. Cl(2) and Cl(4) are bent away (Cl(2)–Ta–Cl(4) angle of 164.9(2)°) from their idealized octahedral positions (or, alternately, from the axial positions of a trigonal bipyramid) and from the hpp ligand, presumably from steric interactions with the closest hydrogens on C(2) and C(7). The Cl(2)···H(7B) distance, 4.25 Å, and Cl(4)···H(2B) distance, 4.23 Å, are approximately the sum (4.30Å) of the van der Waals radii for a hydrogen, 1.91 Å, and a chlorine, 2.39 Å, derived from the van der Waals constants for H<sub>2</sub> and Cl<sub>2</sub>.

Figure 22 shows a view perpendicular to the least squares plane defined by Ta and the guanidinate nitrogens and C(1). As is typical for hpp complexes, the two fused aliphatic six-membered rings are puckered. The Ta-guanidinate core is highly planar, with the dihedral angle between the Ta(1)/N(1)/N(2) plane and the N(1)/N(2)/C(1)/N(3) least squares plane being  $0.2^\circ$ .

The C(1)–N(3) distance of  $1.28(2)$  Å for the bridgehead nitrogen is statistically similar to that of C(1)–N(2),  $1.31(2)$  Å, and shorter than C(1)–N(1),  $1.39(2)$  Å. This suggests that there is a significant electronic contribution from delocalization of the bridgehead nitrogen N(3)'s lone pair to C(1) and thus to N(1) and N(2), increasing the hpp ligand basicity.

Table 5: Bond lengths (Å) for new tantalum hpp complexes. Cp\* = C<sub>5</sub>Me<sub>5</sub>; Cp'' = C<sub>5</sub>Me<sub>4</sub>Et

	Ta(hpp)Cl <sub>4</sub>	Ta(hpp) <sub>2</sub> Cl <sub>3</sub>	Cp*Ta(hpp)Cl <sub>3</sub> (molecule A)	Cp*Ta(hpp)Cl <sub>3</sub> (molecule B)	Cp*Ta(hpp)Cl <sub>2</sub> (molecule A)	Cp*Ta(hpp)Cl <sub>2</sub> (molecule B)	Cp''Ta(hpp)Cl <sub>2</sub>
<b>Ta–N</b>	2.04(1) N1 2.06(1) N2	2.115(5) N1 2.109(5) N2 2.114(5) N4 2.119(5) N5	2.153(5) N1 2.075(5) N2	2.151(5) N21 2.082(5) N22	2.12(3) N1 2.14(2) N2	2.11(3) N21 2.06(3) N22	2.128(8) N1 2.089(7) N2
<b>Ta··C1</b>	2.56(2) C1	2.566(6) C1 2.598(6) C8	2.574(6) C1	2.584(6) C21	2.54(2) C1	2.64(3) C21	2.569(9) C1
<b>Ta–Cl</b>	2.341(5) Cl1 2.368(6) Cl2 2.365(5) Cl3 2.340(4) Cl4	2.463(2) Cl1 2.395(2) Cl2 2.407(2) Cl3	2.426(2) Cl1 2.463(2) Cl2 2.443(2) Cl3	2.424(2) Cl4 2.459(2) Cl5 2.484(2) Cl6	2.424(9) Cl1 2.407(9) Cl2	2.43(1) Cl3 2.421(9) Cl4	2.413(2) Cl1 2.414(9) Cl2
<b>Ta··Cp centroid</b>	N/A	N/A	2.14	2.21	2.066	2.06	2.05
<b>C1–N</b>	1.39(2) N1 1.31(2) N2	1.345(8) N1 1.347(8) N2	1.305(8) N1 1.372(8) N2	1.319(8) N21 1.367(8) N22	1.36(3) N1 1.23(4) N2	1.49(4) N21 1.37(4) N22	1.33(1) N1 1.37(1) N2
<b>C1–N(bridge)</b>	1.28(2) N3	1.333(8) N3	1.339(8) N3	1.329(8) N23	1.41(4) N3	1.25(4) N23	1.33(1) N3
<b>C8–N</b>	N/A	1.348(8) N4 1.357(9) N5	N/A	N/A	N/A	N/A	N/A
<b>C8–N(bridge)</b>	N/A	1.315(8) N6	N/A	N/A	N/A	N/A	N/A

Table 6: Bond and non-bond angles ( $^{\circ}$ ) for new tantalum hpp complexes. Cp\* = C<sub>5</sub>Me<sub>5</sub>; Cp'' = C<sub>5</sub>Me<sub>4</sub>Et

	Ta(hpp)Cl <sub>4</sub>	Ta(hpp) <sub>2</sub> Cl <sub>3</sub>	Cp*Ta(hpp)Cl <sub>3</sub> (molecule A)	Cp*Ta(hpp)Cl <sub>3</sub> (molecule B)	Cp*Ta(hpp)Cl <sub>2</sub> (molecule A)	Cp*Ta(hpp)Cl <sub>2</sub> (molecule B)	Cp''Ta(hpp)Cl <sub>2</sub>
<b>Cl1–Ta–Cl</b>	85.9(2) Cl2 119.4(1) Cl3 86.0(2) Cl4	88.66(6) Cl2 89.13(7) Cl3	84.15(7) Cl2 85.05(7) Cl3	85.50(6) Cl5 86.30(6) Cl6	87.2(4) Cl2	86.6(3) Cl2	86.42(9) Cl2
<b>Cl2–Ta–Cl</b>	86.3(2) Cl3 164.9(2) Cl4	177.77(6) Cl3	154.97(7) Cl3	156.26(6) Cl6	N/A	N/A	N/A
<b>Cl3–Ta–Cl</b>	86.0(2) Cl4		N/A	N/A	N/A	N/A	N/A
<b>Cp centroid–Ta–Cl</b>	N/A	N/A	102.0 Cl1 102.7 Cl2 106.6 Cl3	102.4 Cl4 101.2 Cl5 105.5 Cl5	115.1 Cl1 111.2 Cl2	115.2 Cl3 111.4 Cl4	114.4 Cl1 112.3 Cl2
<b>Cp centroid–Ta–N</b>	N/A	N/A	108.4 N1 170.7 N2	109.9 N21 172.2 N22	116.7 N1 116.7 N2	114.0 N21 109.9 N22	112.4 N1 116.4 N2
<b>N–Ta–N</b>	62.5(7)	62.2(2) N1-N2 62.3(2) N4-N5	62.4(2)	62.3(2)	61.0(9)	61.0(9)	62.9(3)
<b>N–C1–N</b>	106(2)	108.4(5)	109.8(5)	109.2(5)	113(2)	102(3)	109.3(8)
<b>N–C8–N</b>		108.1(5)					
<b>N(bridge)–C1–N</b>	127(2) N1 127(2) N2	126.0(6) N1 125.4(6) N2	125.0(6) N1 125.2(6) N2	126.3(6) N21 124.5(6) N22	119(2) N1 128(3) N2	124(3) N21 135(3) N22	127.3(9) N1 123.4(9) N2
<b>N(bridge)–C8–N</b>	N/A	126.2(6) N4 125.6(6) N5	N/A	N/A	N/A	N/A	N/A
<b>Dihedral angle between N1-Ta-N2 and N1-N2-C1-N3</b>	0.2	21.1 hpp(1) 10.7 hpp(2)	8.3	9.7	10.1	20.3	11.6

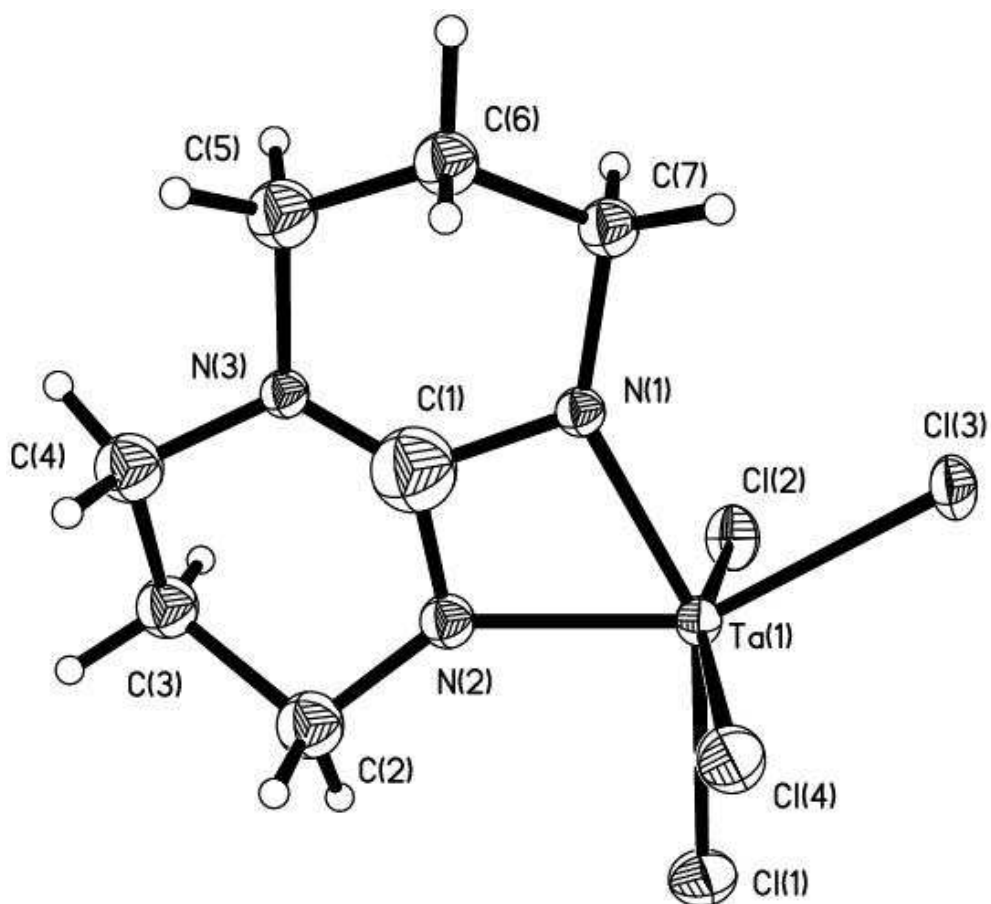


Figure 21: View of solid-state molecular structure of Ta(hpp)Cl<sub>4</sub> approximately perpendicular to the Ta-guanidinate plane. Thermal ellipsoids are shown at the 50% probability level.



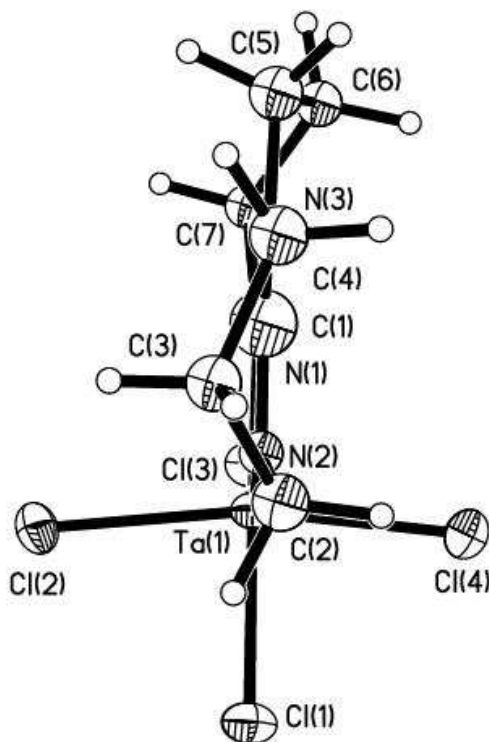


Figure 22: View of solid-state structure of Ta(hpp)Cl<sub>4</sub> approximately parallel to the plane containing Ta(1) and the guanidinate N<sub>2</sub>CN core of the hpp ligand, showing its planarity. Thermal ellipsoids are shown at the 50% probability level.

Ta(hpp)<sub>2</sub>Cl<sub>3</sub> crystallizes with one independent molecule in the unit cell. The solid-state seven-coordinate structure of Ta(hpp)<sub>2</sub>Cl<sub>3</sub> can be best described as a pentagonal bipyramid with the hpp ligands coordinated in the pentagonal plane along with one equatorial chlorine atom, Cl(1), and two axial chlorine atoms, Cl(2) and Cl(3) (Figure 23). The Ta–N distances are longer than those in Ta(hpp)Cl<sub>4</sub>, as might be expected for either the lowered Lewis acidity of a Ta(V) with four basic nitrogen donor atoms and/or greater steric crowding in a seven-coordinate structure.

The guanidinate moieties of the hpp ligands in Ta(hpp)<sub>2</sub>Cl<sub>3</sub> are less planar than in the case of Ta(hpp)Cl<sub>4</sub>. Figures 24 and 25 show two views of Ta(hpp)<sub>2</sub>Cl<sub>3</sub> that differ

in which of the Ta–guanidinate moieties are oriented perpendicular to the page. The dihedral angles between the Ta/N/N planes and the least-squares  $N_2CN$  planes for both hpp ligands are both larger than that in  $Ta(hpp)Cl_4$  ( $0.2^\circ$ ) and differ substantially from each other, with a dihedral angle between the Ta(1)/N(2)/N(3) plane and the least-squares plane N(1)/N(2)/C(1)/N(3) of  $21.1^\circ$  and a dihedral angle between the Ta(1)/N(4)/N(5) plane and the least-squares plane N(4)/N(5)/C(8)/N(6) of  $10.7^\circ$ . We ascribe the difference in dihedral angles of the two hpp ligands in  $Ta(hpp)_2Cl_3$  to steric crowding between the hpp ligands in the pentagonal plane.

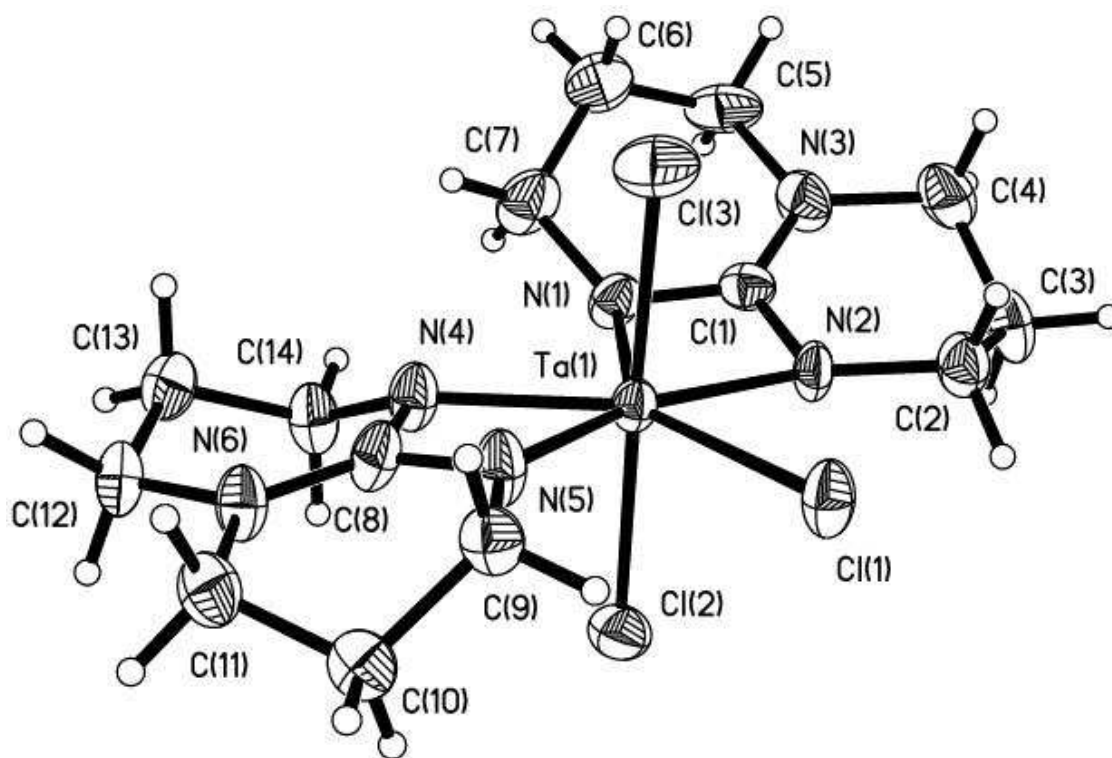


Figure 23: View of solid-state structure of seven-coordinate  $Ta(hpp)_2Cl_3$ . Thermal ellipsoids are shown at the 50% probability level.

The bridgehead nitrogen-to-bridgehead carbon distances of 1.333(8) Å (C(1)–N(3)) and 1.315(8) Å (C(8)–N(6)) for the two hpp ligands are shorter than the Ta-bonded nitrogen-to-bridgehead carbon distances (C(1)–N(1), 1.345(8) Å; C(1)–N(2), 1.347(8) Å; C(8)–N(4), 1.348(8) Å; C(8)–N(5), 1.357(9) Å). This is consistent, as was the case with the less-accurate Ta(hpp)Cl<sub>4</sub> solid-state structure, with significant bridgehead nitrogen lone pair donation to the bridgehead carbons and thus to the tantalum-bonded nitrogens.

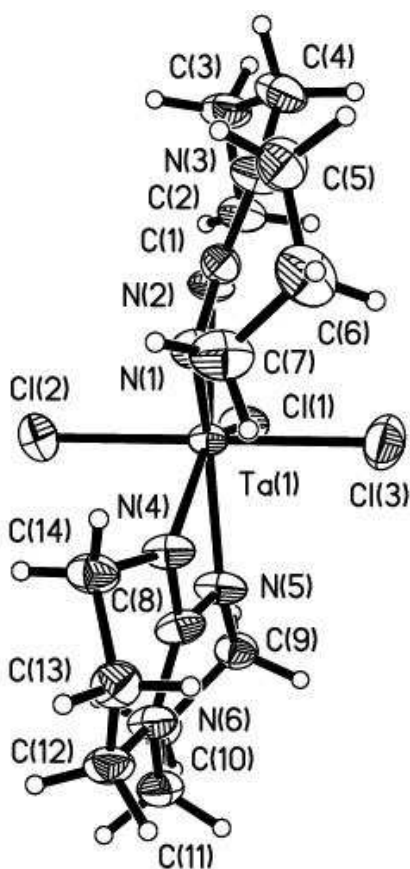


Figure 24: View of solid-state structure of Ta(hpp)<sub>2</sub>Cl<sub>3</sub> approximately parallel to the plane containing Ta(1) and the guanidinate N<sub>2</sub>CN core of the first hpp ligand. Thermal ellipsoids are shown at the 50% probability level.

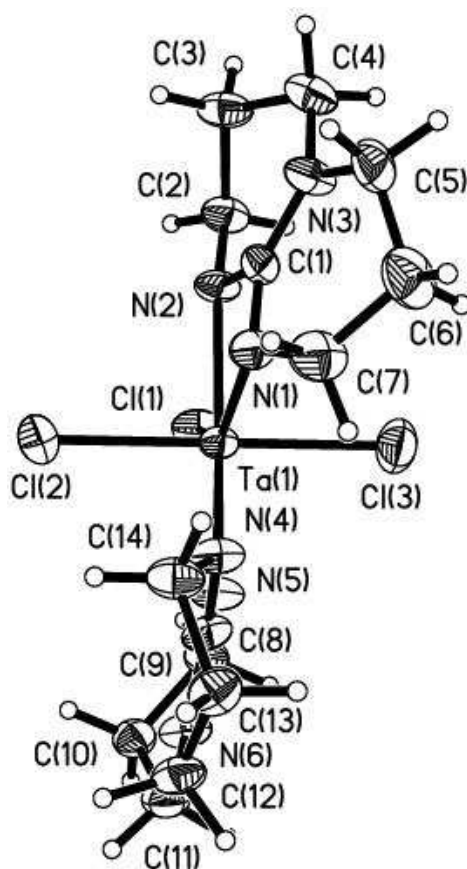
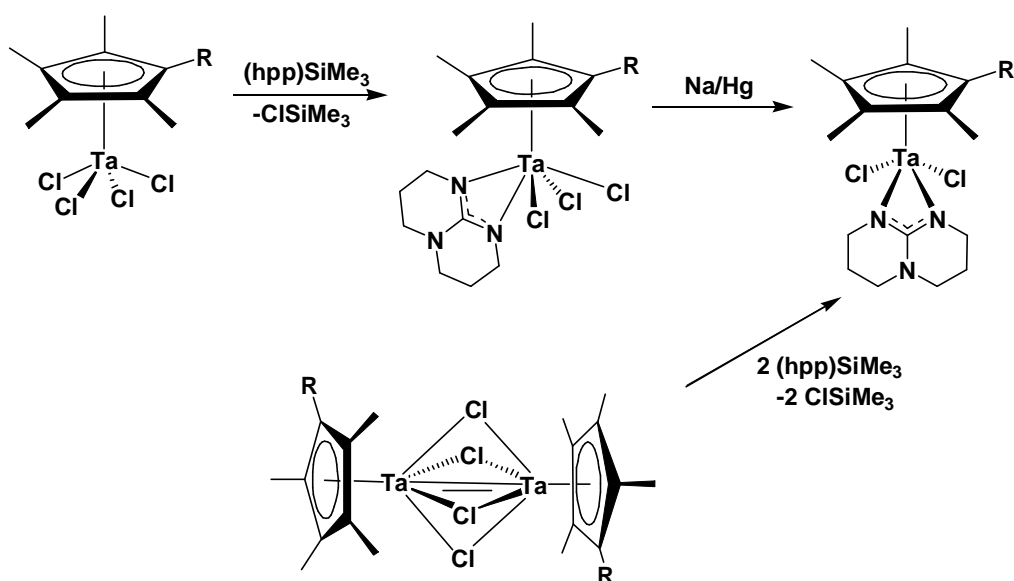


Figure 25: View of solid-state structure of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  approximately parallel to the plane containing Ta(1) and the guanidinate  $\text{N}_2\text{CN}$  core of the second hpp ligand; the view is a slight rotation of that shown in Figure 22. Thermal ellipsoids are shown at the 50% probability level.

### Synthesis and Spectroscopic Characterization of $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_3$ (R = Me, Et).

Addition of  $(\text{hpp})\text{SiMe}_3$  (one equivalent) to a dichloromethane solution of  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{TaCl}_4$  yields the mono(hpp) adduct  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_3$  straightforwardly in high yields (86%, R = Me; 95%, R = Et) (Scheme 21).  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_3$  is a red solid that yields bright pinkish-red solutions in dichloromethane. For R = Me the

compound has unusual solubility characteristics (e.g., soluble in benzene, insoluble in toluene) that suggest ionic character, such as  $[(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_2]^+\text{Cl}^-$ , in the more polar toluene, or disparate solvent coordination with the molecule. Future solution conductivity studies should be able to address these unusual solubility characteristics.



Scheme 21: Synthesis of  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_x$  (R = Me, Et; x = 3, 2).

The  $^1\text{H}$  NMR spectra of  $(\text{C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_3$  in  $\text{C}_6\text{D}_6$  at room temperature are consistent with a static structure with the two nitrogens in very different chemical environments, given the wide range of chemical shifts for the six inequivalent methylene proton resonances. The molecule has a plane of symmetry through the cyclopentadienyl centroid for the  $\text{C}_5\text{Me}_4\text{Et}$  compound, with only two ring methyl singlets (vs. four) and a simple quartet (rather than an AB quartet) for the methylene resonance of the ring Et group. The spectra are therefore consistent with a hpp coordination mode with one nitrogen in a pseudo-equatorial position of a four-legged piano-stool structure and the

other nitrogen coordinated in an axial position *trans* to the centroid of the cyclopentadienyl group. This solution structure is consistent with the solid-state structure (*vide infra*).

**Syntheses and Spectroscopic Characterization of the Ta(IV) hpp Complexes ( $\eta$ -C<sub>5</sub>Me<sub>4</sub>R)Ta(hpp)Cl<sub>2</sub> (R = Me, Et).**

Given the lack of mid- or low-valent hpp complexes of tantalum, we examined reactions of the mid-valent organoditantalum(III) complex ( $\eta$ -C<sub>5</sub>Me<sub>4</sub>Et)<sub>2</sub>Ta<sub>2</sub>( $\mu$ -Cl)<sub>4</sub> with (hpp)SiMe<sub>3</sub> as well as direct reduction of ( $\eta$ -C<sub>5</sub>Me<sub>4</sub>R)Ta(hpp)Cl<sub>3</sub> with sodium amalgam (Scheme 21). Addition of two equivalents of (hpp)SiMe<sub>3</sub> to ( $\eta$ -C<sub>5</sub>Me<sub>4</sub>Et)<sub>2</sub>Ta<sub>2</sub>( $\mu$ -Cl)<sub>4</sub> in toluene under dinitrogen leads to disproportionation and crystallization of the Ta(IV) complex ( $\eta$ -C<sub>5</sub>Me<sub>4</sub>R)Ta(hpp)Cl<sub>2</sub>. Other mid-valent organotantalum complexes (e.g., Ta(II), or higher oxidation state dinitrogen adducts), as would be expected from a disproportionation of the organoditantalum(III) reactant, have not yet been isolated.

**Solid-State Structures of ( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)Ta(hpp)Cl<sub>3</sub> and the Ta(IV) hpp adducts ( $\eta$ -C<sub>5</sub>Me<sub>5</sub>R)Ta(hpp)Cl<sub>2</sub> and ( $\eta$ -C<sub>5</sub>Me<sub>4</sub>Et)Ta(hpp)Cl<sub>2</sub> via Single-Crystal X-ray Diffractometry.**

The Ta(V) hpp adduct ( $\eta$ -C<sub>5</sub>Me<sub>5</sub>)Ta(hpp)Cl<sub>3</sub> crystallizes from dichloromethane with two independent molecules in the unit cell, termed A and B, in which the hpp is coordinated with one nitrogen *trans* axial to the C<sub>5</sub>Me<sub>5</sub> centroid and the other coordinated in the equatorial plane along with the three chlorines. The metrics are similar in molecules A and B (Tables 5 and 6), so only the values in molecule A are discussed here.

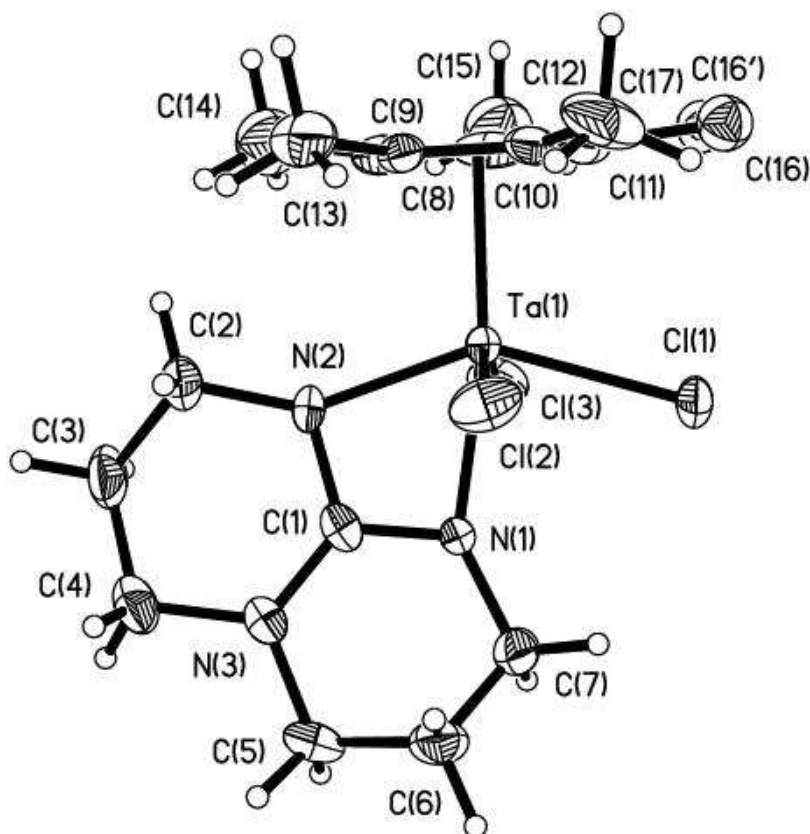


Figure 26: View of solid-state structure of  $(C_5Me_5)Ta(hpp)Cl_3$  (independent molecule A) approximately perpendicular to the hpp ligand plane. Thermal ellipsoids are shown at the 50% probability level.

Figure 26 shows one view of molecule A of  $(\eta-C_5Me_5)Ta(hpp)Cl_3$ . The Ta(1)–N(1) and Ta(1)–N(2) distances are 2.153(5) and 2.075(5) Å, respectively, so the axial nitrogen N(1) is further away. This asymmetry in the hpp coordination suggests that the N–C–N delocalization is somewhat diminished, so that the axial nitrogen interaction is more dative, via a localized imide nitrogen, and the pseudo-equatorial nitrogen is covalently bonded as an amido group. This would suggest that the N(1)–C(1) distance, 1.305(8) Å, should be shorter than the N(2)–C(1) distance, 1.372(8) Å, as is the case. The short C(1)–N(3) distance of 1.339(8) Å is consistent with significant lone pair electron donation from the bridgehead nitrogen N(3) to the guanidinate core. The Ta-guanidinate

core is also quite flat (though less so than in the case of  $\text{Ta}(\text{hpp})\text{Cl}_4$ ), with a dihedral angle between the  $\text{Ta}(1)/\text{N}(1)/\text{N}(2)$  plane and the  $\text{N}(1)/\text{C}(1)/\text{N}(2)/\text{N}(3)$  least-squares plane of  $8.3^\circ$ . The pseudo-equatorial  $\text{Ta}(1)$ –chlorine distances are  $2.426(2)$  Å for  $\text{Cl}(1)$  (trans to  $\text{N}(2)$ ),  $2.463(2)$  for  $\text{Cl}(2)$ , and  $2.443(2)$  for  $\text{Cl}(3)$ .

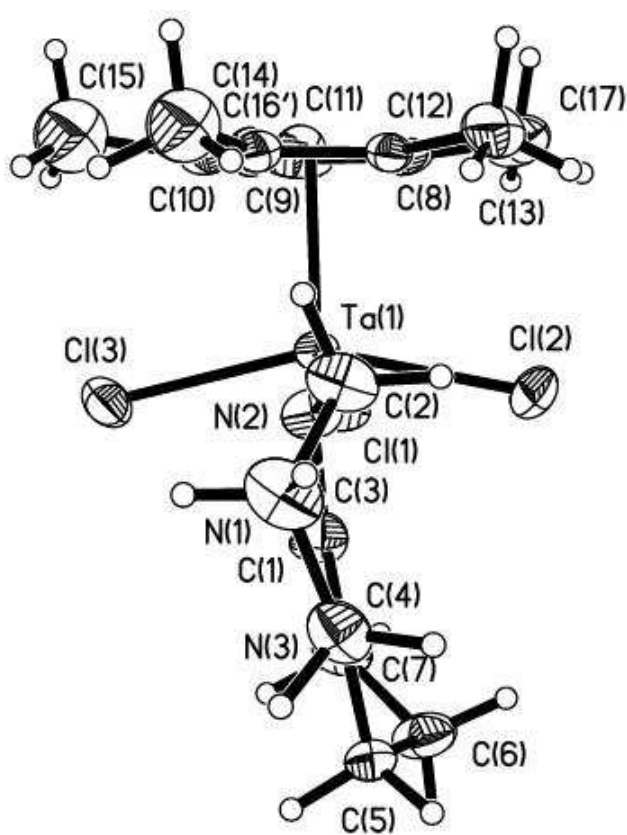


Figure 27: View of solid-state structure of  $(\text{C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_3$  (independent molecule A) approximately parallel to the plane containing  $\text{Ta}(1)$  and the guanidinate  $\text{N}_2\text{CN}$  core of the hpp ligand. Thermal ellipsoids are shown at the 50% probability level.



The Ta(IV) hpp complexes  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_2$  ( $\text{R} = \text{Me}, \text{Et}$ ) have solid-state four-legged piano-stool structures with the two chlorines and two guanidinate nitrogens of an hpp chelate in the equatorial plane.  $(\eta\text{-C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_2$  crystallizes with two independent molecules in the unit cell that, while isostructural, have some differences in bond distances and bond angles because of twinning, as shown in Table 5.

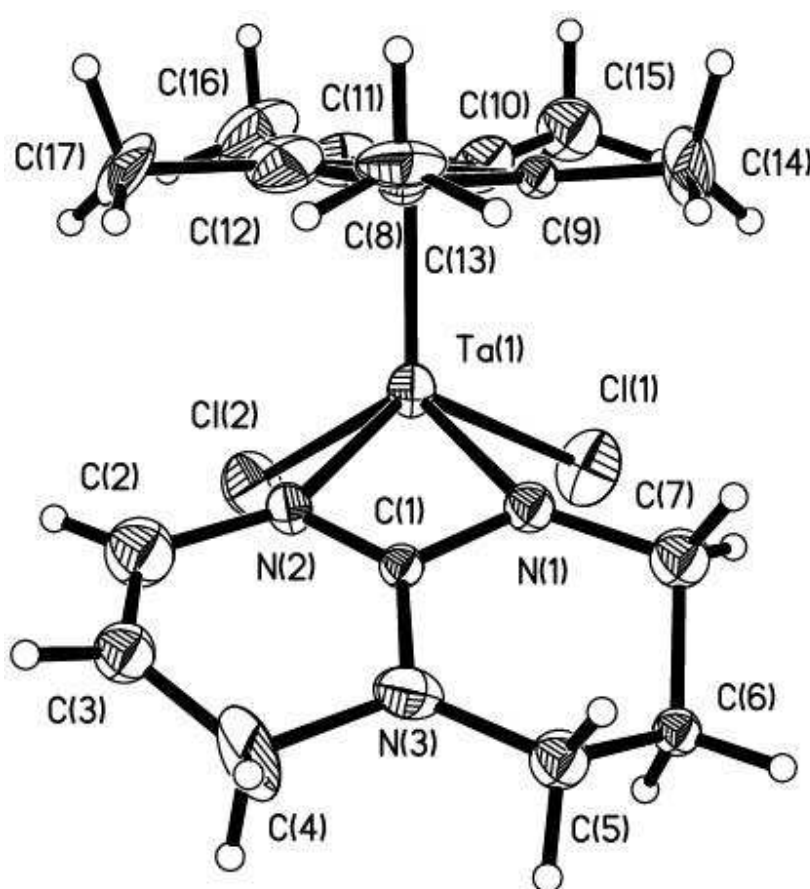


Figure 28: View of solid-state structure of  $(\text{C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_2$  (independent molecule A) approximately perpendicular to the Ta(1)– $\text{C}_5\text{Me}_5$  centroid axis. The hpp ligand is tilted above and away from the plane of the page. Thermal ellipsoids are shown at the 50% probability level.

Figures 28 and 29 show two views of the solid-state structure for molecule A of  $(\eta\text{-C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_2$ .  $(\eta\text{-C}_5\text{Me}_4\text{Et})\text{Ta}(\text{hpp})\text{Cl}_2$  crystallizes with one independent molecule in the unit cell, and the data set was more accurate and not twinned, so it will be discussed here since it is isostructural (Figures 30 and 31) to that of the  $\text{C}_5\text{Me}_5$  derivative.

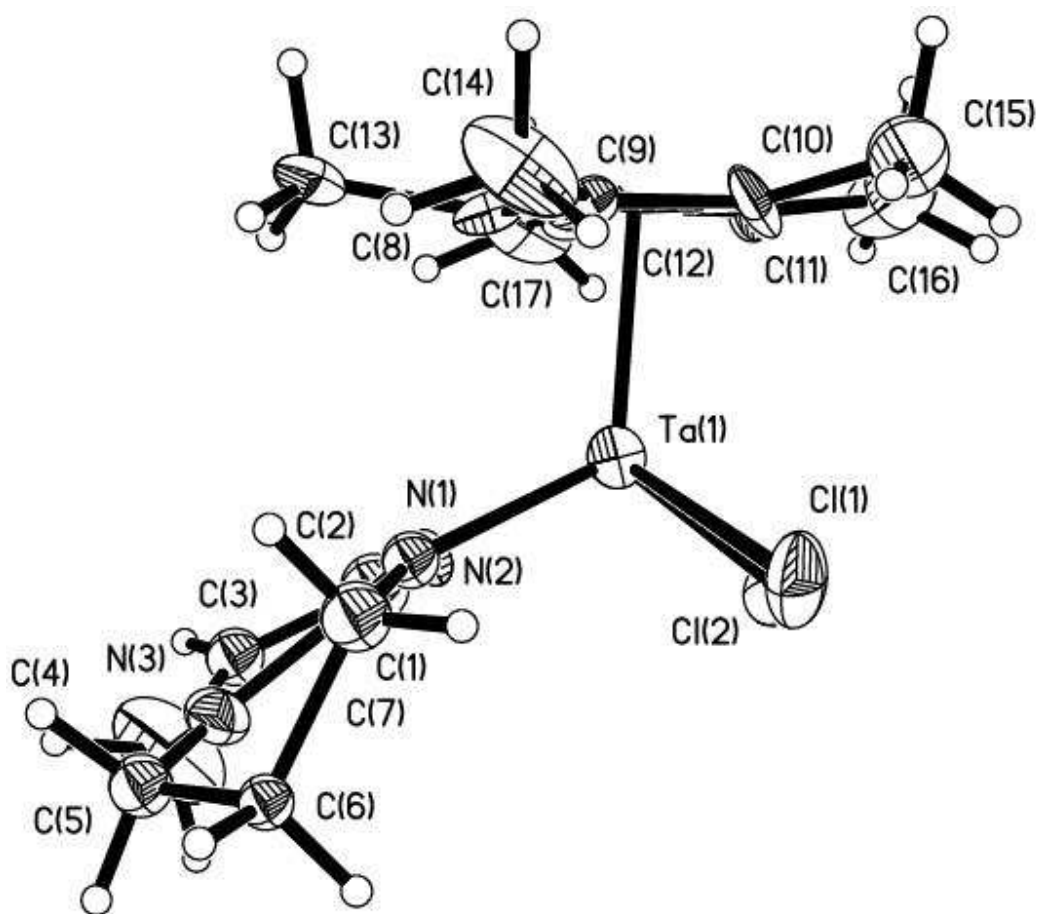


Figure 29: View of solid-state structure of  $(\text{C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_2$  (independent molecule A) approximately parallel to the plane containing Ta(1) and the guanidinate  $\text{N}_2\text{CN}$  core of the hpp ligand. Thermal ellipsoids are shown at the 50% probability level.

The Ta(1)–N(1) and Ta(1)–N(2) distances, 2.128(8) and 2.089(7) Å, are on average shorter than the average in the Ta(V) adduct  $(\eta\text{-C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_3$ . Since a longer distance would be expected for a Ta<sup>IV</sup>–N bond given the greater covalent radius of Ta(IV) over Ta(V), the observed shorter distance for the Ta(IV) compound must be the result of either an electronic effect on equatorial coordination of nitrogen or a greater steric crowding in the Ta(V) adduct.

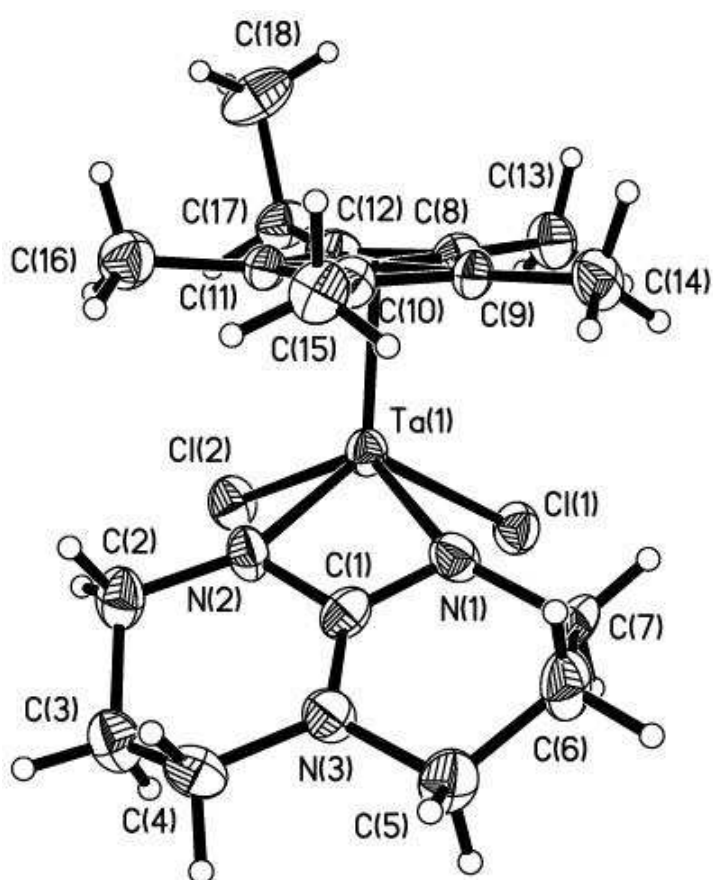


Figure 30: View of solid-state structure of  $(\text{C}_5\text{Me}_4\text{Et})\text{Ta}(\text{hpp})\text{Cl}_2$  approximately perpendicular to the Ta(1)–C<sub>5</sub>Me<sub>5</sub> centroid axis. The hpp ligand is tilted above and away from the plane of the page. Thermal ellipsoids are shown at the 50% probability level.

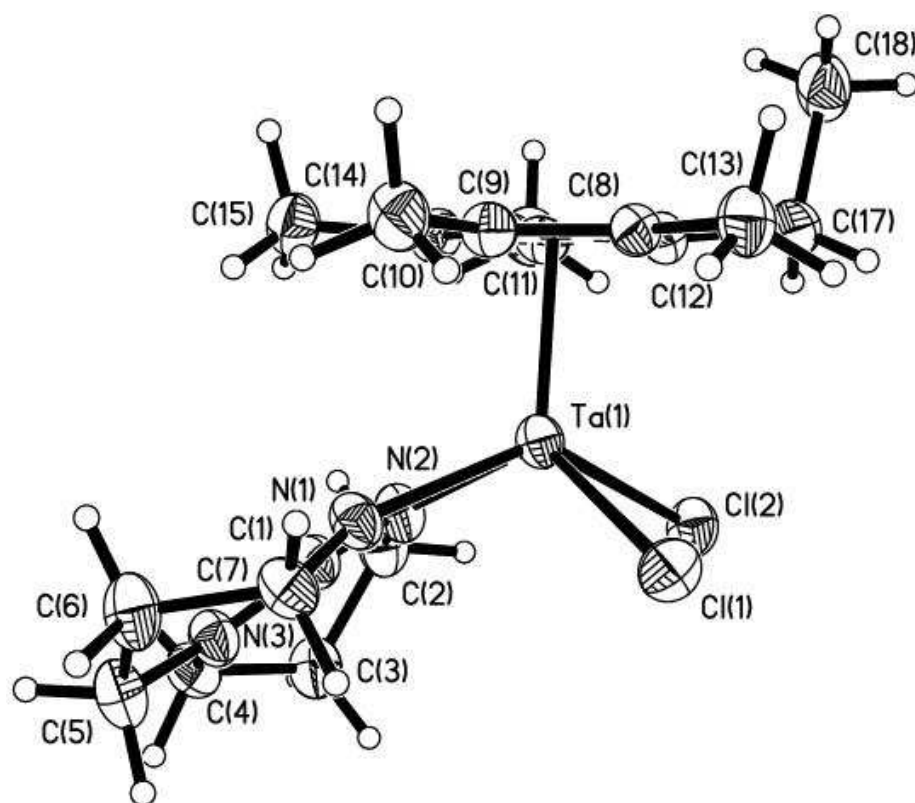


Figure 31: View of solid-state structure of  $(C_5Me_4Et)Ta(hpp)Cl_2$  approximately parallel to the plane containing Ta(1) and the guanidinate  $N_2CN$  core of the hpp ligand. Thermal ellipsoids are shown at the 50% probability level.

The Ta(1)–chlorine distances in the Ta(IV) adduct are statistically shorter than those in the Ta(V) adduct, as would be expected from the shorter Ta(IV) covalent radius. The four-legged piano stool structure has cyclopentadienyl centroid···Ta(1)–chlorine angles of  $114.4^\circ$  for Cl(1) and  $112.3^\circ$  for Cl(2), similar to the centroid···Ta(1)–N angles of  $112.4^\circ$  for N(1) and  $116.4^\circ$  for N(2).

There is a small but statistically insignificant asymmetry in the hpp coordination in  $(\eta\text{-C}_5\text{Me}_4\text{Et})\text{Ta}(\text{hpp})\text{Cl}_2$  as shown by both the Ta(1)–N distances and the C(1)–N(1) and C(1)–N(2) distances (1.33(1) and 1.37(1) Å, respectively).

The short C(1)–N(3) distance for the bridgehead atoms, 1.33(1) Å, is again consistent with significant bridgehead nitrogen lone pair donation into the guanidinate core. The dihedral angle between the Ta(1)/N(1)/N(2) plane and the least-squares N(1)/C(1)/N(2)/N(3) plane is  $11.6^\circ$ , also consistent with strong interaction of a basic guanidinate ligand with the Ta(IV) center.

### Conclusions

The hpp silane  $(\text{hpp})\text{SiMe}_3$  is an effective hpp transfer reagent in tantalum and mono(peralkylcyclopentadienyl)tantalum chemistries, particularly as a pure distilled reagent rather than one generated *in situ* from Li(hpp) and ClSiMe<sub>3</sub> in ethereal solvents. The new complex  $\text{Ta}(\text{hpp})\text{Cl}_4$  can be prepared by  $(\text{hpp})\text{SiMe}_3$  addition in dichloromethane to  $\text{TaCl}_5$ . It has a distorted octahedral six-coordinate structure with a strong Ta–hpp interaction and significant bridgehead nitrogen lone pair donation to the guanidinate core.  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  is obtained by addition of a second equivalent of  $(\text{hpp})\text{SiMe}_3$  to previously-isolated  $\text{Ta}(\text{hpp})\text{Cl}_4$ . The pentagonal bipyramidal structure with two equatorial hpp ligands is highly fluxional in solution by proton NMR spectroscopy. Mono(peralkylcyclopentadienyl) tantalum(V) hpp complexes  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_3$  are prepared in high yield by addition of one equivalent of  $(\text{hpp})\text{SiMe}_3$  to  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{TaCl}_4$ . These bright pinkish-red compounds have axial-ligated, four-legged piano-stool structures with an hpp ligand chelated to the axial and equatorial positions. The Ta(IV) hpp complexes  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{Ta}(\text{hpp})\text{Cl}_2$ , the first mid-valent tantalum hpp complexes, can be obtained from either reduction of  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{TaCl}_5$  or  $(\eta\text{-C}_5\text{Me}_4\text{R})\text{TaCl}_4$ .

$C_5Me_4R)Ta(hpp)Cl_3$  or addition of  $(hpp)SiMe_3$  to  $(\eta-C_5Me_4R)_2Ta_2(\mu-Cl)_4$ . The Ta(IV) complexes have a four-legged piano-stool structure with a chelating hpp ligand. The solid-state structures of all new tantalum hpp complexes have metrics consistent with coordination of highly basic hpp ligands in which the bridgehead nitrogens donate their lone pairs to the guanidinate cores.

### Experimental

**General Procedures.** Compounds were manipulated under dinitrogen in a Vacuum Atmospheres glove box with  $-40^\circ C$  refrigerator, rotary evaporator, and internal vacuum system for filtration and compound drying.  $TaCl_5$  stored in glass ampules was purchased from Cerac or Alfa Aesar and used as received. Chlorotrimethylsilane (Aldrich) was distilled prior to use.  $(\eta-C_5Me_4R)TaCl_4$  ( $R = Me, Et$ ) were prepared from room-temperature reaction of  $(C_5Me_4R)SnBu_3$  and  $TaCl_5$  in  $CH_2Cl_2$  and purified by fractional recrystallization from  $CH_2Cl_2$ , as described elsewhere along with additional procedures and syntheses of the precursors  $MeBrC=CHMe$ ,  $C_5Me_4RH$ ,  $Li(C_5Me_4R)$ , and  $(C_5Me_4R)SnBu_3$ .<sup>104</sup> Na/Hg (0.413% by weight) was prepared in the glove box by slow addition of clean Na pieces in one Erlenmeyer flask to Hg in a second flask, connected to the first via a flexible gooseneck adapter, in a closed system to avoid contamination of the glove box atmosphere with warm Hg vapor; the resulting amalgam was allowed to cool before transfer to a storage bottle.  $(C_5Me_5)_2Ta_2(\mu-Cl)_4$  was prepared<sup>105</sup> by Na/Hg reduction of  $(C_5Me_5)TaCl_4$  with two equivalents of Na/Hg in toluene and purified by recrystallization from toluene/hexanes. HppH (Aldrich) was doubly sublimed before use. The silicon reagent  $(hpp)SiMe_3$  was prepared by a literature procedure<sup>106</sup> from hppH, BuLi, and  $Me_3SiCl$ , purified by distillation, and stored under dinitrogen because of its

water sensitivity. Anhydrous dichloromethane, toluene, and hexanes were purchased from Aldrich and used as received.

$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra were obtained on a Bruker Avance III 300 MHz NMR spectrometer. Variable temperature NMR spectra were recorded on a Bruker DRX 400 MHz spectrometer.

**Preparation of (hpp)SiMe<sub>3</sub>.** In a modification of the published procedure,<sup>106</sup> 1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidine (hppH; 4.7 g, 33.8 mmol) was dissolved in 30 mL dry benzene under argon and cooled in an ice-bath for 10 minutes. A solution of 2.5 M BuLi (21.1 mL, 33.8 mmol) was slowly added to the reaction mixture, with constant stirring. The reaction mixture was stirred at room temperature for 12 hours. Chlorotrimethylsilane (5.14 mL, 40.5 mmol) was slowly added to the ice-bath cooled reaction mixture, leading to the formation of a white precipitate. The mixture was heated at 40°C for 12 hours. After filtration, the filtrate was concentrated under vacuum to yield an oil. The oil was distilled *in vacuo* (96-100°C/10 mm Hg) to yield pure (hpp)SiMe<sub>3</sub> (5.1 g, 71% based on hppH) as a colorless oil. The product is air- and moisture-sensitive and was stored in a glove box.  $^1\text{H}$  NMR ( $\delta$ , CDCl<sub>3</sub>, 300 MHz): 0.17 (s, 9H, (CH<sub>3</sub>)<sub>3</sub>), 1.73 (quintet, 4H, 5.89 Hz, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 2.98 (t, 4H, 6.16 Hz, NCH<sub>2</sub>CH<sub>2</sub>), 3.10 (t, 4H, 5.65 Hz, NCH<sub>2</sub>CH<sub>2</sub>).  $^{13}\text{C}$  NMR ( $\delta$ , CDCl<sub>3</sub>, 75 MHz): 1.35 (CH<sub>3</sub>)<sub>3</sub>, 23.87 (CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 42.11 (NCH<sub>2</sub>CH<sub>2</sub>), 48.21 (NCH<sub>2</sub>CH<sub>2</sub>), 151.71 (NCN).

**Preparation of Ta(hpp)Cl<sub>4</sub>.** A suspension of TaCl<sub>5</sub> (1.0 g, 2.80 mmol) in 50 mL CH<sub>2</sub>Cl<sub>2</sub> was stirred for 5 minutes. A solution of (hpp)SiMe<sub>3</sub> (0.53 g, 2.53 mmol) in 5 mL CH<sub>2</sub>Cl<sub>2</sub> was slowly added dropwise. The solution color changed to deep red-brown and TaCl<sub>5</sub> slowly dissolved. The reaction mixture was stirred overnight at room temperature. Charcoal was added and the mixture stirred for 10 minutes. The deep-brown solution was filtered and concentrated to ~2 mL volume. The solution was layered with hexanes and stored at -40°C. Yellow-brown Ta(hpp)Cl<sub>4</sub> (0.8 g, 70% based on (hpp)SiMe<sub>3</sub>) was recovered by filtration. Yellow crystals suitable for X-ray diffractometry were grown by

dissolution in a minimum amount of  $\text{CH}_2\text{Cl}_2$ , layering with hexanes, and cooling to  $-40^\circ\text{C}$  for 3 days.  $^1\text{H}$  NMR ( $\delta$ ,  $\text{C}_6\text{D}_6$ , 300 MHz): 0.83 (m, 2H,  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 1.77 (t, 2H, 6 Hz,  $\text{NCH}_2$ ), 3.70 (t, 2H, 6 Hz,  $\text{NCH}_2$ ).  $^1\text{H}$  NMR ( $\delta$ ,  $\text{CD}_2\text{Cl}_2$ , 300 MHz): 2.08 (quintet, 2H, 6.0 Hz,  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 3.5 (t, 2H, 6.0 Hz,  $\text{NCH}_2$ ), 4.29 (t, 2H, 6.0 Hz,  $\text{NCH}_2$ ).

**Attempted Synthesis of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  from  $\text{TaCl}_5$  and  $(\text{hpp})\text{SiMe}_3$ .** A solution of  $(\text{hpp})\text{SiMe}_3$  (0.42 g, 2.0 mmol) in 2 mL  $\text{CH}_2\text{Cl}_2$  was slowly added to a suspension of  $\text{TaCl}_5$  (0.36 g, 1.00 mmol) in 15 mL  $\text{CH}_2\text{Cl}_2$ . The yellow-orange solution was stirred overnight at room temperature. The solution was filtered and concentrated under vacuum to  $\sim 2$  mL volume and mixed with hexane to yield ruby-red crystals of the previously reported  $[\text{Ta}(\text{hpp})_4]^+[\text{TaCl}_6]^-$  as determined by X-ray diffractometry. The product is not soluble in any organic solvent except  $\text{CH}_2\text{Cl}_2$ . Crystals for X-ray diffractometry were grown by dissolving in minimum  $\text{CH}_2\text{Cl}_2$ , layering with hexanes, and storing at  $-40^\circ\text{C}$  for one day.

**Preparation of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  via Reaction of  $\text{Ta}(\text{hpp})\text{Cl}_4$  and  $(\text{hpp})\text{SiMe}_3$ .** To a stirred solution of  $\text{Ta}(\text{hpp})\text{Cl}_4$  (0.6 g, 1.3 mmol) in 60 mL at room temperature was added slowly a solution of  $(\text{hpp})\text{SiMe}_3$  (0.28 g, 1.3 mmol) in 5 mL  $\text{CH}_2\text{Cl}_2$ . The mixture was stirred for one hour. The solution was concentrated in vacuum to  $\sim 2$  mL volume, layered with 1 mL hexanes, and stored at  $-40^\circ\text{C}$ . Bright-yellow crystals of  $\text{Ta}(\text{hpp})_2\text{Cl}_3$  (0.72 g, 98% based on  $\text{Ta}(\text{hpp})\text{Cl}_4$ ) were recovered by filtration and dried *in vacuo*.  $^1\text{H}$  NMR ( $\delta$ ,  $\text{C}_6\text{D}_6$ , 300 MHz): 1.34 (m, 2H,  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 2.65 (t, 2H, 6.0 Hz,  $\text{NCH}_2$ ), 3.9-4.2 (broad m, 2H,  $\text{NCH}_2$ ).  $^1\text{H}$  NMR ( $\delta$ ,  $\text{CD}_2\text{Cl}_2$ , 300 MHz): 1.96 (t, 2H, 6.0 Hz,  $\text{CH}_2\text{CH}_2\text{CH}_2$ ), 3.33 (t, 2H, 6.0 Hz,  $\text{NCH}_2$ ), 3.99 (t, 2H, 6.0 Hz,  $\text{NCH}_2$ ).

**Preparation of  $(\text{C}_5\text{Me}_5)\text{Ta}(\text{hpp})\text{Cl}_3$ .**  $(\text{C}_5\text{Me}_5)\text{TaCl}_4$  (2.01 g, 4.39 mmol) was dissolved in 120 mL  $\text{CH}_2\text{Cl}_2$ . A solution of  $(\text{hpp})\text{SiMe}_3$  (0.93 g, 4.39 mmol) in 5 mL  $\text{CH}_2\text{Cl}_2$  was slowly added. The color of the solution immediately changed from bright yellow to deep pink-red. The mixture was stirred for 30 minutes and the solution concentrated under vacuum to  $\sim 5$  mL volume. Hexanes (2 mL) were added and the solution stored at  $-40^\circ\text{C}$



for two hours. Red crystals of  $(C_5Me_5)Ta(hpp)Cl_3$  (2.13 g, 86% based on  $(C_5Me_5)TaCl_4$ ) were recovered by filtration and dried *in vacuo*.  $^1H$  NMR ( $\delta$ ,  $C_6D_6$ , 300 MHz): 1.19 (quintet, 2H, 6.0 Hz,  $CH_2CH_2CH_2$ ), 1.48 (quintet, 2H, 6.0 Hz,  $CH_2CH_2CH_2$ ), 2.17 (t, 2H, 6.0 Hz,  $NCH_2CH_2$ ), 2.25 (s, 15H,  $C_5Me_5$ ), 2.31 (t, 2H,  $NCH_2CH_2$ ), 3.30 (t, 2H, 6.0 Hz,  $NCH_2CH_2$ ), 4.08 (t, 2H, 6.0 Hz,  $NCH_2CH_2$ ).

**Preparation of  $(C_5Me_4Et)Ta(hpp)Cl_3$ .** A solution of  $(hpp)SiMe_3$  (0.22 g, 1.06 mmol) in 2 mL  $CH_2Cl_2$  was slowly added to a stirred solution of  $(C_5Me_4Et)TaCl_4$  (0.5 g, 1.06 mmol) in 5 mL  $CH_2Cl_2$ . An immediate color change was observed from bright yellow to deep pink-red. The mixture was stirred for 30 minutes. The solution was concentrated to ~1 mL volume, layered with 2 mL hexanes, and stored at  $-40^\circ C$  for 2 hours to yield red crystals of  $(C_5Me_4Et)Ta(hpp)Cl_3$  (0.58 g, 95% based on  $(C_5Me_4Et)TaCl_4$ ).  $^1H$  NMR ( $\delta$ ,  $C_6D_6$ , 300 MHz): 0.85 (t, 3H, 7.65 Hz,  $CH_3CH_2$ ), 1.12 (quintet, 2H, 5.76 Hz,  $NCH_2CH_2CH_2N$ ), 1.52 (t, 2H, 5.84 Hz,  $NCH_2$ ), 2.22-2.62 (m, 8H, Me and  $NCH_2$ ), 2.31-2.36 (m, 8H, Me and  $NCH_2$ ), 2.83 (q, 2H, 7.65 Hz,  $CH_3CH_2$ ), 3.33 (t, 2H, 5.63 Hz,  $NCH_2$ ), 4.09 (t, 2H, 5.80 Hz,  $NCH_2$ ).

**Preparation of  $(C_5Me_5)Ta(hpp)Cl_2$  via Reaction of  $(C_5Me_5)_2Ta_2(\mu-Cl)_4$  with  $(hpp)SiMe_3$ .** A solution of  $(C_5Me_4Et)_2Ta_2(\mu-Cl)_4$  (0.2 g, 0.25 mmol) in 10 mL toluene was treated with  $(hpp)SiMe_3$  (2 equivalents, 0.05 g, 0.50 mmol). The solution was stirred overnight at room temperature. The solution was concentrated *in vacuo* to ~2 mL volume and stored at  $-40^\circ C$  overnight. Orange-red crystals of  $(C_5Me_4Et)Ta(hpp)Cl_2$  (0.06 g, 45% based on  $(C_5Me_4Et)_2Ta_2(\mu-Cl)_4$ ) were filtered, washed with hexanes, and dried under vacuum.

**Preparation of  $(C_5Me_5)Ta(hpp)Cl_2$  via Na/Hg Reduction of  $(C_5Me_5)Ta(hpp)Cl_3$ .** To a suspension of  $(C_5Me_5)Ta(hpp)Cl_3$  (0.21 g, 0.37 mmol) in 15 mL benzene was added 0.413% w/w Na/Hg (2.09 g, 0.37 mmol Na) in a 20 mL scintillation vial. The vial was tightly capped and agitated on a vortex mixer for 2 hours. The color changed from deep pink-red to orange-red. Amalgum was allowed to settle for 15 minutes. The supernatant

solution was filtered through Celite to yield an orange-red solution. The solution was concentrated under vacuum to ~3 mL volume and hexanes (2 mL) were layered on top. After sitting undisturbed for 12 hours at room temperature, orange crystals of  $(C_5Me_5)Ta(hpp)Cl_2$  (0.15 g, 75% based on  $(C_5Me_4Et)Ta(hpp)Cl_3$ ) were recovered by filtration, washed with hexane, and dried under vacuum.

**X-Ray Diffractometry:  $Ta(hpp)Cl_4$ .** A yellow prismatic crystal with 0.11 x 0.09 x 0.07 mm<sup>3</sup> dimensions was mounted *via* grease to the tip of a glass fiber (epoxied to a brass pin) and placed on the diffractometer with the long crystal dimension (the *b* unit cell axis) approximately parallel to the diffractometer phi axis. Data were collected on a Nonius KappaCCD diffractometer (Mo  $K_\alpha$  radiation, graphite monochromator) at 150 K (cold N<sub>2</sub> gas stream) using standard CCD data collection techniques. Lorentz and polarization corrections were applied to the 6825 data. A correction for absorption using the multi-scan technique was applied ( $T_{max} = 0.6317$ ,  $T_{min} = 0.5037$ ). Equivalent data were averaged yielding 2078 unique data ( $R-int = 0.1179$ , 1827 with  $F > 4\sigma(F)$ ). Based on preliminary examination of the crystal, the space group *Fdd2* was assigned. The computer programs from the HKLint package were used for data reduction. Structure refinement was performed with the SHELXTL v6.1 package.

The preliminary model of the structure was obtained using XS, a direct methods program. Least-squares refining of the model vs. the data was performed with the XL program. Tables were made with the XCIF program. All non-hydrogen atoms were refined with anisotropic thermal parameters. All H atoms were included with the riding model using the XL program default values. Any restraints and constraints imposed on the data are described in the .cif files.

**X-Ray Diffractometry:  $Ta(hpp)_2Cl_3$ .** A yellow prismatic crystal with 0.12 x 0.07 x 0.05 mm<sup>3</sup> dimensions was mounted *via* grease to the tip of a glass fiber (epoxied to a brass pin) and placed on the diffractometer with the long crystal dimension (the diagonal of the *a* and *c* unit cell axes) approximately parallel to the diffractometer phi axis. Data were collected on a Nonius KappaCCD diffractometer (Mo  $K_\alpha$  radiation, graphite monochromator) at 190 K (cold N<sub>2</sub> gas stream) using standard CCD data collection

techniques. Lorentz and polarization corrections were applied to the 28281 data. A correction for absorption using the multi-scan technique was applied ( $T_{\max} = 0.7442$ ,  $T_{\min} = 0.5195$ ). Equivalent data were averaged yielding 4483 unique data ( $R\text{-int} = 0.0583$ , 3202 with  $F > 4\sigma(F)$ ). Based on preliminary examination of the crystal, the space group *Pbca* was assigned. The computer programs from the HKLint package were used for the data reduction. Structure refinement was performed with the SHELXTL v6.1 package.

The preliminary model of the structure was obtained using XS, a direct methods program. Least-squares refining of the model vs. the data was performed with the XL program. Tables were made with the XCIF program. All non-hydrogen atoms were refined with anisotropic thermal parameters. All H atoms were included with the riding model using the XL program default values. Any restraints and constraints imposed on the data are described in the .cif files.

**X-Ray Diffractometry: (C<sub>5</sub>Me<sub>5</sub>)Ta(hpp)Cl<sub>3</sub>.** An orange prismatic crystal with 0.16 x 0.16 x 0.10 mm<sup>3</sup> dimensions was mounted *via* grease to the tip of a glass fiber (epoxied to a brass pin) and placed on the diffractometer with the long crystal dimension (the diagonal of the *a* and *c* unit cell axes) approximately parallel to the diffractometer phi axis. Data were collected on a Nonius KappaCCD diffractometer (Mo K $\alpha$  radiation, graphite monochromator) at 150 K (cold N<sub>2</sub> gas stream) using standard CCD data collection techniques. Lorentz and polarization corrections were applied to the 33458 data. A correction for absorption using the multi-scan technique was applied ( $T_{\max} = 0.9290$ ,  $T_{\min} = 0.8898$ ). Equivalent data were averaged yielding 9421 unique data ( $R\text{-int} = 0.0420$ , 7671 with  $F > 4\sigma(F)$ ). Based on preliminary examination of the crystal, the space group *C2/c* was assigned. The computer programs from the HKLint package were used for the data reduction. Structure refinement was performed with the SHELXTL v6.1 package.

The preliminary model of the structure was obtained using XS, a direct methods program. Least-squares refining of the model vs. the data was performed with the XL program. Tables were made with the XCIF program. All non-hydrogen atoms were refined with anisotropic thermal parameters. All H atoms were included with the riding

model using the XL program default values. Any restraints and constraints imposed on the data are described in the .cif files.

**X-Ray Diffractometry: (C<sub>5</sub>Me<sub>5</sub>)Ta(hpp)Cl<sub>2</sub>.** An orange plate with 0.12 x 0.07 x 0.03 mm<sup>3</sup> dimensions was mounted *via* grease to the tip of a glass fiber (epoxied to a brass pin) and placed on the diffractometer with the long crystal dimension (the body diagonal of the unit cell axes) approximately parallel to the diffractometer phi axis. Data were collected on a Nonius KappaCCD diffractometer (Mo K<sub>α</sub> radiation, graphite monochromator) at 190 K (cold N<sub>2</sub> gas stream) using standard CCD data collection techniques. Lorentz and polarization corrections were applied to the 8387 data. A correction for absorption using the multi-scan technique was applied (T<sub>max</sub> = 0.8393, T<sub>min</sub> = 0.5303). Equivalent data were averaged yielding 8387 unique data (R-int = 0.0000, 6916 with F > 4σ(F)). Based on preliminary examination of the crystal, the space group *P* $\bar{1}$  was assigned. The computer programs from the HKLint package were used for the data reduction. Structure refinement was performed with the SHELXTL v6.1 package.

The preliminary model of the structure was obtained using XS, a direct methods program. Least-squares refining of the model vs. the data was performed with the XL program. Tables were made with the XCIF program. All non-hydrogen atoms were refined with anisotropic thermal parameters. All H atoms were included with the riding model using the XL program default values. Any restraints and constraints imposed on the data are described in the cif files.

**X-Ray Diffractometry: (C<sub>5</sub>Me<sub>4</sub>Et)Ta(hpp)Cl<sub>2</sub>.** An orange plate with 0.16 x 0.13 x 0.05 mm<sup>3</sup> dimensions was mounted *via* grease to the tip of a glass fiber (epoxied to a brass pin) and placed on the diffractometer with the long crystal dimension (the *b* unit cell axis) approximately parallel to the diffractometer phi axis. Data were collected on a Nonius KappaCCD diffractometer (Mo K<sub>α</sub> radiation, graphite monochromator) at 150 K (cold N<sub>2</sub> gas stream) using standard CCD data collection techniques. Lorentz and polarization corrections were applied to the 8346 data. A correction for absorption using the multi-scan technique was applied (T<sub>max</sub> = 0.7564, T<sub>min</sub> = 0.4512). Equivalent data

were averaged yielding 4630 unique data ( $R\text{-int} = 0.0352$ , 3939 with  $F > 4\sigma(F)$ ). Based on preliminary examination of the crystal, the space group  $P \bar{1}$  was assigned. The computer programs from the HKLint package were used for the data reduction. Structure refinement was performed with the SHELXTL v6.1 package.

The preliminary model of the structure was obtained using XS, a direct methods program. Least-squares refining of the model vs. the data was performed with the XL program. Tables were made with the XCIF program. All non-hydrogen atoms were refined with anisotropic thermal parameters. All H atoms were included with the riding model using the XL program default values. Any restraints and constraints imposed on the data are described in the .cif files.

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APPENDIX A

COMPUTATIONAL DATA

Table A 1: Energies for neutral bicyclic guanidines.

	<b>AM1 (kcal/mol)</b>	<b>AM1-SM2 (kcal/mol)</b>	<b>3-21G (Hartrees)</b>	<b>6-31G* (Hartrees)</b>
1	26.5	-13.2	-433.525791	-435.960229
2	10.23	-8.12	-588.810048	-592.093328
3	24.29	-9.86	-588.788187	-592.070551
4	-1.79	-8.18	-666.431134	-670.149474
5	-4.85	-7.36	-744.065748	-748.206749
6	4.48	-8.9	-666.422225	-670.136997
7	2.69	-5.82	-744.053321	-748.189589
8	20.05	-11.42	-511.165661	-514.026374
9	16.1	-10.49	-511.169891	-514.031063
10	77.34	-11.21	-976.681271	-973.098645
11	-13.49	-8.28	-744.062153	-748.207000
12	-7.58	-7.79	-744.055814	-748.198011
13	-51.35	-12.94	-660.034535	-663.717230
14	-42.97		-1243.804505	-1250.338031
15	34.7		-1302.257060	-1309.027075
16	-60.27	-11.72	-630.195684	-633.684010
17	-286.15	-8.96	-1101.161081	-1107.186733
18	24.51		-718.349412	-722.210779
19	18.29	-11.76	-548.819648	-551.893447
20	18.44	-9.81	-511.167170	-514.028465
21	15.53	-6.42	-588.814145	-592.100019
22	0.51	-5.42	-666.427941	-670.145705
23	-2.94	-12.41	-744.062448	-748.203062
24	12.24	-9.56	-588.802797	-592.089862
25	10.93	-8.51	-588.804445	-592.089582
26	14.29	-7.53	-588.796821	-592.085050
27	11.94	-6.93	-588.810748	-592.097953
28	25.41		-795.978690	-800.263337
29	-52.88	-11.31	-660.042596	-663.727356
30	-50.75		-1243.815398	-1250.349075
31	32.17	-9.72	-1302.263857	-1309.035669
32	-60.79	-13.91	-630.191923	-633.682762
33	-288.82	-9.41	-1101.160210	-1107.192508

Table A 2: Energies for bicyclic guanidates.

	<b>AM1 (kcal/mol)</b>	<b>AM1-SM2 (kcal/mol)</b>	<b>3-21G//AM1 (Hartrees)</b>	<b>6-31G*//AM1 (Hartrees)</b>
1	24.6	-82.86	-432.881630	-435.333420
2	7.75	-75.23	-588.168468	-591.469087
3	22.55	-76.56	-588.139723	-591.439169
4	-4.66	-74.86	-666.788377	-669.523456
5	-8	-73.43	-743.424531	-747.582142
6	0.59	-73.2	-665.785265	-669.517499
7	0.13	-69.06	-743.417995	-747.571211
8	18.87	-79.7	-510.521330	-513.397930
9	14.01	-78.79	-510.524291	-513.402509
10	75.15	-74.67	-967.033991	-972.469261
11	-14.25	-75.02	-743.414645	-747.578598
12	-9.69	-72.72	-743.409805	-747.570405
13	-57.46	-76.88	-659.395277	-663.093993
14	-46.09		-1243.158604	-1249.708335
15	21.96		-1301.631684	-1308.410553
16	-69.53	-76.07	-629.567656	-633.071141
17	-303.61	-65.56	-1100.537023	-1106.576517
18	21.45		-717.705901	-721.580748
19	17.3	-80.45	-548.172691	-551.263953
20	17.33	-74.94	-510.525151	-513.402910
21	14.89	-67.27	-588.175127	-591.477120
22	-1.85	-66.25	-665.792976	-669.526343
23	-6.29	-57.55	-744.441747	-747.584178
24	10.66	-77.79	-588.157839	-591.461712
25	9.27	-75.12	-588.160146	-591.462524
26	13.57	-72.23	-588.154514	-591.459296
27	11.95	-71.15	-588.169583	-591.475144
28	20.21		-795.332385	-799.633078
29	-62.39	-70.46	-659.420193	-663.119338
30	-56.4		-1243.188511	-1249.735202
31	9.86	-63.05	-1301.659406	-1308.435101
32	-73.03	-79.07	-629.575113	-633.081031
33	-314.84	-62.9	-1100.551266	-1106.597546

Table A 3: Dihedral angles for bicyclic guanidates.

	<b>N2C1NIC2</b>	<b>N2C1NIC3</b>	<b>C1NIC2C4</b>	<b>C1NIC3C5</b>	<b>N1C2C4C6</b>	<b>N1C3C5C7</b>
1	41.2	-2.4	-45.1	-22.8	48.6	44.9
2	-43.6	0.4	47.1	25.6	-48.4	-46.1
3	43.8	5.9	-52.8	22.9	56	-49.8
4	37.3	-14.7	-45.9	42.4	52.7	-54.2
5	35.1	-15.3	-41.8	43.3	50	-54.5
6	47.6	23.6	-54.6	-9.1	53.3	-22.5
7	27.2	30.2	-32.6	-45.9	41.7	40.3
8	36.4	-11.4	-44	38.2	51	-52.8
9	36.1	-14.4	-43.1	39.4	50.1	-49.9
10	33.1	-6.3	-35.9	33.6	43.2	-52.1
11	3.6	30.7	-19.9	-49.8	45	48.8
12	27	17.1	-35	-40.8	45.4	50.8
13	22.8	7.8	-21	-29.5	33	45.2
14	44	3.7	-46.4	-22.3	46	36.3
15	31.4	7	-38	-27.2	46.8	41.7
16	15.3	5.9	-23.7	-26.5	42.2	43.4
17	15.1	12.4	-23.3	-31.7	41.3	43.4
18	-55	25.1	60.5	-55	-65.3	68.1
19	-16.4	22.8	53.3	3.2	-40.7	-35.8
20	41.4	-3	-44.8	-22.3	48.3	45.5
21	41.2	-3.9	-43.1	-21.1	47.8	45.4
22	44.9	-2.1	-45.3	-23.7	43.4	46.6
23	45.5	0.3	-44.7	-23.6	43	41.8
24	43.5	3.3	-49.5	-29.8	51.9	49.1
25	38.2	2.3	-44	-26.1	48.2	44.7
26	47.3	7.3	-45.8	-29.3	41.9	42
27	5	-22.2	-46.4	-4.3	52	37.3
28	-50.8	2.1	57.9	-54.6	-65.2	67.8
29	38	-3	-45.1	-23.5	48.9	46.3
30	20.3	-16	-51.7	-7.8	32.9	30.8
31	-3.7	20.9	-11.6	-40.4	37.5	45.7
32	35.2	-4.8	-43.1	-21.5	49.6	46.6
33	35.2	-2.6	-43.8	-22.6	47	43.5

Table A 4: Dihedral angles for various bicyclic guanidines (continued).

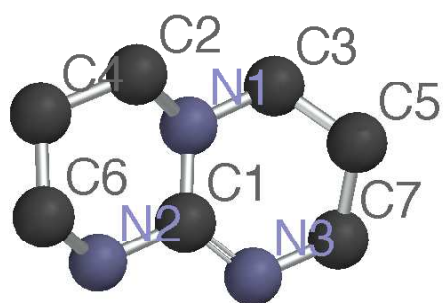
	<b>C2C4C6N2</b>	<b>C3C5C7N3</b>	<b>C4C6N2C1</b>	<b>C5C7N3C1</b>	<b>C6N2C1N1</b>	<b>C7N3C1N1</b>
1	-49.8	-45.2	47.5	23.2	-42.4	2.3
2	48.7	44.5	-47.7	-22.6	43.8	-2
3	-49.7	51.2	40.3	-23.9	-37.1	-5.9
4	-52.8	41	46.7	-15.9	-37.9	0.9
5	-54	40.9	50	-15.9	-39.3	1
6	-47.1	44.7	39.5	-33.7	-38.5	-0.3
7	-47.3	-19.1	41.7	0.6	-31	-5.3
8	-51.6	42.2	45.1	-16.2	-36.8	-0.5
9	-52.2	37.3	47.6	-14	-38.6	1
10	-49.1	45.7	47.2	-19.5	-38.7	-1.5
11	-56.3	-29.4	41.8	8.7	-14.9	-8.4
12	-50.2	-39.7	42.7	15.7	-30.1	-3
13	-48.3	-42.2	51.4	22	-38	-3.3
14	-44	-33.3	-41.2	-0.5	-41.2	-0.5
15	-51.3	-38	46.5	19.7	-35.3	-2.9
16	-55.2	-43	48.8	23.7	-27.8	-3.8
17	-53.3	-37.8	47.2	19.5	-27.2	-5.6
18	58	-43.6	-50.6	6.9	52.4	-1
19	-5.6	48.5	44	-26.5	-33.7	-10.4
20	-48.8	-45.7	46.9	23.5	-42.9	2.2
21	-50.3	-46.5	49.8	24.5	-45.4	1.7
22	-40.6	-45.6	41.2	22.9	-43.5	2.2
23	-41.3	-37.6	43.1	16.4	-45.6	3.4
24	-50.8	-45.3	45.4	20.8	-40.6	1.9
25	-49.6	-43	45.3	21.5	-38.2	0.5
26	-39.6	-33.7	40.6	12.2	-44.3	1.9
27	-18.3	-50.7	-24.3	27.3	32.9	9.6
28	60.3	-44.9	-50.8	8.2	48.9	-2.3
29	-47.4	-45.8	42.4	22.7	-36.9	3.2
30	13.9	-33.8	-46.8	12.7	30.2	13.5
31	-49.8	-33.1	37	13.7	-9.8	-6.5
32	-49.5	-48.6	43.9	25.8	-36.1	2.2
33	-43.6	-42.4	36.8	19.9	-31.7	4

Table A 5: Electrostatic charges on neutral bicyclic guanidines (Coulombs).

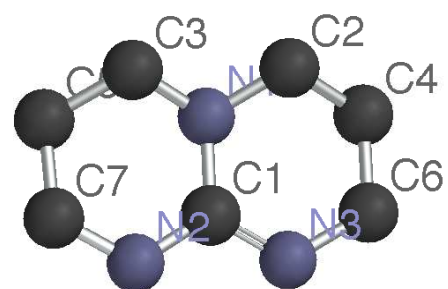
	<b>N1</b>	<b>N3</b>	<b>N2</b>
1	-0.59	-0.85	-0.85
2	-0.51	-0.82	-0.79
3	-1.15	-0.78	-0.84
4	-0.59	-0.85	-0.82
5	-0.6	-0.85	-0.82
6	-0.68	-0.78	-0.62
7	-0.59	-0.82	-0.87
8	-0.8	-0.79	-0.82
9	-0.55	-0.9	-0.82
10	-0.83	-0.8	-0.84
11	-0.84	-0.85	-0.9
12	-0.53	-0.78	-0.84
13	-0.47	-0.85	-0.86
14	-0.72	-0.83	-0.86
15	-0.27	-0.81	-0.8
16	-0.54	-0.82	-0.85
17	-0.3	-0.78	-0.79
18	-0.46	-0.88	-0.84
19	-0.81	-0.89	-0.87
20	-0.69	-0.98	-0.97
21	0.61	-1	-1
22	-0.64	-0.93	-0.94
23	-0.63	-0.96	-0.96
24	-0.84	-0.85	-0.86
25	-0.71	-0.8	-0.83
26	-0.89	-0.97	-0.99
27	-0.85	-0.97	-0.98
28	-0.71	-0.8	-0.9
29	-0.66	-0.96	-0.93
30	-0.76	-0.93	-0.9
31	-0.76	-0.93	-0.9
32	-0.91	-0.9	-0.91
33	-0.57	-0.8	-0.84



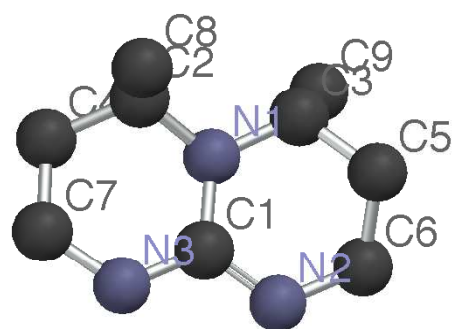
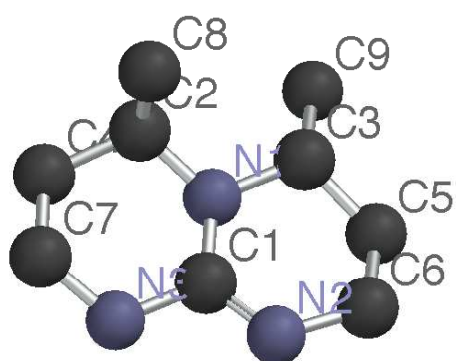
Figure A 1: Ball and Spoke Models of carbon and nitrogen backbones of neutral and anionic ligands from Spartan Calculations.



**hppH**



**hpp<sup>-</sup>**



## APPENDIX B

CRYSTALLOGRAPHIC DATA FOR  $\text{hpp}'\text{H}_2^+\text{I}^-$

## Crystal data and structure refinement for mes1028.

Identification code	mes1028	
Empirical formula	C <sub>9</sub> H <sub>18</sub> I N <sub>3</sub>	
Formula weight	295.16	
Temperature	190(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 2(1) 2(1) 2(1)	
Unit cell dimensions	a = 7.4508(8) Å	α = 90°.
	b = 10.5820(11) Å	β = 90°.
	c = 15.0197(16) Å	γ = 90°.
Volume	1184.2(2) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.656 Mg/m <sup>3</sup>	
Absorption coefficient	2.670 mm <sup>-1</sup>	
F(000)	584	
Crystal size	0.22 x 0.09 x 0.02 mm <sup>3</sup>	
Theta range for data collection	2.35 to 27.87°.	
Index ranges	-9 ≤ h ≤ 9, -13 ≤ k ≤ 13, -19 ≤ l ≤ 19	
Reflections collected	10536	
Independent reflections	2806 [R(int) = 0.0424]	
Completeness to theta = 27.87°	100.0 %	
Max. and min. transmission	0.9485 and 0.5912	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	2806 / 0 / 118	
Goodness-of-fit on F <sup>2</sup>	0.989	
Final R indices [I > 2σ(I)]	R1 = 0.0329, wR2 = 0.0827	
R indices (all data)	R1 = 0.0408, wR2 = 0.1027	
Absolute structure parameter	-0.09(4)	
Largest diff. peak and hole	1.256 and -0.937 e.Å <sup>-3</sup>	

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1028.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

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	x	y	z	$U(\text{eq})$
I(1)	1783(1)	429(1)	9338(1)	31(1)
N(3)	8066(6)	-1155(4)	11721(3)	24(1)
C(1)	7106(7)	-782(5)	12414(3)	26(1)
C(3)	8932(8)	-3045(5)	12547(4)	32(1)
C(2)	8657(8)	-2276(6)	13384(4)	32(1)
C(4)	9396(7)	-2178(5)	11786(3)	28(1)
N(1)	5881(7)	136(5)	12344(3)	36(1)
C(6)	7037(8)	689(5)	10900(4)	32(1)
C(5)	7664(7)	-662(5)	10822(3)	28(1)
N(2)	7359(6)	-1286(5)	13217(3)	32(1)
C(7)	5452(9)	794(6)	11529(4)	37(1)
C(8)	11309(8)	-1648(6)	11836(4)	37(1)
C(9)	6351(10)	-1510(6)	10333(4)	49(2)

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Bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ] for mes1028.

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N(3)-C(1)	1.323(6)
N(3)-C(4)	1.471(7)
N(3)-C(5)	1.478(6)
C(1)-N(2)	1.333(7)
C(1)-N(1)	1.337(7)
C(3)-C(4)	1.506(8)
C(3)-C(2)	1.511(8)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(2)-N(2)	1.447(7)
C(2)-H(4A)	0.9900
C(2)-H(4B)	0.9900
C(4)-C(8)	1.534(8)
C(4)-H(2A)	1.0000
N(1)-C(7)	1.444(7)
N(1)-H(1A)	0.8800
C(6)-C(5)	1.509(8)
C(6)-C(7)	1.517(8)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(5)-C(9)	1.517(8)
C(5)-H(5A)	1.0000
N(2)-H(2B)	0.8800
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(1)-N(3)-C(4)	122.1(4)
C(1)-N(3)-C(5)	120.2(4)

C(4)-N(3)-C(5)	117.2(4)
N(3)-C(1)-N(2)	121.1(5)
N(3)-C(1)-N(1)	121.6(5)
N(2)-C(1)-N(1)	117.2(5)
C(4)-C(3)-C(2)	109.5(5)
C(4)-C(3)-H(3A)	109.8
C(2)-C(3)-H(3A)	109.8
C(4)-C(3)-H(3B)	109.8
C(2)-C(3)-H(3B)	109.8
H(3A)-C(3)-H(3B)	108.2
N(2)-C(2)-C(3)	109.7(4)
N(2)-C(2)-H(4A)	109.7
C(3)-C(2)-H(4A)	109.7
N(2)-C(2)-H(4B)	109.7
C(3)-C(2)-H(4B)	109.7
H(4A)-C(2)-H(4B)	108.2
N(3)-C(4)-C(3)	110.1(4)
N(3)-C(4)-C(8)	111.1(5)
C(3)-C(4)-C(8)	113.5(5)
N(3)-C(4)-H(2A)	107.3
C(3)-C(4)-H(2A)	107.3
C(8)-C(4)-H(2A)	107.3
C(1)-N(1)-C(7)	124.6(5)
C(1)-N(1)-H(1A)	117.7
C(7)-N(1)-H(1A)	117.7
C(5)-C(6)-C(7)	111.0(5)
C(5)-C(6)-H(6A)	109.4
C(7)-C(6)-H(6A)	109.4
C(5)-C(6)-H(6B)	109.4
C(7)-C(6)-H(6B)	109.4
H(6A)-C(6)-H(6B)	108.0
N(3)-C(5)-C(6)	109.0(4)
N(3)-C(5)-C(9)	111.3(5)
C(6)-C(5)-C(9)	113.5(5)
N(3)-C(5)-H(5A)	107.6
C(6)-C(5)-H(5A)	107.6

C(9)-C(5)-H(5A)	107.6
C(1)-N(2)-C(2)	122.7(4)
C(1)-N(2)-H(2B)	118.6
C(2)-N(2)-H(2B)	118.6
N(1)-C(7)-C(6)	108.7(5)
N(1)-C(7)-H(7A)	110.0
C(6)-C(7)-H(7A)	110.0
N(1)-C(7)-H(7B)	110.0
C(6)-C(7)-H(7B)	110.0
H(7A)-C(7)-H(7B)	108.3
C(4)-C(8)-H(8A)	109.5
C(4)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(4)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(5)-C(9)-H(9A)	109.5
C(5)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(5)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5

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Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for mes1028. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
I(1)	32(1)	35(1)	26(1)	2(1)	-1(1)	-1(1)
N(3)	24(2)	29(2)	19(2)	1(2)	-2(2)	2(2)
C(1)	30(3)	28(2)	22(2)	-1(2)	2(2)	-1(2)
C(3)	36(3)	31(3)	30(3)	4(2)	-7(2)	0(2)
C(2)	34(3)	34(3)	30(3)	6(2)	-2(2)	-2(2)
C(4)	29(3)	34(3)	22(2)	-4(2)	-3(2)	7(2)
N(1)	37(3)	45(3)	27(2)	6(2)	8(2)	14(2)
C(6)	36(3)	27(3)	33(3)	7(2)	-2(2)	0(2)
C(5)	30(3)	35(3)	20(2)	4(2)	1(2)	2(2)
N(2)	36(2)	39(2)	21(2)	0(2)	2(2)	7(2)
C(7)	39(3)	37(3)	36(3)	5(2)	2(2)	11(2)
C(8)	29(3)	49(3)	34(3)	3(3)	5(2)	7(2)
C(9)	60(5)	40(3)	45(3)	-11(3)	-26(3)	5(3)



Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1028.

	x	y	z	U(eq)
H(3A)	9914	-3661	12640	39
H(3B)	7823	-3520	12406	39
H(4A)	8222	-2830	13869	39
H(4B)	9811	-1898	13572	39
H(2A)	9311	-2686	11226	34
H(1A)	5295	354	12829	44
H(6A)	6687	1006	10304	39
H(6B)	8034	1221	11122	39
H(5A)	8811	-660	10476	34
H(2B)	6709	-1005	13665	39
H(7A)	5197	1694	11658	44
H(7B)	4372	416	11252	44
H(8A)	11524	-1091	11326	56
H(8B)	12173	-2346	11826	56
H(8C)	11453	-1167	12390	56
H(9A)	6834	-2370	10305	73
H(9B)	6168	-1189	9728	73
H(9C)	5202	-1518	10651	73

Torsion angles [°] for mes1028.

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C(4)-N(3)-C(1)-N(2)	2.8(8)
C(5)-N(3)-C(1)-N(2)	174.9(5)
C(4)-N(3)-C(1)-N(1)	-178.9(5)
C(5)-N(3)-C(1)-N(1)	-6.9(8)
C(4)-C(3)-C(2)-N(2)	53.2(6)
C(1)-N(3)-C(4)-C(3)	24.7(7)
C(5)-N(3)-C(4)-C(3)	-147.6(4)
C(1)-N(3)-C(4)-C(8)	-101.9(6)
C(5)-N(3)-C(4)-C(8)	85.8(6)
C(2)-C(3)-C(4)-N(3)	-51.8(6)
C(2)-C(3)-C(4)-C(8)	73.5(6)
N(3)-C(1)-N(1)-C(7)	0.9(9)
N(2)-C(1)-N(1)-C(7)	179.3(6)
C(1)-N(3)-C(5)-C(6)	34.1(7)
C(4)-N(3)-C(5)-C(6)	-153.4(5)
C(1)-N(3)-C(5)-C(9)	-91.8(6)
C(4)-N(3)-C(5)-C(9)	80.6(6)
C(7)-C(6)-C(5)-N(3)	-55.3(6)
C(7)-C(6)-C(5)-C(9)	69.5(6)
N(3)-C(1)-N(2)-C(2)	-1.0(8)
N(1)-C(1)-N(2)-C(2)	-179.4(5)
C(3)-C(2)-N(2)-C(1)	-27.9(7)
C(1)-N(1)-C(7)-C(6)	-22.9(8)
C(5)-C(6)-C(7)-N(1)	49.4(7)

---

## Observed and calculated structure factors for mes1028

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	0	0	1516	1572	9	3	10	0	182	175	5	-6	4	1	259	264	6	-6	9	1	194	185	16	-5	2	2	468	463	3
4	0	0	340	340	8	4	10	0	68	89	34	-5	4	1	476	470	5	-5	9	1	245	232	14	-4	2	2	393	396	3
6	0	0	650	641	10	5	10	0	170	179	14	-4	4	1	523	530	5	-4	9	1	107	120	25	-3	2	2	567	571	3
8	0	0	491	454	9	6	10	0	305	308	18	-3	4	1	389	393	3	-3	9	1	431	413	4	-2	2	2	695	718	4
1	1	0	560	566	3	1	11	0	411	387	5	-2	4	1	655	665	4	-2	9	1	284	279	4	-1	2	2	1431	1479	7
2	1	0	624	638	4	2	11	0	145	158	9	-1	4	1	1050	1081	6	-1	9	1	276	277	4	0	2	2	1131	1156	6
3	1	0	112	131	4	3	11	0	119	112	7	0	4	1	404	414	3	0	9	1	396	396	4	1	2	2	1458	1485	7
4	1	0	157	175	3	4	11	0	0	65	1	1	4	1	1050	1082	5	1	9	1	269	273	4	2	2	2	709	727	4
5	1	0	854	830	6	5	11	0	154	182	17	2	4	1	669	681	4	2	9	1	303	291	5	3	2	2	560	565	4
6	1	0	165	170	4	6	11	0	0	6	1	3	4	1	394	392	3	3	9	1	435	418	6	4	2	2	382	388	3
7	1	0	628	605	7	0	12	0	416	394	9	4	4	1	540	537	9	4	9	1	90	121	19	5	2	2	477	473	7
8	1	0	46	54	41	1	12	0	0	6	1	5	4	1	477	470	8	5	9	1	235	237	10	6	2	2	416	409	5
9	1	0	149	184	25	2	12	0	246	249	9	6	4	1	268	268	5	6	9	1	173	184	12	7	2	2	410	389	6
0	2	0	1550	1612	12	3	12	0	35	37	35	7	4	1	425	400	7	7	9	1	61	67	61	8	2	2	241	247	10
1	2	0	134	132	1	4	12	0	75	67	31	8	4	1	129	145	12	-6	10	1	90	119	27	9	2	2	165	200	17
2	2	0	1026	1070	6	1	13	0	374	349	10	9	4	1	139	150	18	-5	10	1	175	155	12	-9	3	2	142	155	14
3	2	0	649	680	4	2	13	0	122	112	13	-9	5	1	66	120	65	-4	10	1	411	397	10	-8	3	2	226	247	5
4	2	0	20	12	19	3	13	0	64	17	32	-8	5	1	332	333	9	-3	10	1	210	208	4	-7	3	2	206	209	5
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-5	5	12	340	330	11	7	1	13	46	86	46	2	8	13	176	190	12	-3	6	14	365	374	23	5	4	15	181	209	13
-4	5	12	123	131	21	-7	2	13	100	91	24	3	8	13	145	151	14	-2	6	14	182	183	9	-5	5	15	46	56	46
-3	5	12	591	568	8	-6	2	13	213	207	10	4	8	13	121	133	30	-1	6	14	210	210	7	-4	5	15	0	46	1
-2	5	12	182	183	5	-5	2	13	306	319	11	-3	9	13	186	172	19	0	6	14	53	50	24	-3	5	15	121	135	18
-1	5	12	370	368	5	-4	2	13	320	328	5	-2	9	13	289	283	25	1	6	14	201	216	15	-2	5	15	302	306	7
0	5	12	195	192	5	-3	2	13	426	410	8	-1	9	13	208	210	11	2	6	14	187	180	14	-1	5	15	67	71	19
1	5	12	357	356	5	-2	2	13	349	351	5	0	9	13	200	203	14	3	6	14	386	385	11	0	5	15	497	470	6
2	5	12	172	181	6	-1	2	13	295	300	5	1	9	13	192	207	14	4	6	14	165	166	15	1	5	15	60	70	29
3	5	12	569	557	9	0	2	13	163	167	4	2	9	13	268	284	10	5	6	14	247	239	13	2	5	15	320	313	9
4	5	12	138	136	11	1	2	13	307	314	4	3	9	13	162	177	15	-4	7	14	355	353	11	3	5	15	137	129	17
5	5	12	331	326	9	2	2	13	375	365	10	-2	10	13	117	105	22	-3	7	14	169	168	12	4	5	15	29	43	29
6	5	12	93	88	23	3	2	13	429	424	5	-1	10	13	71	101	70	-2	7	14	354	344	11	5	5	15	0	53	1
-6	6	12	140	146	15	4	2	13	329	335	6	0	10	13	45	34	44	-1	7	14	77	102	15	-4	6	15	39	27	38
-5	6	12	44	94	43	5	2	13	329	321	5	1	10	13	92	104	21	0	7	14	115	139	11	-3	6	15	118	102	12
-4	6	12	463	449	7	6	2	13	204	205	11	2	10	13	96	105	37	1	7	14	90	98	15	-2	6	15	44	60	44
-3	6	12	184	175	7	7	2	13	96	87	26	0	0	14	453	402	7	2	7	14	337	338	9	-1	6	15	471	464	8
0	6	15	7	37	6	-2	2	16	307	306	7	-2	6	16	102	111	20	0	3	17	244	244	15	-2	2	18	154	159	13
1	6	15	459	462	8	-1	2	16	200	197	8	-1	6	16	176	198	12	1	3	17	189	177	11	-1	2	18	353	350	9
2	6	15	72	57	29																								

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
4	0	16	105	105	18	0	4	16	201	208	8	2	1	17	244	255	10	1	6	17	280	268	10	-1	1	19	69	66	47
5	0	16	93	84	20	1	4	16	232	241	8	3	1	17	326	328	9	2	6	17	136	174	18	0	1	19	57	70	57
-5	1	16	228	239	12	2	4	16	114	126	14	4	1	17	228	237	11	0	7	17	270	269	11	1	1	19	42	67	42
-4	1	16	120	105	23	3	4	16	378	384	9	-4	2	17	202	199	20	0	0	18	139	135	51	2	1	19	361	337	12
-3	1	16	70	79	15	4	4	16	110	115	18	-3	2	17	256	269	12	1	0	18	418	394	8	-2	2	19	126	152	19
-2	1	16	106	130	8	5	4	16	287	291	14	-2	2	17	299	289	20	2	0	18	68	57	31	-1	2	19	139	160	14
-1	1	16	529	506	8	-4	5	16	318	306	11	-1	2	17	225	224	7	3	0	18	52	67	51	0	2	19	294	280	15
0	1	16	243	254	8	-3	5	16	176	194	9	0	2	17	187	210	11	4	0	18	0	51	1	1	2	19	150	163	15
1	1	16	538	508	7	-2	5	16	284	295	9	1	2	17	211	224	8	-3	1	18	129	136	15	2	2	19	151	156	23
2	1	16	133	132	13	-1	5	16	130	140	10	2	2	17	291	291	9	-2	1	18	228	253	13	-1	3	19	210	231	14
3	1	16	54	74	54	0	5	16	82	113	21	3	2	17	274	271	18	-1	1	18	203	196	15	0	3	19	0	12	1
4	1	16	115	117	14	1	5	16	141	142	13	4	2	17	193	206	21	0	1	18	396	386	8	1	3	19	206	222	17
5	1	16	252	236	10	2	5	16	301	302	11	-4	3	17	209	208	12	1	1	18	198	192	9						
-5	2	16	192	196	15	3	5	16	182	194	16	-3	3	17	137	149	11	2	1	18	242	258	9						
-4	2	16	94	131	18	4	5	16	303	307	11	-2	3	17	251	258	12	3	1	18	114	141	18						
-3	2	16	242	257	8	-3	6	16	403	387	19	-1	3	17	160	165	7	-3	2	18	76	94	67						

## APPENDIX C

CRYSTALLOGRAPHIC DATA FOR Ta(hpp)Cl<sub>4</sub>

Identification code	mes1015a	
Empirical formula	C7 H8 Cl4 N3 Ta	
Formula weight	456.91	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Fdd2	
Unit cell dimensions	a = 20.708(3) Å	$\alpha = 90^\circ$ .
	b = 22.603(3) Å	$\beta = 90^\circ$ .
	c = 10.6647(12) Å	$\gamma = 90^\circ$ .
Volume	4991.8(11) Å <sup>3</sup>	
Z	12	
Density (calculated)	1.824 Mg/m <sup>3</sup>	
Absorption coefficient	7.225 mm <sup>-1</sup>	
F(000)	2544	
Crystal size	0.11 x 0.09 x 0.07 mm <sup>3</sup>	
Theta range for data collection	3.45 to 25.36°.	
Index ranges	-24 ≤ h ≤ 24, -27 ≤ k ≤ 27, -12 ≤ l ≤ 10	
Reflections collected	6825	
Independent reflections	2078 [R(int) = 0.1179]	
Completeness to theta = 25.36°	99.8 %	
Max. and min. transmission	0.6317 and 0.5037	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	2078 / 1 / 83	
Goodness-of-fit on F <sup>2</sup>	1.241	
Final R indices [I > 2σ(I)]	R1 = 0.0741, wR2 = 0.1706	
R indices (all data)	R1 = 0.0846, wR2 = 0.1774	
Absolute structure parameter	1.01(3)	
Largest diff. peak and hole	11.170 and -2.027 e.Å <sup>-3</sup>	

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1015a.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

	x	y	z	$U(\text{eq})$
Ta(1)	8005(1)	-417(1)	978(1)	18(1)
Cl(3)	7804(3)	-1383(2)	1670(6)	25(1)
Cl(2)	8431(3)	-241(2)	3005(5)	27(1)
Cl(4)	7795(3)	-785(2)	-1066(5)	27(1)
Cl(1)	9034(3)	-174(3)	244(6)	30(1)
N(2)	7685(9)	427(7)	618(17)	21(4)
N(3)	6629(7)	726(7)	1028(18)	16(3)
N(1)	7067(8)	-228(8)	1384(16)	16(4)
C(5)	5974(12)	532(10)	1460(20)	31(6)
C(4)	6692(12)	1318(11)	570(30)	32(6)
C(7)	6476(10)	-470(9)	2020(20)	23(5)
C(3)	7427(10)	1496(10)	640(20)	29(5)
C(6)	5885(10)	-140(10)	1410(20)	25(5)
C(2)	7867(11)	977(11)	10(30)	32(6)

Bond lengths [Å] and angles [°] for mes1015a.

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Ta(1)-N(1)	2.035(16)
Ta(1)-N(2)	2.056(17)
Ta(1)-Cl(3)	2.341(5)
Ta(1)-Cl(1)	2.337(6)
Ta(1)-Cl(2)	2.368(6)
Ta(1)-Cl(4)	2.373(6)
N(2)-C(2)	1.45(3)
N(3)-C(4)	1.43(3)
N(3)-C(5)	1.50(3)
N(1)-C(7)	1.50(3)
C(5)-C(6)	1.53(3)
C(5)-H(4A)	0.9900
C(5)-H(4B)	0.9900
C(4)-C(3)	1.58(3)
C(4)-H(5A)	0.9900
C(4)-H(5B)	0.9900
C(7)-C(6)	1.57(3)
C(7)-H(2A)	0.9900
C(7)-H(2B)	0.9900
C(3)-C(2)	1.63(3)
C(3)-H(6A)	0.9900
C(3)-H(6B)	0.9900
C(6)-H(3A)	0.9900
C(6)-H(3B)	0.9900
C(2)-H(7A)	0.9900
C(2)-H(7B)	0.9900
N(1)-Ta(1)-N(2)	62.5(7)
N(1)-Ta(1)-Cl(3)	87.6(5)
N(2)-Ta(1)-Cl(3)	150.1(5)
N(1)-Ta(1)-Cl(1)	153.2(5)
N(2)-Ta(1)-Cl(1)	90.8(5)
Cl(3)-Ta(1)-Cl(1)	119.1(2)
N(1)-Ta(1)-Cl(2)	97.2(5)

N(2)-Ta(1)-Cl(2)	97.7(5)
Cl(3)-Ta(1)-Cl(2)	86.3(2)
Cl(1)-Ta(1)-Cl(2)	85.8(2)
N(1)-Ta(1)-Cl(4)	95.4(5)
N(2)-Ta(1)-Cl(4)	95.5(5)
Cl(3)-Ta(1)-Cl(4)	86.0(2)
Cl(1)-Ta(1)-Cl(4)	86.7(2)
Cl(2)-Ta(1)-Cl(4)	164.88(19)
C(2)-N(2)-Ta(1)	142.6(15)
C(4)-N(3)-C(5)	117.4(17)
C(7)-N(1)-Ta(1)	142.9(14)
N(3)-C(5)-C(6)	112.8(19)
N(3)-C(5)-H(4A)	109.0
C(6)-C(5)-H(4A)	109.0
N(3)-C(5)-H(4B)	109.0
C(6)-C(5)-H(4B)	109.0
H(4A)-C(5)-H(4B)	107.8
N(3)-C(4)-C(3)	108.1(19)
N(3)-C(4)-H(5A)	110.1
C(3)-C(4)-H(5A)	110.1
N(3)-C(4)-H(5B)	110.1
C(3)-C(4)-H(5B)	110.1
H(5A)-C(4)-H(5B)	108.4
N(1)-C(7)-C(6)	106.0(17)
N(1)-C(7)-H(2A)	110.5
C(6)-C(7)-H(2A)	110.5
N(1)-C(7)-H(2B)	110.5
C(6)-C(7)-H(2B)	110.5
H(2A)-C(7)-H(2B)	108.7
C(2)-C(3)-C(4)	109.6(18)
C(2)-C(3)-H(6A)	109.7
C(4)-C(3)-H(6A)	109.8
C(2)-C(3)-H(6B)	109.8
C(4)-C(3)-H(6B)	109.7
H(6A)-C(3)-H(6B)	108.2
C(5)-C(6)-C(7)	111.1(18)

C(5)-C(6)-H(3A)	109.4
C(7)-C(6)-H(3A)	109.4
C(5)-C(6)-H(3B)	109.4
C(7)-C(6)-H(3B)	109.4
H(3A)-C(6)-H(3B)	108.0
N(2)-C(2)-C(3)	106.7(18)
N(2)-C(2)-H(7A)	110.4
C(3)-C(2)-H(7A)	110.4
N(2)-C(2)-H(7B)	110.4
C(3)-C(2)-H(7B)	110.4
H(7A)-C(2)-H(7B)	108.6

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Symmetry transformations used to generate equivalent atoms.

Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for mes1015a. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Ta(1)	17(1)	16(1)	23(1)	2(1)	2(1)	1(1)
Cl(3)	27(3)	14(3)	34(3)	2(2)	1(3)	0(2)
Cl(2)	29(3)	25(3)	26(3)	0(2)	-7(2)	2(2)
Cl(4)	32(3)	26(3)	24(3)	-1(2)	1(2)	0(2)
Cl(1)	21(3)	35(3)	35(4)	2(3)	4(2)	-2(2)



Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1015a.

	x	y	z	U(eq)
H(4A)	5641	723	928	38
H(4B)	5906	668	2333	38
H(5A)	6430	1591	1092	39
H(5B)	6537	1342	-304	39
H(2A)	6492	-392	2931	28
H(2B)	6442	-902	1881	28
H(6A)	7496	1874	194	34
H(6B)	7555	1553	1531	34
H(3A)	5840	-266	520	30
H(3B)	5484	-251	1854	30
H(7A)	7785	955	-901	39
H(7B)	8331	1060	147	39

Torsion angles [ $^\circ$ ] for mes1015a.

N(1)-Ta(1)-N(2)-C(2)	-170(3)
Cl(3)-Ta(1)-N(2)-C(2)	-168(2)
Cl(1)-Ta(1)-N(2)-C(2)	10(2)
Cl(2)-Ta(1)-N(2)-C(2)	96(2)
Cl(4)-Ta(1)-N(2)-C(2)	-77(2)
N(2)-Ta(1)-N(1)-C(7)	-171(3)
Cl(3)-Ta(1)-N(1)-C(7)	10(2)
Cl(1)-Ta(1)-N(1)-C(7)	-170.8(18)
Cl(2)-Ta(1)-N(1)-C(7)	-76(2)
Cl(4)-Ta(1)-N(1)-C(7)	96(2)
C(4)-N(3)-C(5)-C(6)	156(2)
C(5)-N(3)-C(4)-C(3)	159.7(19)
Ta(1)-N(1)-C(7)-C(6)	-156.8(18)

N(3)-C(4)-C(3)-C(2)	49(3)
N(3)-C(5)-C(6)-C(7)	48(3)
N(1)-C(7)-C(6)-C(5)	-53(2)
Ta(1)-N(2)-C(2)-C(3)	-158.8(18)
C(4)-C(3)-C(2)-N(2)	-53(2)

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Observed and calculated structure factors for Ta(hpp)Cl<sub>4</sub>.

h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s
0	0	-12	387	252	21	0	10	-10	307	319	10	3	19	-9	138	130	15	7	1	-7	240	270	6	3	21	-7	208	199	32
4	0	-12	110	124	21	2	10	-10	149	154	10	0	0	-8	413	296	6	9	1	-7	168	188	5	5	21	-7	171	168	22
8	0	-12	231	275	13	4	10	-10	60	59	27	4	0	-8	154	158	6	11	1	-7	174	160	6	7	21	-7	145	157	17
2	2	-12	244	176	21	6	10	-10	91	120	18	8	0	-8	485	485	6	13	1	-7	433	426	8	2	0	-6	667	591	35
4	2	-12	130	140	14	8	10	-10	305	292	19	12	0	-8	432	397	12	15	1	-7	418	428	7	6	0	-6	437	449	9
6	2	-12	44	82	44	10	10	-10	88	70	27	16	0	-8	317	332	13	17	1	-7	210	226	8	10	0	-6	506	478	7
8	2	-12	80	94	30	12	10	-10	272	222	16	2	2	-8	493	412	35	19	1	-7	115	127	15	14	0	-6	152	137	19
0	4	-12	213	194	25	2	12	-10	252	241	8	4	2	-8	218	202	9	1	3	-7	151	161	4	18	0	-6	399	405	12
2	4	-12	205	168	16	4	12	-10	34	37	33	6	2	-8	234	248	7	3	3	-7	284	264	24	22	0	-6	445	372	14
4	4	-12	88	58	74	6	12	-10	156	166	12	8	2	-8	55	58	17	5	3	-7	393	410	9	0	2	-6	700	616	40
6	4	-12	251	249	14	8	12	-10	84	86	24	10	2	-8	454	449	9	7	3	-7	422	444	7	2	2	-6	280	247	24
8	4	-12	126	112	27	10	12	-10	401	322	10	12	2	-8	225	220	16	9	3	-7	247	260	5	4	2	-6	318	309	5
2	6	-12	0	11	1	0	14	-10	392	351	21	14	2	-8	100	102	12	11	3	-7	265	276	9	6	2	-6	286	283	4
4	6	-12	354	305	14	2	14	-10	61	44	34	16	2	-8	209	202	9	13	3	-7	220	229	7	8	2	-6	514	516	4
6	6	-12	110	79	28	4	14	-10	54	79	53	18	2	-8	300	281	11	15	3	-7	325	338	10	10	2	-6	72	87	15
0	8	-12	79	141	62	6	14	-10	159	175	13	0	4	-8	282	260	11	17	3	-7	270	281	9	12	2	-6	449	433	5
2	8	-12	158	183	19	8	14	-10	278	248	11	2	4	-8	273	258	10	19	3	-7	208	212	16	14	2	-6	273	275	6
4	8	-12	57	28	57	2	16	-10	140	154	16	4	4	-8	305	259	22	1	5	-7	360	380	7	16	2	-6	172	187	13
1	1	-11	236	189	16	4	16	-10	239	252	21	6	4	-8	575	495	44	3	5	-7	309	322	9	18	2	-6	104	122	14
3	1	-11	190	204	9	1	1	-9	160	145	5	8	4	-8	212	222	5	5	5	-7	273	258	16	20	2	-6	358	312	25
5	1	-11	283	291	11	3	1	-9	314	303	8	10	4	-8	51	79	21	7	5	-7	232	242	4	22	2	-6	144	142	29
7	1	-11	199	234	11	5	1	-9	393	403	4	12	4	-8	312	297	5	9	5	-7	428	423	5	2	4	-6	474	456	3
9	1	-11	167	169	10	7	1	-9	262	284	5	14	4	-8	426	409	8	11	5	-7	404	422	6	4	4	-6	803	729	73
11	1	-11	121	97	15	9	1	-9	149	162	9	16	4	-8	85	108	22	13	5	-7	239	246	7	6	4	-6	219	213	10
1	3	-11	141	162	12	11	1	-9	147	147	10	18	4	-8	124	129	18	15	5	-7	127	119	16	8	4	-6	320	316	5
3	3	-11	342	283	29	13	1	-9	229	230	7	2	6	-8	109	123	14	17	5	-7	222	223	9	10	4	-6	268	265	5
5	3	-11	114	112	18	15	1	-9	267	294	11	4	6	-8	647	608	27	19	5	-7	264	249	18	12	4	-6	200	221	8
7	3	-11	181	208	12	17	1	-9	173	186	11	6	6	-8	35	28	34	1	7	-7	322	353	10	14	4	-6	178	190	9
9	3	-11	164	188	10	1	3	-9	283	281	14	8	6	-8	425	386	50	3	7	-7	385	413	8	16	4	-6	428	413	6
11	3	-11	210	212	11	3	3	-9	384	344	26	10	6	-8	46	68	45	5	7	-7	63	57	9	18	4	-6	148	138	13
1	5	-11	296	312	11	5	3	-9	241	248	14	12	6	-8	236	250	10	7	7	-7	252	240	16	20	4	-6	92	53	20
3	5	-11	70	89	23	7	3	-9	313	330	5	14	6	-8	0	55	1	9	7	-7	392	390	7	0	6	-6	253	257	9
5	5	-11	98	88	22	9	3	-9	170	183	6	16	6	-8	410	365	17	11	7	-7	347	362	5	2	6	-6	482	460	6
7	5	-11	167	172	11	11	3	-9	197	196	14	18	6	-8	60	75	50	13	7	-7	268	286	16	4	6	-6	76	71	9
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3	9	5	472	476	8	4	2	6	357	327	14	8	16	6	450	382	43	5	13	7	379	385	9	12	14	8	148	126	32
5	9	5	425	408	5	6	2	6	315	306	7	10	16	6	255	221	12	7	13	7	374	359	10	14	14	8	172	141	31
7	9	5	468	452	15	8	2	6	512	502	16	12	16	6	253	244	15	9	13	7	221	190	21	0	16	8	188	183	25
9	9	5	290	279	39	10	2	6	70	88	21	14	16	6	0	64	1	11	13	7	144	71	31	2	16	8	308	301	21
11	9	5	143	144	17	12	2	6	450	450	13	16	16	6	342	310	30	13	13	7	262	237	16	4	16	8	81	98	47
13	9	5	206	196	11	14	2	6	281	277	16	0	18	6	98	74	26	15	13	7	218	216	20	6	16	8	504	419	28
15	9	5	288																										

h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s
17	13	5	288	292	16	6	8	6	48	37	48	9	3	7	275	285	13	1	21	7	214	175	21	11	11	9	143	127	30
19	13	5	77	115	76	8	8	6	198	151	10	11	3	7	236	263	16	3	21	7	311	218	26	13	11	9	268	207	20
1	15	5	260	277	7	10	8	6	395	369	78	5	5	7	300	251	13	5	21	7	118	148	30	3	13	9	289	265	20
3	15	5	279	294	11	12	8	6	342	325	33	7	5	7	264	268	9	7	21	7	147	153	28	5	13	9	213	238	28
5	15	5	319	325	17	14	8	6	162	172	22	9	5	7	414	407	11	6	4	8	629	487	21	7	13	9	251	263	15
7	15	5	208	216	21	16	8	6	472	411	60	11	5	7	443	439	10	8	4	8	219	220	15	9	13	9	173	133	26
9	15	5	421	355	48	18	8	6	90	110	51	13	5	7	232	236	18	6	6	8	0	29	1	11	13	9	243	236	25
11	15	5	266	231	30	20	8	6	30	74	30	15	5	7	103	120	40	8	6	8	504	367	20	13	13	9	249	191	22
13	15	5	187	220	18	0	10	6	593	571	10	17	5	7	273	231	19	10	6	8	44	69	43	3	15	9	205	219	27
15	15	5	156	169	75	2	10	6	115	95	10	1	7	7	318	343	12	12	6	8	258	257	13	5	15	9	275	284	24
17	15	5	218	211	24	4	10	6	142	126	15	3	7	7	414	408	13	4	8	8	92	39	38	7	15	9	193	162	26
1	17	5	334	332	11	6	10	6	284	256	7	5	7	7	55	41	55	6	8	8	554	511	11	9	15	9	245	187	37
3	17	5	250	266	9	8	10	6	455	423	9	7	7	7	268	245	39	8	8	8	261	222	22	11	15	9	198	182	65
5	17	5	81	116	22	10	10	6	111	112	66	9	7	7	388	381	10	10	8	8	48	27	47	3	17	9	127	127	49
7	17	5	219	187	39	12	10	6	416	310	14	11	7	7	367	354	11	12	8	8	172	141	21	5	17	9	84	72	43
9	17	5	407	386	11	14	10	6	428	331	40	13	7	7	251	274	13	14	8	8	494	437	56	7	17	9	60	101	59
11	17	5	329	317	11	16	10	6	183	161	22	15	7	7	140	105	60	16	8	8	108	36	91	3	19	9	86	118	85
13	17	5	138	153	21	18	10	6	78	52	78	17	7	7	224	205	31	18	8	8	74	107	73	6	12	10	127	162	36
15	17	5	16	102	15	20	10	6	362	355	33	19	7	7	217	209	33	2	10	8	356	352	14	8	12	10	37	87	37
1	19	5	310	315	8	2	12	6	468	436	6	1	9	7	311	327	7	4	10	8	284	279	15	10	12	10	381	327	25
3	19	5	103	121	19	4	12	6	124	131	11	3	9	7	254	273	7	6	10	8	170	159	17	6	14	10	131	167	38
5	19	5	166	88	39	6	12	6	188	184	8	5	9	7	285	269	10	8	10	8	194	192	41	8	14	10	273	248	30
7	19	5	218	216	20	8	12	6	155	159	34	7	9	7	342	324	17	10	10	8	514	401	31						
9	19	5	293	322	20	10	12	6	669	548	61	9	9	7	263	236	25	12	10	8	188	162	20						



## APPENDIX D

CRYSTALLOGRAPHIC DATA FOR Ta(hpp)<sub>2</sub>Cl<sub>3</sub>

Identification code	mes1021	
Empirical formula	C14 H24 Cl3 N6 Ta	
Formula weight	563.69	
Temperature	190(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 10.1922(11) Å	$\alpha = 90^\circ$ .
	b = 14.6406(15) Å	$\beta = 90^\circ$ .
	c = 25.205(3) Å	$\gamma = 90^\circ$ .
Volume	3761.1(7) Å <sup>3</sup>	
Z	8	
Density (calculated)	1.991 Mg/m <sup>3</sup>	
Absorption coefficient	6.281 mm <sup>-1</sup>	
F(000)	2192	
Crystal size	0.12 x 0.07 x 0.05 mm <sup>3</sup>	
Theta range for data collection	1.62 to 27.90°.	
Index ranges	-13 ≤ h ≤ 13, -19 ≤ k ≤ 19, -33 ≤ l ≤ 31	
Reflections collected	28281	
Independent reflections	4483 [R(int) = 0.0583]	
Completeness to theta = 27.90°	99.6 %	
Max. and min. transmission	0.7442 and 0.5195	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	4483 / 0 / 217	
Goodness-of-fit on F <sup>2</sup>	1.081	
Final R indices [I > 2σ(I)]	R1 = 0.0327, wR2 = 0.0846	
R indices (all data)	R1 = 0.0648, wR2 = 0.1289	
Largest diff. peak and hole	1.909 and -2.418 e.Å <sup>-3</sup>	

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1021.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

	x	y	z	$U(\text{eq})$
Ta(1)	1230(1)	1666(1)	3726(1)	24(1)
Cl(1)	-115(2)	3063(1)	3720(1)	36(1)
N(1)	2102(5)	721(3)	3199(2)	30(1)
C(1)	1629(6)	1149(4)	2767(2)	29(1)
Cl(2)	3137(2)	2602(1)	3840(1)	38(1)
N(2)	1235(5)	1991(4)	2911(2)	29(1)
C(2)	567(6)	2561(4)	2521(2)	36(2)
Cl(3)	-738(2)	777(1)	3617(1)	49(1)
C(3)	1096(7)	2357(6)	1970(3)	41(2)
N(3)	1482(6)	783(4)	2287(2)	38(1)
C(4)	941(7)	1335(6)	1848(3)	43(2)
C(5)	1757(8)	-193(5)	2212(3)	47(2)
C(6)	1586(8)	-689(5)	2732(3)	52(2)
C(7)	2312(8)	-272(5)	3172(3)	45(2)
C(8)	1636(7)	1135(5)	4696(3)	33(2)
N(4)	2141(6)	744(4)	4257(2)	33(1)
N(5)	746(6)	1772(4)	4542(2)	34(1)
C(9)	82(6)	2305(5)	4947(2)	32(1)
N(6)	1980(5)	962(4)	5189(2)	33(1)
C(10)	999(6)	2433(5)	5419(3)	34(2)
C(11)	1449(7)	1508(5)	5626(3)	37(2)
C(12)	2917(7)	229(5)	5318(2)	36(2)
C(13)	3242(7)	-323(5)	4831(3)	37(2)
C(14)	3384(6)	263(5)	4342(3)	36(2)

Bond lengths [Å] and angles [°] for mes1021.

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Ta(1)-N(2)	2.109(5)
Ta(1)-N(4)	2.114(5)
Ta(1)-N(1)	2.115(5)
Ta(1)-N(5)	2.119(5)
Ta(1)-Cl(2)	2.3953(17)
Ta(1)-Cl(3)	2.4067(19)
Ta(1)-Cl(1)	2.4631(17)
Ta(1)-C(1)	2.566(6)
Ta(1)-C(8)	2.598(6)
N(1)-C(1)	1.345(8)
N(1)-C(7)	1.470(8)
C(1)-N(3)	1.333(8)
C(1)-N(2)	1.347(8)
N(2)-C(2)	1.458(7)
C(2)-C(3)	1.520(9)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.535(10)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
N(3)-C(5)	1.468(9)
N(3)-C(4)	1.475(9)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.510(10)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.467(10)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-N(6)	1.315(8)
C(8)-N(4)	1.348(8)

C(8)-N(5)	1.357(8)
N(4)-C(14)	1.466(8)
N(5)-C(9)	1.453(8)
C(9)-C(10)	1.523(8)
C(9)-H(15A)	0.9900
C(9)-H(15B)	0.9900
N(6)-C(11)	1.466(8)
N(6)-C(12)	1.472(8)
C(10)-C(11)	1.523(9)
C(10)-H(13A)	0.9900
C(10)-H(13B)	0.9900
C(11)-H(12A)	0.9900
C(11)-H(12B)	0.9900
C(12)-C(13)	1.506(9)
C(12)-H(11A)	0.9900
C(12)-H(11B)	0.9900
C(13)-C(14)	1.510(9)
C(13)-H(10A)	0.9900
C(13)-H(10B)	0.9900
C(14)-H(9A)	0.9900
C(14)-H(9B)	0.9900
N(2)-Ta(1)-N(4)	139.4(2)
N(2)-Ta(1)-N(1)	62.2(2)
N(4)-Ta(1)-N(1)	78.2(2)
N(2)-Ta(1)-N(5)	158.2(2)
N(4)-Ta(1)-N(5)	62.3(2)
N(1)-Ta(1)-N(5)	139.1(2)
N(2)-Ta(1)-Cl(2)	89.14(15)
N(4)-Ta(1)-Cl(2)	86.19(16)
N(1)-Ta(1)-Cl(2)	96.23(16)
N(5)-Ta(1)-Cl(2)	91.78(18)
N(2)-Ta(1)-Cl(3)	90.69(15)
N(4)-Ta(1)-Cl(3)	95.38(17)
N(1)-Ta(1)-Cl(3)	85.65(16)
N(5)-Ta(1)-Cl(3)	87.55(18)

Cl(2)-Ta(1)-Cl(3)	177.77(6)
N(2)-Ta(1)-Cl(1)	78.85(15)
N(4)-Ta(1)-Cl(1)	141.15(14)
N(1)-Ta(1)-Cl(1)	140.61(14)
N(5)-Ta(1)-Cl(1)	79.43(15)
Cl(2)-Ta(1)-Cl(1)	88.66(6)
Cl(3)-Ta(1)-Cl(1)	89.13(7)
N(2)-Ta(1)-C(1)	31.6(2)
N(4)-Ta(1)-C(1)	109.8(2)
N(1)-Ta(1)-C(1)	31.6(2)
N(5)-Ta(1)-C(1)	166.5(2)
Cl(2)-Ta(1)-C(1)	98.79(15)
Cl(3)-Ta(1)-C(1)	82.19(15)
Cl(1)-Ta(1)-C(1)	109.05(15)
N(2)-Ta(1)-C(8)	169.6(2)
N(4)-Ta(1)-C(8)	31.1(2)
N(1)-Ta(1)-C(8)	109.2(2)
N(5)-Ta(1)-C(8)	31.4(2)
Cl(2)-Ta(1)-C(8)	85.96(16)
Cl(3)-Ta(1)-C(8)	94.55(16)
Cl(1)-Ta(1)-C(8)	110.10(15)
C(1)-Ta(1)-C(8)	140.7(2)
C(1)-N(1)-C(7)	118.4(5)
C(1)-N(1)-Ta(1)	93.1(4)
C(7)-N(1)-Ta(1)	137.4(4)
N(3)-C(1)-N(1)	126.0(6)
N(3)-C(1)-N(2)	125.4(6)
N(1)-C(1)-N(2)	108.4(5)
N(3)-C(1)-Ta(1)	163.1(5)
N(1)-C(1)-Ta(1)	55.4(3)
N(2)-C(1)-Ta(1)	55.1(3)
C(1)-N(2)-C(2)	118.8(5)
C(1)-N(2)-Ta(1)	93.3(4)
C(2)-N(2)-Ta(1)	141.7(4)
N(2)-C(2)-C(3)	109.7(5)
N(2)-C(2)-H(2A)	109.7

C(3)-C(2)-H(2A)	109.7
N(2)-C(2)-H(2B)	109.7
C(3)-C(2)-H(2B)	109.7
H(2A)-C(2)-H(2B)	108.2
C(2)-C(3)-C(4)	109.8(6)
C(2)-C(3)-H(3A)	109.7
C(4)-C(3)-H(3A)	109.7
C(2)-C(3)-H(3B)	109.7
C(4)-C(3)-H(3B)	109.7
H(3A)-C(3)-H(3B)	108.2
C(1)-N(3)-C(5)	119.1(6)
C(1)-N(3)-C(4)	120.1(6)
C(5)-N(3)-C(4)	120.6(6)
N(3)-C(4)-C(3)	110.2(6)
N(3)-C(4)-H(4A)	109.6
C(3)-C(4)-H(4A)	109.6
N(3)-C(4)-H(4B)	109.6
C(3)-C(4)-H(4B)	109.6
H(4A)-C(4)-H(4B)	108.1
N(3)-C(5)-C(6)	109.6(6)
N(3)-C(5)-H(5A)	109.8
C(6)-C(5)-H(5A)	109.8
N(3)-C(5)-H(5B)	109.8
C(6)-C(5)-H(5B)	109.8
H(5A)-C(5)-H(5B)	108.2
C(7)-C(6)-C(5)	113.5(7)
C(7)-C(6)-H(6A)	108.9
C(5)-C(6)-H(6A)	108.9
C(7)-C(6)-H(6B)	108.9
C(5)-C(6)-H(6B)	108.9
H(6A)-C(6)-H(6B)	107.7
C(6)-C(7)-N(1)	111.9(6)
C(6)-C(7)-H(7A)	109.2
N(1)-C(7)-H(7A)	109.2
C(6)-C(7)-H(7B)	109.2
N(1)-C(7)-H(7B)	109.2

H(7A)-C(7)-H(7B)	107.9
N(6)-C(8)-N(4)	126.2(6)
N(6)-C(8)-N(5)	125.6(6)
N(4)-C(8)-N(5)	108.1(5)
N(6)-C(8)-Ta(1)	171.4(5)
N(4)-C(8)-Ta(1)	54.2(3)
N(5)-C(8)-Ta(1)	54.4(3)
C(8)-N(4)-C(14)	114.5(5)
C(8)-N(4)-Ta(1)	94.6(4)
C(14)-N(4)-Ta(1)	141.1(4)
C(8)-N(5)-C(9)	118.5(5)
C(8)-N(5)-Ta(1)	94.2(4)
C(9)-N(5)-Ta(1)	146.1(4)
N(5)-C(9)-C(10)	109.2(5)
N(5)-C(9)-H(15A)	109.8
C(10)-C(9)-H(15A)	109.8
N(5)-C(9)-H(15B)	109.8
C(10)-C(9)-H(15B)	109.8
H(15A)-C(9)-H(15B)	108.3
C(8)-N(6)-C(11)	120.4(6)
C(8)-N(6)-C(12)	121.5(5)
C(11)-N(6)-C(12)	118.0(5)
C(9)-C(10)-C(11)	110.1(6)
C(9)-C(10)-H(13A)	109.6
C(11)-C(10)-H(13A)	109.6
C(9)-C(10)-H(13B)	109.6
C(11)-C(10)-H(13B)	109.6
H(13A)-C(10)-H(13B)	108.2
N(6)-C(11)-C(10)	109.7(6)
N(6)-C(11)-H(12A)	109.7
C(10)-C(11)-H(12A)	109.7
N(6)-C(11)-H(12B)	109.7
C(10)-C(11)-H(12B)	109.7
H(12A)-C(11)-H(12B)	108.2
N(6)-C(12)-C(13)	110.7(5)
N(6)-C(12)-H(11A)	109.5



C(13)-C(12)-H(11A)	109.5
N(6)-C(12)-H(11B)	109.5
C(13)-C(12)-H(11B)	109.5
H(11A)-C(12)-H(11B)	108.1
C(12)-C(13)-C(14)	112.4(5)
C(12)-C(13)-H(10A)	109.1
C(14)-C(13)-H(10A)	109.1
C(12)-C(13)-H(10B)	109.1
C(14)-C(13)-H(10B)	109.1
H(10A)-C(13)-H(10B)	107.9
N(4)-C(14)-C(13)	108.0(5)
N(4)-C(14)-H(9A)	110.1
C(13)-C(14)-H(9A)	110.1
N(4)-C(14)-H(9B)	110.1
C(13)-C(14)-H(9B)	110.1
H(9A)-C(14)-H(9B)	108.4

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Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for mes1021. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Ta(1)	29(1)	23(1)	20(1)	2(1)	0(1)	3(1)
Cl(1)	43(1)	35(1)	30(1)	4(1)	1(1)	16(1)
N(1)	40(3)	23(3)	27(3)	-1(2)	3(2)	3(2)
C(1)	31(3)	28(3)	27(3)	-5(3)	0(3)	-3(3)
Cl(2)	34(1)	37(1)	43(1)	-3(1)	-7(1)	-3(1)
N(2)	39(3)	27(3)	19(3)	6(2)	-4(2)	6(2)
C(2)	46(4)	36(4)	25(3)	2(3)	-3(3)	5(3)
Cl(3)	40(1)	49(1)	57(1)	-4(1)	7(1)	-15(1)
C(3)	44(4)	55(5)	23(3)	6(3)	-3(3)	12(3)
N(3)	50(3)	41(4)	23(3)	-4(3)	-4(2)	3(3)
C(4)	49(4)	54(5)	27(4)	-4(3)	-8(3)	3(4)
C(5)	54(4)	41(4)	45(4)	-20(3)	5(4)	-14(4)
C(6)	68(5)	30(4)	57(5)	-9(4)	-11(4)	1(4)
C(7)	69(5)	28(4)	39(4)	-6(3)	4(4)	5(4)
C(8)	44(4)	30(4)	27(3)	9(3)	6(3)	5(3)
N(4)	44(3)	32(3)	22(3)	4(2)	4(2)	10(2)
N(5)	46(3)	36(3)	21(3)	2(2)	1(2)	15(3)
C(9)	35(3)	35(3)	26(3)	-3(3)	3(3)	5(3)
N(6)	37(3)	40(3)	23(3)	7(2)	5(2)	8(3)
C(10)	32(3)	40(4)	28(3)	-2(3)	5(3)	0(3)
C(11)	41(4)	46(5)	24(3)	2(3)	3(3)	0(3)
C(12)	40(4)	40(4)	29(3)	13(3)	0(3)	6(3)
C(13)	38(4)	34(4)	40(4)	8(3)	1(3)	6(3)
C(14)	40(4)	41(4)	28(3)	4(3)	0(3)	13(3)

Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1021.

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	x	y	z	U(eq)
H(2A)	709	3214	2607	43
H(2B)	-388	2439	2532	43
H(3A)	2034	2528	1951	49
H(3B)	611	2721	1704	49
H(4A)	0	1188	1800	52
H(4B)	1407	1186	1515	52
H(5A)	2666	-274	2082	56
H(5B)	1151	-450	1944	56
H(6A)	641	-703	2823	62
H(6B)	1883	-1329	2689	62
H(7A)	3260	-397	3128	54
H(7B)	2026	-553	3511	54
H(15A)	-725	1985	5063	38
H(15B)	-171	2907	4802	38
H(13A)	537	2770	5704	40
H(13B)	1771	2797	5309	40
H(12A)	2131	1595	5901	44
H(12B)	699	1184	5790	44
H(11A)	2533	-175	5592	44
H(11B)	3730	500	5464	44
H(10A)	2540	-779	4771	45
H(10B)	4071	-659	4893	45
H(9A)	4105	708	4391	43
H(9B)	3591	-123	4030	43

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Torsion angles [°] for mes1021.

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N(2)-Ta(1)-N(1)-C(1)	-10.9(4)
N(4)-Ta(1)-N(1)-C(1)	178.6(4)
N(5)-Ta(1)-N(1)-C(1)	163.3(4)
Cl(2)-Ta(1)-N(1)-C(1)	-96.7(4)
Cl(3)-Ta(1)-N(1)-C(1)	82.2(4)
Cl(1)-Ta(1)-N(1)-C(1)	-1.1(5)
C(8)-Ta(1)-N(1)-C(1)	175.5(4)
N(2)-Ta(1)-N(1)-C(7)	-150.6(7)
N(4)-Ta(1)-N(1)-C(7)	38.9(7)
N(5)-Ta(1)-N(1)-C(7)	23.7(9)
Cl(2)-Ta(1)-N(1)-C(7)	123.7(7)
Cl(3)-Ta(1)-N(1)-C(7)	-57.5(7)
Cl(1)-Ta(1)-N(1)-C(7)	-140.8(6)
C(1)-Ta(1)-N(1)-C(7)	-139.7(9)
C(8)-Ta(1)-N(1)-C(7)	35.8(7)
C(7)-N(1)-C(1)-N(3)	-9.3(10)
Ta(1)-N(1)-C(1)-N(3)	-159.4(6)
C(7)-N(1)-C(1)-N(2)	166.2(6)
Ta(1)-N(1)-C(1)-N(2)	16.1(5)
C(7)-N(1)-C(1)-Ta(1)	150.1(7)
N(2)-Ta(1)-C(1)-N(3)	-97.9(18)
N(4)-Ta(1)-C(1)-N(3)	99.3(18)
N(1)-Ta(1)-C(1)-N(3)	100.8(19)
N(5)-Ta(1)-C(1)-N(3)	47(2)
Cl(2)-Ta(1)-C(1)-N(3)	-171.6(18)
Cl(3)-Ta(1)-C(1)-N(3)	6.3(18)
Cl(1)-Ta(1)-C(1)-N(3)	-80.0(18)
C(8)-Ta(1)-C(1)-N(3)	94.0(18)
N(2)-Ta(1)-C(1)-N(1)	161.3(6)
N(4)-Ta(1)-C(1)-N(1)	-1.5(4)
N(5)-Ta(1)-C(1)-N(1)	-53.4(10)
Cl(2)-Ta(1)-C(1)-N(1)	87.6(4)
Cl(3)-Ta(1)-C(1)-N(1)	-94.4(4)
Cl(1)-Ta(1)-C(1)-N(1)	179.2(3)

C(8)-Ta(1)-C(1)-N(1)	-6.8(6)
N(4)-Ta(1)-C(1)-N(2)	-162.8(4)
N(1)-Ta(1)-C(1)-N(2)	-161.3(6)
N(5)-Ta(1)-C(1)-N(2)	145.2(8)
Cl(2)-Ta(1)-C(1)-N(2)	-73.7(4)
Cl(3)-Ta(1)-C(1)-N(2)	104.3(4)
Cl(1)-Ta(1)-C(1)-N(2)	17.9(4)
C(8)-Ta(1)-C(1)-N(2)	-168.1(4)
N(3)-C(1)-N(2)-C(2)	1.2(10)
N(1)-C(1)-N(2)-C(2)	-174.2(5)
Ta(1)-C(1)-N(2)-C(2)	-158.1(6)
N(3)-C(1)-N(2)-Ta(1)	159.4(6)
N(1)-C(1)-N(2)-Ta(1)	-16.1(5)
N(4)-Ta(1)-N(2)-C(1)	25.3(5)
N(1)-Ta(1)-N(2)-C(1)	10.9(4)
N(5)-Ta(1)-N(2)-C(1)	-158.9(6)
Cl(2)-Ta(1)-N(2)-C(1)	108.4(3)
Cl(3)-Ta(1)-N(2)-C(1)	-73.8(4)
Cl(1)-Ta(1)-N(2)-C(1)	-162.8(4)
C(8)-Ta(1)-N(2)-C(1)	46.7(13)
N(4)-Ta(1)-N(2)-C(2)	173.5(6)
N(1)-Ta(1)-N(2)-C(2)	159.1(8)
N(5)-Ta(1)-N(2)-C(2)	-10.7(11)
Cl(2)-Ta(1)-N(2)-C(2)	-103.4(7)
Cl(3)-Ta(1)-N(2)-C(2)	74.4(7)
Cl(1)-Ta(1)-N(2)-C(2)	-14.6(7)
C(1)-Ta(1)-N(2)-C(2)	148.2(9)
C(8)-Ta(1)-N(2)-C(2)	-165.1(10)
C(1)-N(2)-C(2)-C(3)	-32.6(8)
Ta(1)-N(2)-C(2)-C(3)	-175.7(6)
N(2)-C(2)-C(3)-C(4)	56.5(7)
N(1)-C(1)-N(3)-C(5)	4.9(10)
N(2)-C(1)-N(3)-C(5)	-169.8(6)
Ta(1)-C(1)-N(3)-C(5)	-83.8(19)
N(1)-C(1)-N(3)-C(4)	179.7(6)
N(2)-C(1)-N(3)-C(4)	5.0(10)

Ta(1)-C(1)-N(3)-C(4)	91.0(18)
C(1)-N(3)-C(4)-C(3)	20.8(9)
C(5)-N(3)-C(4)-C(3)	-164.5(6)
C(2)-C(3)-C(4)-N(3)	-50.6(8)
C(1)-N(3)-C(5)-C(6)	25.1(9)
C(4)-N(3)-C(5)-C(6)	-149.7(7)
N(3)-C(5)-C(6)-C(7)	-51.4(9)
C(5)-C(6)-C(7)-N(1)	48.1(9)
C(1)-N(1)-C(7)-C(6)	-18.0(9)
Ta(1)-N(1)-C(7)-C(6)	114.7(7)
N(2)-Ta(1)-C(8)-N(6)	69(4)
N(4)-Ta(1)-C(8)-N(6)	96(3)
N(1)-Ta(1)-C(8)-N(6)	102(3)
N(5)-Ta(1)-C(8)-N(6)	-93(3)
Cl(2)-Ta(1)-C(8)-N(6)	7(3)
Cl(3)-Ta(1)-C(8)-N(6)	-171(3)
Cl(1)-Ta(1)-C(8)-N(6)	-80(3)
C(1)-Ta(1)-C(8)-N(6)	106(3)
N(2)-Ta(1)-C(8)-N(4)	-27.4(14)
N(1)-Ta(1)-C(8)-N(4)	5.9(4)
N(5)-Ta(1)-C(8)-N(4)	170.6(7)
Cl(2)-Ta(1)-C(8)-N(4)	-89.3(4)
Cl(3)-Ta(1)-C(8)-N(4)	92.9(4)
Cl(1)-Ta(1)-C(8)-N(4)	-176.4(4)
C(1)-Ta(1)-C(8)-N(4)	9.6(6)
N(2)-Ta(1)-C(8)-N(5)	162.0(11)
N(4)-Ta(1)-C(8)-N(5)	-170.6(7)
N(1)-Ta(1)-C(8)-N(5)	-164.7(4)
Cl(2)-Ta(1)-C(8)-N(5)	100.1(4)
Cl(3)-Ta(1)-C(8)-N(5)	-77.7(4)
Cl(1)-Ta(1)-C(8)-N(5)	13.0(5)
C(1)-Ta(1)-C(8)-N(5)	-161.0(4)
N(6)-C(8)-N(4)-C(14)	-15.8(10)
N(5)-C(8)-N(4)-C(14)	161.6(6)
Ta(1)-C(8)-N(4)-C(14)	153.6(6)
N(6)-C(8)-N(4)-Ta(1)	-169.3(6)

N(5)-C(8)-N(4)-Ta(1)	8.0(6)
N(2)-Ta(1)-N(4)-C(8)	172.7(4)
N(1)-Ta(1)-N(4)-C(8)	-174.3(4)
N(5)-Ta(1)-N(4)-C(8)	-5.5(4)
Cl(2)-Ta(1)-N(4)-C(8)	88.5(4)
Cl(3)-Ta(1)-N(4)-C(8)	-89.9(4)
Cl(1)-Ta(1)-N(4)-C(8)	5.4(6)
C(1)-Ta(1)-N(4)-C(8)	-173.5(4)
N(2)-Ta(1)-N(4)-C(14)	32.9(9)
N(1)-Ta(1)-N(4)-C(14)	45.9(7)
N(5)-Ta(1)-N(4)-C(14)	-145.3(8)
Cl(2)-Ta(1)-N(4)-C(14)	-51.3(7)
Cl(3)-Ta(1)-N(4)-C(14)	130.3(7)
Cl(1)-Ta(1)-N(4)-C(14)	-134.4(6)
C(1)-Ta(1)-N(4)-C(14)	46.7(8)
C(8)-Ta(1)-N(4)-C(14)	-139.8(10)
N(6)-C(8)-N(5)-C(9)	-1.3(11)
N(4)-C(8)-N(5)-C(9)	-178.7(5)
Ta(1)-C(8)-N(5)-C(9)	-170.7(7)
N(6)-C(8)-N(5)-Ta(1)	169.4(6)
N(4)-C(8)-N(5)-Ta(1)	-8.0(6)
N(2)-Ta(1)-N(5)-C(8)	-171.4(5)
N(4)-Ta(1)-N(5)-C(8)	5.5(4)
N(1)-Ta(1)-N(5)-C(8)	22.4(6)
Cl(2)-Ta(1)-N(5)-C(8)	-79.3(4)
Cl(3)-Ta(1)-N(5)-C(8)	102.9(4)
Cl(1)-Ta(1)-N(5)-C(8)	-167.6(5)
C(1)-Ta(1)-N(5)-C(8)	62.3(11)
N(2)-Ta(1)-N(5)-C(9)	-6.2(13)
N(4)-Ta(1)-N(5)-C(9)	170.7(9)
N(1)-Ta(1)-N(5)-C(9)	-172.4(7)
Cl(2)-Ta(1)-N(5)-C(9)	85.9(8)
Cl(3)-Ta(1)-N(5)-C(9)	-91.9(8)
Cl(1)-Ta(1)-N(5)-C(9)	-2.4(8)
C(1)-Ta(1)-N(5)-C(9)	-132.5(9)
C(8)-Ta(1)-N(5)-C(9)	165.2(11)

C(8)-N(5)-C(9)-C(10)	31.6(9)
Ta(1)-N(5)-C(9)-C(10)	-131.5(7)
N(4)-C(8)-N(6)-C(11)	173.4(7)
N(5)-C(8)-N(6)-C(11)	-3.5(11)
Ta(1)-C(8)-N(6)-C(11)	84(3)
N(4)-C(8)-N(6)-C(12)	-5.7(11)
N(5)-C(8)-N(6)-C(12)	177.4(7)
Ta(1)-C(8)-N(6)-C(12)	-95(3)
N(5)-C(9)-C(10)-C(11)	-56.6(7)
C(8)-N(6)-C(11)-C(10)	-22.9(8)
C(12)-N(6)-C(11)-C(10)	156.3(5)
C(9)-C(10)-C(11)-N(6)	52.1(7)
C(8)-N(6)-C(12)-C(13)	-7.1(9)
C(11)-N(6)-C(12)-C(13)	173.7(6)
N(6)-C(12)-C(13)-C(14)	39.5(8)
C(8)-N(4)-C(14)-C(13)	46.6(8)
Ta(1)-N(4)-C(14)-C(13)	-178.4(5)
C(12)-C(13)-C(14)-N(4)	-59.3(8)

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Observed and calculated structure factors for Ta(hpp)<sub>2</sub>Cl<sub>3</sub>.

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
4	0	0	5218	5122	30	4	15	0	267	239	116	7	6	1	117	126	29	2	14	1	493	477	20	10	3	2	0	104	1
8	0	0	2155	2040	39	6	15	0	272	2	79	8	6	1	0	17	1	3	14	1	892	877	17	11	3	2	296	304	38
12	0	0	1130	1051	43	8	15	0	0	50	1	9	6	1	235	220	29	4	14	1	1094	1086	22	12	3	2	71	9	71
2	1	0	6645	6498	29	0	16	0	656	604	141	10	6	1	165	190	48	5	14	1	695	731	18	13	3	2	0	88	1
4	1	0	674	616	6	2	16	0	0	223	1	11	6	1	217	165	70	6	14	1	85	146	85	0	4	2	213	240	11
6	1	0	4073	3930	21	4	16	0	919	914	50	12	6	1	120	177	119	7	14	1	818	849	21	1	4	2	664	688	4
8	1	0	496	508	11	6	16	0	283	268	89	1	7	1	663	657	6	8	14	1	1088	1041	21	2	4	2	163	178	12
10	1	0	2056	1936	31	2	17	0	1664	1578	30	2	7	1	1063	1077	7	9	14	1	424	453	47	3	4	2	1727	1708	7
12	1	0	210	83	81	4	17	0	0	56	1	3	7	1	295	336	12	1	15	1	1275	1260	20	4	4	2	134	122	15
0	2	0	295	3290	97	6	17	0	892	957	41	4	7	1	938	936	8	2	15	1	1393	1351	14	5	4	2	876	883	8
2	2	0	3120	3003	12	0	18	0	866	927	40	5	7	1	1046	1038	6	3	15	1	769	778	18	6	4	2	201	217	18
4	2	0	2003	1961	11	2	18	0	0	5	1	6	7	1	1047	1047	8	4	15	1	132	10	132	7	4	2	1487	1464	9
6	2	0	281	322	9	4	18	0	1505	1425	51	7	7	1	1166	1139	9	5	15	1	659	703	24	8	4	2	80	90	79
8	2	0	1735	1704	12	2	19	0	1137	1135	30	8	7	1	383	385	20	6	15	1	1060	1048	20	9	4	2	1057	1050	10
10	2	0	576	564	19	1	1	1	0	1854	1	9	7	1	591	597	14	7	15	1	739	733	22	10	4	2	145	169	61
12	2	0	608	675	36	2	1	1	3276	3227	8	10	7	1	196	262	59	8	15	1	115	179	115	11	4	2	741	757	19
2	3	0	109	10	20	3	1	1	1396	1359	5	11	7	1	324	396	55	0	16	1	965	949	21	12	4	2	0	122	1
4	3	0	2810	2684	15	4	1	1	1328	1315	5	12	7	1	141	45	140	1	16	1	632	650	17	13	4	2	365	338	55
6	3	0	726	748	10	5	1	1	1266	1243	8	0	8	1	1528	1643	22	2	16	1	118	112	117	1	5	2	3059	3025	11
8	3	0	354	371	17	6	1	1	1252	1229	6	1	8	1	1717	1734	10	3	16	1	1068	1022	17	2	5	2	40	13	40
10	3	0	0	69	1	7	1	1	78	108	78	2	8	1	431	415	8	4	16	1	924	925	20	3	5	2	3019	2993	12
12	3	0	365	348	64	8	1	1	72	42	71	3	8	1	1994	1960	11	5	16	1	551	584	25	4	5	2	113	10	28
0	4	0	883	924	7	9	1	1	522	582	16	4	8	1	1803	1795	11	6	16	1	0	134	1	5	5	2	1859	1802	8
2	4	0	419	402	9	10	1	1	977	940	12	5	8	1	1313	1295	9	7	16	1	712	739	31	6	5	2	93	99	33
4	4	0	2634	2577	16	11	1	1	423	408	30	6	8	1	90	70	89	1	17	1	614	606	21	7	5	2	1939	1887	9
6	4	0	625	659	9	12	1	1	189	168	75	7	8	1	1254	1222	12	2	17	1	716	710	21	8	5	2	143	73	34
8	4	0	1547	1467	14	13	1	1	173	270	173	8	8	1	1310	1284	11	3	17	1	547	530	29	9	5	2	1509	1466	11
10	4	0	163	127	69	0	2	1	0	4862	1	9	8	1	458	487	25	4	17	1	245	188	65	10	5	2	112	13	112
12	4	0	573	587	38	1	2	1	3632	3574	14	10	8	1	126	196	125	5	17	1	178	120	117	11	5	2	448	505	26
2	5	0	5265	5194	62	2	2	1	51	43	50	11	8	1	477	510	35	6	17	1	379	496	52	12	5	2	237	138	93
4	5	0	210	176	77	3	2	1	1676	1635	6	12	8	1	762	762	35	0	18	1	88	32	87	0	6	2	418	429	10
6	5	0	2619	2504	15	4	2	1	3822	3714	12	1	9	1	1756	1748	10	1	18	1	0	54	1	1	6	2	2446	2465	9
8	5	0	146	104	51	5	2	1	1891	1853	7	2	9	1	2191	2178	11	2	18	1	0	18	1	2	6	2	167	175	13
10	5	0	1113	1101	25	6	2	1	780	791	6	3	9	1	1373	1377	13	3	18	1	47	69	46	3	6	2	4478	4337	22
12	5	0	174	166	174	7	2	1	1171	1155	7	4	9	1	107	157	67	4	18	1	147	30	147	4	6	2	297	315	7
0	6	0	2117	2158	18	8	2	1	1431	1398	9	5	9	1	1898	1831	11	1	19	1	145	302	145	5	6	2	1777	1749	8
2	6	0	728	715	13	9	2	1	1081	1048	14	6	9	1	1991	1896	12	2	19	1	409	419	52	6	6	2	20	39	20
4	6	0	3430	3343	25	10	2	1	126	78	72	7	9	1	833	815	11	0	0	2	0	256	1	7	6	2	2802	2673	14
6	6	0	105	30	38	11	2	1	987	975	15	8	9	1	252	300	26	1	0	2	52	5194	52	8	6	2	183	210	29
8	6	0	3396	3169	23	12	2	1	847	807	22	9	9	1	859	895	16	2	0	2	349	328	6	9	6	2	1326	1258	15
10	6	0	186	56	87	13	2	1	422	417	45	10	9	1	1212	1179	36	3	0	2	1244	1242	6	10	6	2	265	280	34
12	6	0	1348	1222	43	1	3	1	3259	3207	8	11	9	1	816	748	25	4	0	2	883	868	7	11	6	2	890	898	25
2	7	0	3099	3138	28	2	3	1	4303	4209	13	0	10	1	2853	2776	19	5	0	2	3654	3624	18	12	6	2	123	49	123
4	7	0	133	141	97	3	3	1	3945	3907	13	1	10	1	1629	1626	11	6	0	2	254	254	8	1	7	2	3623	3598	14
6	7	0	2696	2596	21	4	3	1	657	601	6	2	10	1	382	372	13	7	0	2	1975	1909	12	2	7	2	116	106	19
8	7	0	314	335	44	5	3	1	2169	2116	8	3	10	1	971	963	12	8	0	2	291	274	31	3	7	2	2293	2262	13
10	7	0	1153	1132	34	6	3	1	2686	2601	18	4	10	1	1745	1703	15	9	0	2	1263	1223	15	4	7	2	89	96	27
12	7	0	218	188	103	7	3	1	1174	1184	13	5	10	1	1163	1119	12	10	0	2	253	256	42	5	7	2	1436	1402	7
0	8	0	2316	2295	22	8	3	1	0	66	1	6	10	1	407	399	19	11	0	2	1417	1404	19	6	7	2	121	135	28
2	8	0	926	938	12	9	3	1	931	932	14	7	10	1	945	930	14	12	0	2	88	79	88	7	7	2	1652	1602	10
4	8	0	1175	1161	13	10	3	1	1451	1413	14	8	10	1	781	766	21	13	0	2	647	702	39	8	7	2	108	133	107
6	8	0	272	276	21	11	3	1	704	690	16	9	10	1	508	544	20	1	1	2	82	4221	47	9	7	2	1524	1459	13
8	8	0	1491	1422	15	12	3	1	250	259	52	10	10	1	0	81	1	2	1	2	301	278	4	10	7	2	236	211	37
10	8	0	0	24	1	13	3	1	607	654	37	11	10	1	661	697	28	3	1	2	823	857	4	11	7	2	464	473	33
12	8	0	553	578	48	0	4	1	1809	1790	8	1	11	1	675	679	10	4	1	2	159	146	9	12	7	2	0	63	1
2	9	0	0	27	1	1	4	1	1873	1868	6	2	11	1	857	869	10	5	1	2	2668	2585	9	0	8	2	381	355	15
4	9	0	1701	1657	16	2	4	1	699	698	6	3																	

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
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8	3	20	0	30	1	4	13	20	207	75	102	8	8	21	1285	1275	23	8	4	22	0	53	1	6	2	23	96	72	96
9	3	20	157	89	90	5	13	20	0	191	1	1	9	21	531	527	23	9	4	22	423	378	35	7	2	23	1025	1018	27
10	3	20	0	14	1	0	14	20	756	719	40	2	9	21	1958	1882	24	1	5	22	1729	1691	13	8	2	23	619	632	25
0	4	20	1644	1646	16	1	14	20	186	22	144	3	9	21	469	481	23	2	5	22	688	692	15	9	2	23	719	679	33
1	4	20	814	808	11	2	14	20	0	14	1	4	9	21	125	21	124	3	5	22	1213	1203	13	1	3	23	1433	1386	12
2	4	20	114	162	74	3	14	20	157	267	157	5	9	21	651	639	21	4	5	22	554	529	14	2	3	23	1110	1094	12
3	4	20	167	149	34	4	14	20	602	626	53	6	9	21	1547	1503	26	5	5	22	1247	1246	15	3	3	23	1577	1521	13
4	4	20	1345	1323	11	1	15	20	0	81	1	7	9	21	511	533	30	6	5	22	457	492	23	4	3	23	91	72	90
5	4	20	544	563	17	2	15	20	239	13	120	8	9	21	316	246	51	7	5	22	1072	1026	20	5	3	23	1817	1745	16
6	4	20	213	167	33	1	1	21	240	269	22	0	10	21	1813	1769	35	8	5	22	112	150	111	6	3	23	862	873	18
7	4	20	142	92	142	2	1	21	1827	1804	12	1	10	21	594	637	22	9	5	22	732	782	36	7	3	23	783	759	24
8	4	20	925	916	18	3	1	21	568	585	13	2	10	21	115	123	115	0	6	22	856	807	22	8	3	23	0	36	1
9	4	20	0	53	1	4	1	21	589	587	13	3	10	21	552	542	27	1	6	22	1250	1204	14	9	3	23	869	865	25

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
4	6	23	213	242	41	1	4	24	368	359	19	7	3	25	298	213	84	3	3	26	93	113	93	3	5	27	907	898	18
5	6	23	0	40	1	2	4	24	159	101	54	8	3	25	182	121	182	4	3	26	104	56	104	4	5	27	137	43	136
6	6	23	0	108	1	3	4	24	162	199	74	0	4	25	1827	1742	32	5	3	26	245	268	50	5	5	27	644	714	24
7	6	23	0	137	1	4	4	24	807	805	19	1	4	25	449	444	22	6	3	26	232	107	91	6	5	27	236	224	79
8	6	23	256	99	92	5	4	24	348	342	26	2	4	25	315	314	25	7	3	26	143	42	142	0	6	27	0	94	1
1	7	23	686	685	25	6	4	24	139	111	138	3	4	25	422	456	23	8	3	26	0	45	1	1	6	27	0	45	1
2	7	23	100	218	100	7	4	24	187	48	99	4	4	25	1962	1898	25	0	4	26	661	660	24	2	6	27	172	25	79
3	7	23	465	461	20	8	4	24	680	665	32	5	4	25	417	447	46	1	4	26	590	633	17	3	6	27	0	7	1
4	7	23	41	113	41	1	5	24	205	253	38	6	4	25	97	172	97	2	4	26	127	150	102	4	6	27	0	18	1
5	7	23	355	410	40	2	5	24	1590	1564	14	7	4	25	405	383	33	3	4	26	861	862	16	5	6	27	137	13	136
6	7	23	407	417	33	3	5	24	185	228	42	8	4	25	1001	980	27	4	4	26	362	388	34	6	6	27	172	91	171
7	7	23	397	436	41	4	5	24	157	32	87	1	5	25	496	520	24	5	4	26	316	397	39	1	7	27	872	847	21
8	7	23	110	78	110	5	5	24	534	513	19	2	5	25	1284	1273	18	6	4	26	0	10	1	2	7	27	0	133	1
0	8	23	1000	1006	30	6	5	24	1348	1323	19	3	5	25	569	580	21	7	4	26	756	720	27	3	7	27	432	460	36
1	8	23	1059	1058	19	7	5	24	377	335	47	4	5	25	100	71	100	1	5	26	1384	1339	16	4	7	27	0	40	1
2	8	23	28	38	27	8	5	24	176	102	174	5	5	25	119	229	119	2	5	26	608	642	20	5	7	27	327	359	77
3	8	23	1036	1046	18	0	6	24	2300	2218	27	6	5	25	703	743	27	3	5	26	1064	1059	25	6	7	27	334	298	62
4	8	23	650	672	31	1	6	24	865	833	21	7	5	25	153	84	153	4	5	26	301	290	70	0	8	27	729	704	35
5	8	23	789	821	21	2	6	24	145	132	67	8	5	25	146	160	146	5	5	26	1110	1103	19	1	8	27	819	858	24
6	8	23	190	13	91	3	6	24	488	509	19	0	6	25	158	17	157	6	5	26	725	719	24	2	8	27	0	62	1
7	8	23	864	887	23	4	6	24	1773	1710	17	1	6	25	59	126	58	7	5	26	533	579	50	3	8	27	949	962	24
1	9	23	1236	1254	20	5	6	24	250	297	56	2	6	25	253	277	53	0	6	26	1031	1056	24	4	8	27	396	489	45
2	9	23	715	706	32	6	6	24	0	65	1	3	6	25	0	11	1	1	6	26	1531	1478	26	5	8	27	585	679	41
3	9	23	1235	1192	19	7	6	24	290	250	86	4	6	25	133	200	133	2	6	26	134	168	134	1	9	27	1119	1112	25
4	9	23	247	226	50	8	6	24	1267	1271	27	5	6	25	0	15	1	3	6	26	1224	1207	38	2	9	27	685	672	28
5	9	23	1020	1036	24	1	7	24	450	451	28	6	6	25	0	71	1	4	6	26	734	758	21	3	9	27	929	927	44
6	9	23	658	657	27	2	7	24	1795	1779	20	7	6	25	101	26	101	5	6	26	1102	1062	22	4	9	27	0	64	1
7	9	23	610	610	46	3	7	24	109	202	109	1	7	25	375	386	30	6	6	26	271	266	54	0	10	27	450	464	64
0	10	23	590	665	45	4	7	24	139	184	102	2	7	25	981	985	20	7	6	26	974	998	27	1	10	27	847	825	29
1	10	23	900	923	24	5	7	24	443	426	30	3	7	25	124	152	123	1	7	26	1440	1423	22	2	10	27	148	107	147
2	10	23	290	116	45	6	7	24	1360	1319	22	4	7	25	0	66	1	2	7	26	560	618	28	3	10	27	879	891	34
3	10	23	879	899	27	7	7	24	385	401	59	5	7	25	288	332	45	3	7	26	1012	1018	26	1	11	27	335	382	73
4	10	23	598	590	31	0	8	24	1182	1142	33	6	7	25	710	691	39	4	7	26	0	110	1	0	0	28	1686	1714	37
5	10	23	794	802	42	1	8	24	328	345	35	7	7	25	0	28	1	5	7	26	895	892	29	1	0	28	216	283	67
6	10	23	71	45	71	2	8	24	113	163	113	0	8	25	1781	1699	33	6	7	26	629	677	31	2	0	28	270	261	63
1	11	23	293	370	54	3	8	24	139	84	117	1	8	25	545	557	26	0	8	26	634	615	63	3	0	28	661	654	25
2	11	23	415	446	38	4	8	24	655	669	22	2	8	25	0	31	1	1	8	26	626	597	26	4	0	28	1554	1529	27
3	11	23	576	643	41	5	8	24	271	350	53	3	8	25	233	231	72	2	8	26	0	89	1	5	0	28	585	581	36
4	11	23	0	185	1	6	8	24	225	111	81	4	8	25	1288	1288	21	3	8	26	659	689	27	6	0	28	44	201	43
5	11	23	596	595	29	7	8	24	164	43	163	5	8	25	202	315	202	4	8	26	316	249	57	7	0	28	618	659	42
0	12	23	184	272	183	1	9	24	0	66	1	6	8	25	52	117	52	5	8	26	520	576	33	1	1	28	753	728	17
1	12	23	0	75	1	2	9	24	0	67	1	1	9	25	403	413	46	6	8	26	37	71	36	2	1	28	1539	1473	20
2	12	23	264	97	67	3	9	24	323	361	37	2	9	25	1742	1687	24	1	9	26	98	147	98	3	1	28	520	522	20
3	12	23	174	72	174	4	9	24	0	36	1	3	9	25	501	458	33	2	9	26	266	239	55	4	1	28	0	16	1
4	12	23	209	133	163	5	9	24	181	88	101	4	9	25	134	33	133	3	9	26	192	113	104	5	1	28	341	342	35
1	13	23	723	706	34	6	9	24	195	208	195	5	9	25	474	525	39	4	9	26	0	51	1	6	1	28	1280	1259	20
2	13	23	0	280	1	0	10	24	650	674	39	6	9	25	1209	1249	25	5	9	26	0	101	1	7	1	28	241	281	83
3	13	23	0	131	1	1	10	24	209	202	136	0	10	25	1307	1271	57	0	10	26	0	71	1	0	2	28	1151	1122	22
0	0	24	2365	2293	28	2	10	24	0	122	1	1	10	25	615	559	30	1	10	26	455	501	45	1	2	28	185	169	62
1	0	24	365	335	37	3	10	24	363	329	42	2	10	25	126	97	126	2	10	26	101	47	100	2	2	28	88	39	87
2	0	24	828	777	22	4	10	24	701	756	26	3	10	25	583	573	33	3	10	26	444	535	37	3	2	28	332	309	32
3	0	24	498	517	27	5	10	24	55	134	55	4	10	25	1445	1418	36	4	10	26	226	339	114	4	2	28	623	633	40
4	0	24	1671	1638	20	6	10	24	0	248	1	5	10	25	342	390	53	1	11	26	755	745	30	5	2	28	0	82	1
5	0	24	428	455	69	1	11	24	419	427	39	1	11	25	0	109	1	2	11	26	439	466	67	6	2	28	0	14	1
6	0	24	205	128	103	2	11	24	1319	1309	27	2	11	25	773	782	46	3	11	26	849	864	40	7	2	28	391	445	47
7	0	24	749	740	37	3	11	24	441	456	35	3	11	25	233	230	233	1	1	27	615	621	17	1	3	28	135	51	134
8	0	24	1644	1603	29	4																							

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
3	3	24	220	225	33	7	2	25	519	544	28	3	2	26	596	608	16	2	4	27	0	81	1	5	6	28	341	421	55
4	3	24	177	75	46	8	2	25	1068	1014	25	4	2	26	229	254	47	3	4	27	1058	1077	16	1	7	28	293	306	43
5	3	24	399	386	27	1	3	25	606	586	15	5	2	26	628	624	23	4	4	27	661	649	20	2	7	28	1359	1328	21
6	3	24	0	58	1	2	3	25	2196	2121	14	6	2	26	209	14	65	5	4	27	798	814	22	3	7	28	134	111	134
7	3	24	0	94	1	3	3	25	465	476	18	7	2	26	483	513	34	6	4	27	204	187	73	4	7	28	0	12	1
5	7	28	272	286	150	4	3	29	30	64	30	1	9	29	330	339	78	1	5	30	992	1014	23	2	4	31	0	69	1
0	8	28	919	931	39	5	3	29	382	371	36	1	0	30	876	875	34	2	5	30	590	567	26	3	4	31	744	776	30
1	8	28	258	249	63	6	3	29	1171	1199	24	2	0	30	323	295	192	3	5	30	742	776	34	1	5	31	137	227	136
2	8	28	147	235	147	0	4	29	1183	1164	25	3	0	30	1149	1124	25	4	5	30	0	46	1	2	5	31	251	235	73
3	8	28	0	32	1	1	4	29	340	388	50	4	0	30	712	658	32	0	6	30	838	782	52	3	5	31	513	508	35
4	8	28	547	539	43	2	4	29	281	326	50	5	0	30	914	945	37	1	6	30	974	980	24	0	6	31	257	205	121
1	9	28	94	91	94	3	4	29	247	243	53	1	1	30	911	940	18	2	6	30	159	134	158	1	6	31	127	24	126
2	9	28	0	19	1	4	4	29	1382	1333	22	2	1	30	624	660	25	3	6	30	701	755	36	2	6	31	95	39	94
3	9	28	284	133	80	5	4	29	228	323	79	3	1	30	692	697	19	1	7	30	889	935	31	0	0	32	1590	1541	39
0	10	28	467	506	145	1	5	29	191	239	74	4	1	30	166	216	113	2	7	30	325	342	56	1	0	32	282	265	81
1	10	28	276	136	62	2	5	29	706	724	24	5	1	30	643	667	32	0	8	30	329	457	190	2	0	32	232	159	106
1	1	29	126	82	126	3	5	29	229	244	64	0	2	30	200	278	91	1	8	30	368	350	53	3	0	32	464	384	58
2	1	29	724	751	23	4	5	29	140	73	140	1	2	30	520	532	24	1	1	31	359	404	32	1	1	32	503	531	27
3	1	29	228	247	50	5	5	29	120	265	119	2	2	30	86	129	85	2	1	31	338	333	73	2	1	32	1194	1169	21
4	1	29	73	127	73	0	6	29	252	122	96	3	2	30	349	345	38	3	1	31	444	462	33	3	1	32	328	298	57
5	1	29	0	87	1	1	6	29	219	171	85	4	2	30	0	190	1	4	1	31	0	78	1	0	2	32	763	759	33
6	1	29	718	736	31	2	6	29	216	221	71	5	2	30	493	492	37	0	2	31	89	261	89	1	2	32	0	67	1
0	2	29	1165	1151	25	3	6	29	132	102	132	1	3	30	112	138	111	1	2	31	940	931	20	2	2	32	230	230	74
1	2	29	277	296	41	4	6	29	126	198	125	2	3	30	75	189	74	2	2	31	199	15	71	3	2	32	70	139	69
2	2	29	90	88	90	1	7	29	259	310	71	3	3	30	0	41	1	3	2	31	872	942	25	1	3	32	0	5	1
3	2	29	434	424	39	2	7	29	583	646	31	4	3	30	0	206	1	4	2	31	471	447	39	2	3	32	131	64	131
4	2	29	1495	1457	39	3	7	29	0	114	1	5	3	30	0	65	1	1	3	31	893	901	22	0	4	32	632	630	49
5	2	29	134	159	133	4	7	29	78	124	77	0	4	30	509	448	38	2	3	31	468	482	36	1	4	32	470	482	33
6	2	29	0	120	1	0	8	29	1275	1266	35	1	4	30	542	570	49	3	3	31	1204	1223	24	2	4	32	121	19	121
1	3	29	305	357	35	1	8	29	276	263	57	2	4	30	101	111	101	4	3	31	208	15	103	1	1	33	180	84	104
2	3	29	1659	1571	21	2	8	29	195	44	132	3	4	30	528	530	30	0	4	31	494	500	41						
3	3	29	423	419	35	3	8	29	308	309	62	4	4	30	261	367	80	1	4	31	924	900	29						

## APPENDIX E

CRYSTALLOGRAPHIC DATA FOR  $(C_5Me_5)Ta(hpp)Cl_3$

Identification code	mes109	
Empirical formula	C17 H27 N3 Cl3 Ta1	
Formula weight	560.72	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 29.858(3) Å	$\alpha = 90^\circ$ .
	b = 8.7229(10) Å	$\beta = 98.529(5)^\circ$ .
	c = 30.672(4) Å	$\gamma = 90^\circ$ .
Volume	7900.1(16) Å <sup>3</sup>	
Z	16	
Density (calculated)	1.886 Mg/m <sup>3</sup>	
Absorption coefficient	5.976 mm <sup>-1</sup>	
F(000)	4384	
Crystal size	0.16 x 0.16 x 0.10 mm <sup>3</sup>	
Theta range for data collection	1.34 to 27.88°.	
Index ranges	-39 ≤ h ≤ 39, -11 ≤ k ≤ 11, -40 ≤ l ≤ 40	
Reflections collected	33458	
Independent reflections	9421 [R(int) = 0.0420]	
Completeness to theta = 27.88°	99.8 %	
Max. and min. transmission	0.9290 and 0.8898	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	9421 / 0 / 413	
Goodness-of-fit on F <sup>2</sup>	1.155	
Final R indices [I > 2σ(I)]	R1 = 0.0420, wR2 = 0.1057	
R indices (all data)	R1 = 0.0606, wR2 = 0.1322	
Extinction coefficient	0.00061(3)	
Largest diff. peak and hole	4.367 and -2.457 e.Å <sup>-3</sup>	

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes109.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^j$  tensor.

	x	y	z	$U(\text{eq})$
Ta(1)	1303(1)	8189(1)	1635(1)	20(1)
Cl(1)	1751(1)	9389(2)	1124(1)	40(1)
N(3)	463(2)	6862(6)	606(2)	28(1)
C(1)	769(2)	7362(7)	938(2)	26(1)
Ta(2)	1145(1)	2974(1)	3970(1)	21(1)
Cl(2)	879(1)	6084(2)	1920(1)	41(1)
N(2)	1162(2)	6723(6)	1067(2)	26(1)
C(7)	1282(2)	5295(8)	877(2)	34(2)
Cl(3)	1948(1)	6492(2)	1813(1)	43(1)
N(1)	716(2)	8607(6)	1199(2)	29(1)
C(6)	853(3)	4422(8)	702(2)	41(2)
Cl(4)	1491(1)	4379(2)	4641(1)	35(1)
N(23)	1906(2)	-61(7)	4662(2)	37(1)
C(5)	544(2)	5425(8)	393(2)	34(2)
Cl(5)	425(1)	3400(2)	4227(1)	34(1)
N(22)	1728(2)	1632(6)	4050(2)	29(1)
C(4)	47(2)	7703(9)	455(2)	37(2)
Cl(6)	761(1)	829(2)	3549(1)	33(1)
N(21)	1208(2)	1229(6)	4471(2)	27(1)
C(3)	65(3)	9288(10)	662(2)	44(2)
C(2)	259(2)	9262(9)	1151(2)	38(2)
C(8)	954(2)	9609(7)	2186(2)	24(1)
C(9)	1127(3)	10749(7)	1918(2)	34(2)
C(10)	1596(3)	10666(9)	1988(3)	43(2)
C(11)	1732(2)	9500(10)	2295(3)	44(2)
C(12)	1336(2)	8845(7)	2423(2)	32(2)
C(13)	479(2)	9398(10)	2271(3)	46(2)
C(14)	863(3)	12026(10)	1663(3)	61(3)
C(15)	1897(4)	11895(12)	1813(4)	72(3)
C(16')	2236(5)	9404(18)	2437(5)	36(3)
C(16)	2194(5)	8900(20)	2567(5)	41(4)

C(17)	1326(4)	7761(10)	2786(2)	60(3)
C(21)	1623(2)	862(7)	4409(2)	27(1)
C(22)	2170(3)	1336(11)	3916(3)	54(2)
C(23)	2517(4)	1013(15)	4303(4)	89(3)
C(24)	2370(3)	-318(12)	4575(3)	62(3)
C(25)	1765(3)	-813(9)	5048(2)	44(2)
C(26)	1264(3)	-658(8)	5055(2)	38(2)
C(27)	1088(2)	931(8)	4905(2)	32(1)
C(28)	1570(2)	4845(8)	3605(2)	30(1)
C(29)	1190(3)	5655(8)	3720(2)	37(2)
C(30)	794(2)	5087(8)	3470(2)	36(2)
C(31)	922(2)	3936(8)	3189(2)	32(1)
C(32)	1394(2)	3764(8)	3273(2)	30(1)
C(33)	2053(3)	5254(10)	3758(3)	54(2)
C(34)	1208(3)	7034(10)	4015(3)	60(2)
C(35)	327(3)	5723(13)	3441(3)	74(3)
C(36)	611(3)	3237(10)	2809(3)	51(2)
C(37)	1651(3)	2803(10)	2982(3)	50(2)

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Bond lengths [Å] and angles [°] for mes109.

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Ta(1)-N(1)	2.074(5)
Ta(1)-N(2)	2.153(5)
Ta(1)-Cl(3)	2.4257(16)
Ta(1)-Cl(1)	2.4429(17)
Ta(1)-C(8)	2.447(6)
Ta(1)-Cl(2)	2.4628(17)
Ta(1)-C(12)	2.470(6)
Ta(1)-C(9)	2.480(6)
Ta(1)-C(11)	2.505(7)
Ta(1)-C(10)	2.514(7)
Ta(1)-C(1)	2.574(6)
N(3)-C(1)	1.338(8)



N(3)-C(5)	1.450(8)
N(3)-C(4)	1.458(8)
C(1)-N(2)	1.305(8)
C(1)-N(1)	1.372(8)
Ta(2)-N(22)	2.082(5)
Ta(2)-N(21)	2.151(5)
Ta(2)-Cl(5)	2.4236(15)
Ta(2)-C(28)	2.441(6)
Ta(2)-Cl(6)	2.4595(16)
Ta(2)-C(32)	2.465(6)
Ta(2)-C(29)	2.472(7)
Ta(2)-Cl(4)	2.4841(16)
Ta(2)-C(30)	2.524(6)
Ta(2)-C(31)	2.533(6)
Ta(2)-C(21)	2.584(6)
N(2)-C(7)	1.443(8)
C(7)-C(6)	1.516(10)
C(7)-H(2A)	0.9900
C(7)-H(2B)	0.9900
N(1)-C(2)	1.466(8)
C(6)-C(5)	1.502(10)
C(6)-H(3A)	0.9900
C(6)-H(3B)	0.9900
N(23)-C(21)	1.329(8)
N(23)-C(24)	1.466(9)
N(23)-C(25)	1.470(9)
C(5)-H(4A)	0.9900
C(5)-H(4B)	0.9900
N(22)-C(21)	1.367(8)
N(22)-C(22)	1.464(8)
C(4)-C(3)	1.518(11)
C(4)-H(5A)	0.9900
C(4)-H(5B)	0.9900
N(21)-C(21)	1.320(8)
N(21)-C(27)	1.452(8)
C(3)-C(2)	1.526(9)

C(3)-H(6A)	0.9900
C(3)-H(6B)	0.9900
C(2)-H(7A)	0.9900
C(2)-H(7B)	0.9900
C(8)-C(12)	1.424(9)
C(8)-C(9)	1.434(9)
C(8)-C(13)	1.492(9)
C(9)-C(10)	1.386(11)
C(9)-C(14)	1.514(11)
C(10)-C(11)	1.404(12)
C(10)-C(15)	1.545(12)
C(11)-C(12)	1.419(10)
C(11)-C(16')	1.506(16)
C(11)-C(16)	1.590(17)
C(12)-C(17)	1.464(10)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16')-C(16)	0.617(19)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(22)-C(23)	1.482(14)
C(22)-H(19A)	0.9900
C(22)-H(19B)	0.9900
C(23)-C(24)	1.531(14)
C(23)-H(20A)	0.9900
C(23)-H(20B)	0.9900
C(24)-H(21A)	0.9900
C(24)-H(21B)	0.9900

C(25)-C(26)	1.506(11)
C(25)-H(22A)	0.9900
C(25)-H(22B)	0.9900
C(26)-C(27)	1.529(9)
C(26)-H(23A)	0.9900
C(26)-H(23B)	0.9900
C(27)-H(24A)	0.9900
C(27)-H(24B)	0.9900
C(28)-C(29)	1.424(10)
C(28)-C(32)	1.428(9)
C(28)-C(33)	1.492(10)
C(29)-C(30)	1.402(11)
C(29)-C(34)	1.502(11)
C(30)-C(31)	1.414(10)
C(30)-C(35)	1.489(11)
C(31)-C(32)	1.402(9)
C(31)-C(36)	1.509(10)
C(32)-C(37)	1.514(9)
C(33)-H(30A)	0.9800
C(33)-H(30B)	0.9800
C(33)-H(30C)	0.9800
C(34)-H(31A)	0.9800
C(34)-H(31B)	0.9800
C(34)-H(31C)	0.9800
C(35)-H(32A)	0.9800
C(35)-H(32B)	0.9800
C(35)-H(32C)	0.9800
C(36)-H(33A)	0.9800
C(36)-H(33B)	0.9800
C(36)-H(33C)	0.9800
C(37)-H(34A)	0.9800
C(37)-H(34B)	0.9800
C(37)-H(34C)	0.9800
N(1)-Ta(1)-N(2)	62.37(19)
N(1)-Ta(1)-Cl(3)	144.93(15)

N(2)-Ta(1)-Cl(3)	82.67(14)
N(1)-Ta(1)-Cl(1)	90.06(16)
N(2)-Ta(1)-Cl(1)	78.14(16)
Cl(3)-Ta(1)-Cl(1)	85.05(7)
N(1)-Ta(1)-C(8)	87.2(2)
N(2)-Ta(1)-C(8)	143.8(2)
Cl(3)-Ta(1)-C(8)	124.00(15)
Cl(1)-Ta(1)-C(8)	123.76(15)
N(1)-Ta(1)-Cl(2)	85.94(17)
N(2)-Ta(1)-Cl(2)	78.13(16)
Cl(3)-Ta(1)-Cl(2)	84.15(7)
Cl(1)-Ta(1)-Cl(2)	154.97(6)
C(8)-Ta(1)-Cl(2)	80.76(15)
N(1)-Ta(1)-C(12)	119.9(2)
N(2)-Ta(1)-C(12)	154.7(2)
Cl(3)-Ta(1)-C(12)	90.39(16)
Cl(1)-Ta(1)-C(12)	125.64(17)
C(8)-Ta(1)-C(12)	33.7(2)
Cl(2)-Ta(1)-C(12)	76.98(16)
N(1)-Ta(1)-C(9)	82.2(2)
N(2)-Ta(1)-C(9)	142.4(2)
Cl(3)-Ta(1)-C(9)	132.36(18)
Cl(1)-Ta(1)-C(9)	90.19(16)
C(8)-Ta(1)-C(9)	33.8(2)
Cl(2)-Ta(1)-C(9)	113.65(16)
C(12)-Ta(1)-C(9)	55.2(2)
N(1)-Ta(1)-C(11)	136.3(2)
N(2)-Ta(1)-C(11)	160.2(2)
Cl(3)-Ta(1)-C(11)	78.70(18)
Cl(1)-Ta(1)-C(11)	93.5(2)
C(8)-Ta(1)-C(11)	55.3(2)
Cl(2)-Ta(1)-C(11)	106.4(2)
C(12)-Ta(1)-C(11)	33.1(2)
C(9)-Ta(1)-C(11)	54.3(2)
N(1)-Ta(1)-C(10)	109.5(3)
N(2)-Ta(1)-C(10)	150.8(2)

Cl(3)-Ta(1)-C(10)	102.4(2)
Cl(1)-Ta(1)-C(10)	73.73(17)
C(8)-Ta(1)-C(10)	54.9(2)
Cl(2)-Ta(1)-C(10)	130.75(17)
C(12)-Ta(1)-C(10)	54.5(2)
C(9)-Ta(1)-C(10)	32.2(2)
C(11)-Ta(1)-C(10)	32.5(3)
N(1)-Ta(1)-C(1)	32.1(2)
N(2)-Ta(1)-C(1)	30.43(19)
Cl(3)-Ta(1)-C(1)	112.84(15)
Cl(1)-Ta(1)-C(1)	85.24(14)
C(8)-Ta(1)-C(1)	116.1(2)
Cl(2)-Ta(1)-C(1)	78.29(14)
C(12)-Ta(1)-C(1)	143.9(2)
C(9)-Ta(1)-C(1)	113.9(2)
C(11)-Ta(1)-C(1)	168.2(2)
C(10)-Ta(1)-C(1)	137.0(3)
C(1)-N(3)-C(5)	118.8(5)
C(1)-N(3)-C(4)	122.1(6)
C(5)-N(3)-C(4)	119.0(5)
N(2)-C(1)-N(3)	125.0(6)
N(2)-C(1)-N(1)	109.8(5)
N(3)-C(1)-N(1)	125.2(6)
N(2)-C(1)-Ta(1)	56.7(3)
N(3)-C(1)-Ta(1)	173.6(5)
N(1)-C(1)-Ta(1)	53.5(3)
N(22)-Ta(2)-N(21)	62.3(2)
N(22)-Ta(2)-Cl(5)	144.53(15)
N(21)-Ta(2)-Cl(5)	82.26(14)
N(22)-Ta(2)-C(28)	86.9(2)
N(21)-Ta(2)-C(28)	143.6(2)
Cl(5)-Ta(2)-C(28)	126.38(17)
N(22)-Ta(2)-Cl(6)	87.13(17)
N(21)-Ta(2)-Cl(6)	79.78(15)
Cl(5)-Ta(2)-Cl(6)	85.50(6)
C(28)-Ta(2)-Cl(6)	120.03(16)

N(22)-Ta(2)-C(32)	84.4(2)
N(21)-Ta(2)-C(32)	144.2(2)
Cl(5)-Ta(2)-C(32)	129.47(16)
C(28)-Ta(2)-C(32)	33.8(2)
Cl(6)-Ta(2)-C(32)	86.20(16)
N(22)-Ta(2)-C(29)	119.0(2)
N(21)-Ta(2)-C(29)	152.7(2)
Cl(5)-Ta(2)-C(29)	92.77(18)
C(28)-Ta(2)-C(29)	33.7(2)
Cl(6)-Ta(2)-C(29)	126.81(18)
C(32)-Ta(2)-C(29)	55.1(2)
N(22)-Ta(2)-Cl(4)	86.71(16)
N(21)-Ta(2)-Cl(4)	77.06(15)
Cl(5)-Ta(2)-Cl(4)	86.30(6)
C(28)-Ta(2)-Cl(4)	82.49(16)
Cl(6)-Ta(2)-Cl(4)	156.26(6)
C(32)-Ta(2)-Cl(4)	115.95(16)
C(29)-Ta(2)-Cl(4)	75.81(17)
N(22)-Ta(2)-C(30)	138.2(2)
N(21)-Ta(2)-C(30)	159.3(2)
Cl(5)-Ta(2)-C(30)	77.14(16)
C(28)-Ta(2)-C(30)	55.2(2)
Cl(6)-Ta(2)-C(30)	96.71(18)
C(32)-Ta(2)-C(30)	54.6(2)
C(29)-Ta(2)-C(30)	32.6(2)
Cl(4)-Ta(2)-C(30)	103.11(18)
N(22)-Ta(2)-C(31)	113.2(2)
N(21)-Ta(2)-C(31)	153.3(2)
Cl(5)-Ta(2)-C(31)	97.84(16)
C(28)-Ta(2)-C(31)	54.9(2)
Cl(6)-Ta(2)-C(31)	73.63(16)
C(32)-Ta(2)-C(31)	32.5(2)
C(29)-Ta(2)-C(31)	53.9(2)
Cl(4)-Ta(2)-C(31)	129.65(16)
C(30)-Ta(2)-C(31)	32.5(2)
N(22)-Ta(2)-C(21)	31.8(2)

N(21)-Ta(2)-C(21)	30.66(19)
Cl(5)-Ta(2)-C(21)	112.85(15)
C(28)-Ta(2)-C(21)	115.5(2)
Cl(6)-Ta(2)-C(21)	84.84(15)
C(32)-Ta(2)-C(21)	115.9(2)
C(29)-Ta(2)-C(21)	141.7(2)
Cl(4)-Ta(2)-C(21)	77.91(14)
C(30)-Ta(2)-C(21)	170.0(2)
C(31)-Ta(2)-C(21)	140.9(2)
C(1)-N(2)-C(7)	120.7(5)
C(1)-N(2)-Ta(1)	92.9(4)
C(7)-N(2)-Ta(1)	144.1(4)
N(2)-C(7)-C(6)	109.3(6)
N(2)-C(7)-H(2A)	109.8
C(6)-C(7)-H(2A)	109.8
N(2)-C(7)-H(2B)	109.8
C(6)-C(7)-H(2B)	109.8
H(2A)-C(7)-H(2B)	108.3
C(1)-N(1)-C(2)	115.9(5)
C(1)-N(1)-Ta(1)	94.4(3)
C(2)-N(1)-Ta(1)	144.8(4)
C(5)-C(6)-C(7)	109.7(6)
C(5)-C(6)-H(3A)	109.7
C(7)-C(6)-H(3A)	109.7
C(5)-C(6)-H(3B)	109.7
C(7)-C(6)-H(3B)	109.7
H(3A)-C(6)-H(3B)	108.2
C(21)-N(23)-C(24)	121.5(6)
C(21)-N(23)-C(25)	120.5(6)
C(24)-N(23)-C(25)	118.0(6)
N(3)-C(5)-C(6)	110.4(5)
N(3)-C(5)-H(4A)	109.6
C(6)-C(5)-H(4A)	109.6
N(3)-C(5)-H(4B)	109.6
C(6)-C(5)-H(4B)	109.6
H(4A)-C(5)-H(4B)	108.1

C(21)-N(22)-C(22)	117.6(5)
C(21)-N(22)-Ta(2)	94.8(4)
C(22)-N(22)-Ta(2)	147.2(5)
N(3)-C(4)-C(3)	110.5(5)
N(3)-C(4)-H(5A)	109.6
C(3)-C(4)-H(5A)	109.6
N(3)-C(4)-H(5B)	109.6
C(3)-C(4)-H(5B)	109.6
H(5A)-C(4)-H(5B)	108.1
C(21)-N(21)-C(27)	117.0(5)
C(21)-N(21)-Ta(2)	93.1(4)
C(27)-N(21)-Ta(2)	140.7(4)
C(4)-C(3)-C(2)	112.5(6)
C(4)-C(3)-H(6A)	109.1
C(2)-C(3)-H(6A)	109.1
C(4)-C(3)-H(6B)	109.1
C(2)-C(3)-H(6B)	109.1
H(6A)-C(3)-H(6B)	107.8
N(1)-C(2)-C(3)	108.5(6)
N(1)-C(2)-H(7A)	110.0
C(3)-C(2)-H(7A)	110.0
N(1)-C(2)-H(7B)	110.0
C(3)-C(2)-H(7B)	110.0
H(7A)-C(2)-H(7B)	108.4
C(12)-C(8)-C(9)	106.8(6)
C(12)-C(8)-C(13)	124.3(6)
C(9)-C(8)-C(13)	128.2(6)
C(12)-C(8)-Ta(1)	74.1(3)
C(9)-C(8)-Ta(1)	74.3(4)
C(13)-C(8)-Ta(1)	124.9(4)
C(10)-C(9)-C(8)	108.4(6)
C(10)-C(9)-C(14)	123.8(7)
C(8)-C(9)-C(14)	127.0(7)
C(10)-C(9)-Ta(1)	75.3(4)
C(8)-C(9)-Ta(1)	71.8(3)
C(14)-C(9)-Ta(1)	127.0(5)



C(9)-C(10)-C(11)	109.1(6)
C(9)-C(10)-C(15)	122.3(8)
C(11)-C(10)-C(15)	127.7(8)
C(9)-C(10)-Ta(1)	72.5(4)
C(11)-C(10)-Ta(1)	73.4(4)
C(15)-C(10)-Ta(1)	129.0(6)
C(10)-C(11)-C(12)	107.9(6)
C(10)-C(11)-C(16')	114.3(9)
C(12)-C(11)-C(16')	137.4(10)
C(10)-C(11)-C(16)	136.9(9)
C(12)-C(11)-C(16)	114.6(9)
C(16')-C(11)-C(16)	22.8(7)
C(10)-C(11)-Ta(1)	74.1(4)
C(12)-C(11)-Ta(1)	72.1(4)
C(16')-C(11)-Ta(1)	125.2(7)
C(16)-C(11)-Ta(1)	125.0(7)
C(11)-C(12)-C(8)	107.9(6)
C(11)-C(12)-C(17)	125.6(7)
C(8)-C(12)-C(17)	125.8(7)
C(11)-C(12)-Ta(1)	74.8(4)
C(8)-C(12)-Ta(1)	72.3(3)
C(17)-C(12)-Ta(1)	126.3(5)
C(8)-C(13)-H(13A)	109.5
C(8)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(8)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(9)-C(14)-H(14A)	109.5
C(9)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(9)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(10)-C(15)-H(15A)	109.5
C(10)-C(15)-H(15B)	109.5

H(15A)-C(15)-H(15B)	109.5
C(10)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(16)-C(16')-C(11)	86(2)
C(16')-C(16)-C(11)	71(2)
C(12)-C(17)-H(17A)	109.5
C(12)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(12)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
N(21)-C(21)-N(23)	126.3(6)
N(21)-C(21)-N(22)	109.2(5)
N(23)-C(21)-N(22)	124.5(6)
N(21)-C(21)-Ta(2)	56.2(3)
N(23)-C(21)-Ta(2)	171.7(5)
N(22)-C(21)-Ta(2)	53.4(3)
N(22)-C(22)-C(23)	111.2(7)
N(22)-C(22)-H(19A)	109.4
C(23)-C(22)-H(19A)	109.4
N(22)-C(22)-H(19B)	109.4
C(23)-C(22)-H(19B)	109.4
H(19A)-C(22)-H(19B)	108.0
C(22)-C(23)-C(24)	110.9(9)
C(22)-C(23)-H(20A)	109.5
C(24)-C(23)-H(20A)	109.5
C(22)-C(23)-H(20B)	109.5
C(24)-C(23)-H(20B)	109.5
H(20A)-C(23)-H(20B)	108.0
N(23)-C(24)-C(23)	109.8(7)
N(23)-C(24)-H(21A)	109.7
C(23)-C(24)-H(21A)	109.7
N(23)-C(24)-H(21B)	109.7
C(23)-C(24)-H(21B)	109.7
H(21A)-C(24)-H(21B)	108.2

N(23)-C(25)-C(26)	112.0(6)
N(23)-C(25)-H(22A)	109.2
C(26)-C(25)-H(22A)	109.2
N(23)-C(25)-H(22B)	109.2
C(26)-C(25)-H(22B)	109.2
H(22A)-C(25)-H(22B)	107.9
C(25)-C(26)-C(27)	112.0(6)
C(25)-C(26)-H(23A)	109.2
C(27)-C(26)-H(23A)	109.2
C(25)-C(26)-H(23B)	109.2
C(27)-C(26)-H(23B)	109.2
H(23A)-C(26)-H(23B)	107.9
N(21)-C(27)-C(26)	108.5(6)
N(21)-C(27)-H(24A)	110.0
C(26)-C(27)-H(24A)	110.0
N(21)-C(27)-H(24B)	110.0
C(26)-C(27)-H(24B)	110.0
H(24A)-C(27)-H(24B)	108.4
C(29)-C(28)-C(32)	106.4(6)
C(29)-C(28)-C(33)	124.8(7)
C(32)-C(28)-C(33)	128.1(7)
C(29)-C(28)-Ta(2)	74.3(4)
C(32)-C(28)-Ta(2)	74.0(4)
C(33)-C(28)-Ta(2)	124.0(5)
C(30)-C(29)-C(28)	109.2(6)
C(30)-C(29)-C(34)	124.3(7)
C(28)-C(29)-C(34)	126.1(7)
C(30)-C(29)-Ta(2)	75.8(4)
C(28)-C(29)-Ta(2)	72.0(4)
C(34)-C(29)-Ta(2)	124.7(5)
C(29)-C(30)-C(31)	107.4(6)
C(29)-C(30)-C(35)	127.5(8)
C(31)-C(30)-C(35)	124.3(8)
C(29)-C(30)-Ta(2)	71.6(4)
C(31)-C(30)-Ta(2)	74.1(4)
C(35)-C(30)-Ta(2)	127.5(5)

C(32)-C(31)-C(30)	108.7(6)
C(32)-C(31)-C(36)	125.9(7)
C(30)-C(31)-C(36)	124.5(7)
C(32)-C(31)-Ta(2)	71.1(3)
C(30)-C(31)-Ta(2)	73.4(4)
C(36)-C(31)-Ta(2)	130.0(5)
C(31)-C(32)-C(28)	108.2(6)
C(31)-C(32)-C(37)	122.3(7)
C(28)-C(32)-C(37)	128.4(7)
C(31)-C(32)-Ta(2)	76.4(4)
C(28)-C(32)-Ta(2)	72.1(3)
C(37)-C(32)-Ta(2)	127.0(5)
C(28)-C(33)-H(30A)	109.5
C(28)-C(33)-H(30B)	109.5
H(30A)-C(33)-H(30B)	109.5
C(28)-C(33)-H(30C)	109.5
H(30A)-C(33)-H(30C)	109.5
H(30B)-C(33)-H(30C)	109.5
C(29)-C(34)-H(31A)	109.5
C(29)-C(34)-H(31B)	109.5
H(31A)-C(34)-H(31B)	109.5
C(29)-C(34)-H(31C)	109.5
H(31A)-C(34)-H(31C)	109.5
H(31B)-C(34)-H(31C)	109.5
C(30)-C(35)-H(32A)	109.5
C(30)-C(35)-H(32B)	109.5
H(32A)-C(35)-H(32B)	109.5
C(30)-C(35)-H(32C)	109.5
H(32A)-C(35)-H(32C)	109.5
H(32B)-C(35)-H(32C)	109.5
C(31)-C(36)-H(33A)	109.5
C(31)-C(36)-H(33B)	109.5
H(33A)-C(36)-H(33B)	109.5
C(31)-C(36)-H(33C)	109.5
H(33A)-C(36)-H(33C)	109.5
H(33B)-C(36)-H(33C)	109.5

C(32)-C(37)-H(34A)	109.5
C(32)-C(37)-H(34B)	109.5
H(34A)-C(37)-H(34B)	109.5
C(32)-C(37)-H(34C)	109.5
H(34A)-C(37)-H(34C)	109.5
H(34B)-C(37)-H(34C)	109.5

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Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for mes109. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Ta(1)	16(1)	22(1)	22(1)	-3(1)	0(1)	2(1)
Cl(1)	43(1)	51(1)	28(1)	-5(1)	14(1)	-12(1)
N(3)	24(3)	32(3)	25(3)	0(2)	-1(2)	-1(2)
C(1)	23(3)	30(3)	22(3)	4(3)	-1(2)	-3(2)
Ta(2)	19(1)	25(1)	19(1)	1(1)	5(1)	2(1)
Cl(2)	55(1)	22(1)	50(1)	-3(1)	18(1)	-8(1)
N(2)	22(3)	29(3)	26(3)	-9(2)	-6(2)	8(2)
C(7)	40(4)	34(4)	27(3)	-3(3)	-1(3)	11(3)
Cl(3)	36(1)	55(1)	34(1)	-9(1)	-9(1)	26(1)
N(1)	20(2)	30(3)	34(3)	-3(2)	-4(2)	10(2)
C(6)	60(5)	25(4)	34(4)	-4(3)	-5(3)	3(3)
Cl(4)	42(1)	40(1)	23(1)	-3(1)	3(1)	-10(1)
N(23)	37(3)	43(4)	32(3)	5(3)	4(2)	14(3)
C(5)	42(4)	34(4)	24(3)	-7(3)	2(3)	-12(3)
Cl(5)	26(1)	46(1)	33(1)	1(1)	11(1)	8(1)
N(22)	25(3)	37(3)	26(3)	7(2)	10(2)	10(2)
C(4)	29(3)	54(5)	28(4)	3(3)	-3(3)	2(3)
Cl(6)	41(1)	29(1)	29(1)	-2(1)	5(1)	-7(1)
N(21)	28(3)	31(3)	22(3)	8(2)	5(2)	6(2)
C(3)	33(4)	61(5)	35(4)	6(3)	-6(3)	19(3)
C(2)	29(3)	50(4)	33(4)	-3(3)	-3(3)	12(3)
C(8)	26(3)	24(3)	23(3)	-5(2)	6(2)	1(2)
C(9)	56(4)	21(3)	26(3)	-3(2)	11(3)	-2(3)
C(10)	50(4)	43(4)	42(4)	-26(3)	23(4)	-25(4)
C(11)	29(4)	62(5)	39(4)	-30(4)	-2(3)	7(3)
C(12)	47(4)	23(3)	23(3)	-7(2)	-1(3)	0(3)
C(13)	26(3)	61(5)	55(5)	-22(4)	19(3)	-11(3)
C(17)	110(8)	39(4)	24(4)	3(3)	-9(4)	-8(5)
C(21)	26(3)	27(3)	27(3)	1(2)	2(2)	4(2)
C(22)	38(4)	78(6)	52(5)	21(5)	23(4)	29(4)
C(24)	44(5)	82(7)	62(6)	23(5)	12(4)	36(5)

C(25)	64(5)	38(4)	30(4)	7(3)	10(3)	18(4)
C(26)	60(5)	28(4)	25(3)	3(3)	4(3)	-8(3)
C(27)	32(3)	42(4)	22(3)	8(3)	7(3)	-5(3)
C(28)	31(3)	36(4)	25(3)	5(3)	4(3)	-7(3)
C(29)	59(5)	27(3)	27(3)	3(3)	16(3)	3(3)
C(30)	41(4)	39(4)	28(3)	13(3)	6(3)	13(3)
C(31)	38(4)	30(3)	27(3)	8(3)	5(3)	-4(3)
C(32)	37(4)	30(3)	25(3)	4(3)	15(3)	1(3)
C(33)	54(5)	63(6)	43(5)	17(4)	0(4)	-29(4)
C(35)	67(6)	91(8)	64(6)	33(6)	14(5)	46(6)
C(36)	55(5)	60(5)	34(4)	14(4)	-7(4)	-19(4)
C(37)	65(6)	51(5)	39(4)	-1(4)	26(4)	10(4)

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Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes109.

	x	y	z	U(eq)
H(2A)	1472	4675	1103	41
H(2B)	1457	5501	633	41
H(3A)	931	3490	545	49
H(3B)	698	4100	951	49
H(4A)	684	5631	126	40
H(4B)	253	4890	302	40
H(5A)	-215	7131	536	45
H(5B)	6	7800	130	45
H(6A)	-245	9722	627	53
H(6B)	253	9965	505	53
H(7A)	272	10316	1272	46
H(7B)	64	8635	1316	46
H(13A)	420	10096	2506	69
H(13B)	436	8337	2361	69
H(13C)	269	9623	2002	69
H(14A)	846	12908	1857	92
H(14B)	557	11666	1551	92
H(14C)	1016	12329	1414	92
H(15A)	1953	12731	2027	108
H(15B)	1742	12300	1532	108
H(15C)	2185	11434	1767	108
H(17A)	1344	8325	3064	89
H(17B)	1584	7059	2800	89
H(17C)	1043	7174	2735	89
H(19A)	2264	2239	3756	65
H(19B)	2147	449	3712	65
H(20A)	2565	1940	4490	107
H(20B)	2808	753	4202	107
H(21A)	2384	-1291	4411	75
H(21B)	2577	-399	4857	75



H(22A)	1932	-353	5320	52
H(22B)	1845	-1914	5046	52
H(23A)	1200	-848	5358	46
H(23B)	1101	-1443	4859	46
H(24A)	755	972	4893	38
H(24B)	1225	1718	5116	38
H(30A)	2151	6027	3560	81
H(30B)	2242	4336	3756	81
H(30C)	2083	5667	4058	81
H(31A)	1225	7966	3840	90
H(31B)	1476	6970	4242	90
H(31C)	935	7068	4157	90
H(32A)	285	6536	3218	110
H(32B)	284	6147	3728	110
H(32C)	106	4906	3358	110
H(33A)	598	3903	2550	77
H(33B)	307	3128	2889	77
H(33C)	726	2226	2740	77
H(34A)	1706	3407	2726	74
H(34B)	1473	1892	2883	74
H(34C)	1942	2486	3150	74

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Torsion angles [°] for mes109.

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C(5)-N(3)-C(1)-N(2)	6.1(10)
C(4)-N(3)-C(1)-N(2)	-174.7(6)
C(5)-N(3)-C(1)-N(1)	-172.8(6)
C(4)-N(3)-C(1)-N(1)	6.4(10)
C(5)-N(3)-C(1)-Ta(1)	-97(4)
C(4)-N(3)-C(1)-Ta(1)	82(4)
N(1)-Ta(1)-C(1)-N(2)	172.3(6)
Cl(3)-Ta(1)-C(1)-N(2)	-8.0(4)
Cl(1)-Ta(1)-C(1)-N(2)	74.6(4)
C(8)-Ta(1)-C(1)-N(2)	-159.9(4)
Cl(2)-Ta(1)-C(1)-N(2)	-86.4(4)
C(12)-Ta(1)-C(1)-N(2)	-134.1(4)
C(9)-Ta(1)-C(1)-N(2)	162.7(4)
C(11)-Ta(1)-C(1)-N(2)	158.8(11)
C(10)-Ta(1)-C(1)-N(2)	134.5(4)
N(1)-Ta(1)-C(1)-N(3)	-80(4)
N(2)-Ta(1)-C(1)-N(3)	107(5)
Cl(3)-Ta(1)-C(1)-N(3)	99(4)
Cl(1)-Ta(1)-C(1)-N(3)	-178(100)
C(8)-Ta(1)-C(1)-N(3)	-53(4)
Cl(2)-Ta(1)-C(1)-N(3)	21(4)
C(12)-Ta(1)-C(1)-N(3)	-27(5)
C(9)-Ta(1)-C(1)-N(3)	-90(4)
C(11)-Ta(1)-C(1)-N(3)	-94(5)
C(10)-Ta(1)-C(1)-N(3)	-118(4)
N(2)-Ta(1)-C(1)-N(1)	-172.3(6)
Cl(3)-Ta(1)-C(1)-N(1)	179.7(3)
Cl(1)-Ta(1)-C(1)-N(1)	-97.7(4)
C(8)-Ta(1)-C(1)-N(1)	27.7(4)
Cl(2)-Ta(1)-C(1)-N(1)	101.2(4)
C(12)-Ta(1)-C(1)-N(1)	53.6(5)
C(9)-Ta(1)-C(1)-N(1)	-9.7(5)
C(11)-Ta(1)-C(1)-N(1)	-13.5(13)
C(10)-Ta(1)-C(1)-N(1)	-37.9(5)

N(3)-C(1)-N(2)-C(7)	-5.6(10)
N(1)-C(1)-N(2)-C(7)	173.4(6)
Ta(1)-C(1)-N(2)-C(7)	166.9(7)
N(3)-C(1)-N(2)-Ta(1)	-172.5(6)
N(1)-C(1)-N(2)-Ta(1)	6.5(5)
N(1)-Ta(1)-N(2)-C(1)	-4.6(4)
Cl(3)-Ta(1)-N(2)-C(1)	172.6(4)
Cl(1)-Ta(1)-N(2)-C(1)	-101.0(4)
C(8)-Ta(1)-N(2)-C(1)	31.4(6)
Cl(2)-Ta(1)-N(2)-C(1)	87.0(4)
C(12)-Ta(1)-N(2)-C(1)	97.4(6)
C(9)-Ta(1)-N(2)-C(1)	-26.4(6)
C(11)-Ta(1)-N(2)-C(1)	-167.4(7)
C(10)-Ta(1)-N(2)-C(1)	-85.1(6)
N(1)-Ta(1)-N(2)-C(7)	-165.1(9)
Cl(3)-Ta(1)-N(2)-C(7)	12.0(8)
Cl(1)-Ta(1)-N(2)-C(7)	98.5(8)
C(8)-Ta(1)-N(2)-C(7)	-129.1(7)
Cl(2)-Ta(1)-N(2)-C(7)	-73.5(8)
C(12)-Ta(1)-N(2)-C(7)	-63.1(10)
C(9)-Ta(1)-N(2)-C(7)	173.0(7)
C(11)-Ta(1)-N(2)-C(7)	32.1(13)
C(10)-Ta(1)-N(2)-C(7)	114.3(8)
C(1)-Ta(1)-N(2)-C(7)	-160.6(10)
C(1)-N(2)-C(7)-C(6)	-25.3(9)
Ta(1)-N(2)-C(7)-C(6)	132.0(7)
N(2)-C(1)-N(1)-C(2)	-168.5(6)
N(3)-C(1)-N(1)-C(2)	10.5(10)
Ta(1)-C(1)-N(1)-C(2)	-161.7(7)
N(2)-C(1)-N(1)-Ta(1)	-6.8(6)
N(3)-C(1)-N(1)-Ta(1)	172.3(6)
N(2)-Ta(1)-N(1)-C(1)	4.4(4)
Cl(3)-Ta(1)-N(1)-C(1)	-0.5(6)
Cl(1)-Ta(1)-N(1)-C(1)	80.9(4)
C(8)-Ta(1)-N(1)-C(1)	-155.3(4)
Cl(2)-Ta(1)-N(1)-C(1)	-74.4(4)

C(12)-Ta(1)-N(1)-C(1)	-146.8(4)
C(9)-Ta(1)-N(1)-C(1)	171.1(4)
C(11)-Ta(1)-N(1)-C(1)	176.0(4)
C(10)-Ta(1)-N(1)-C(1)	153.6(4)
N(2)-Ta(1)-N(1)-C(2)	155.1(9)
Cl(3)-Ta(1)-N(1)-C(2)	150.2(7)
Cl(1)-Ta(1)-N(1)-C(2)	-128.4(8)
C(8)-Ta(1)-N(1)-C(2)	-4.6(8)
Cl(2)-Ta(1)-N(1)-C(2)	76.3(8)
C(12)-Ta(1)-N(1)-C(2)	3.9(9)
C(9)-Ta(1)-N(1)-C(2)	-38.2(8)
C(11)-Ta(1)-N(1)-C(2)	-33.3(10)
C(10)-Ta(1)-N(1)-C(2)	-55.7(9)
C(1)-Ta(1)-N(1)-C(2)	150.7(11)
N(2)-C(7)-C(6)-C(5)	53.9(8)
C(1)-N(3)-C(5)-C(6)	24.8(8)
C(4)-N(3)-C(5)-C(6)	-154.4(6)
C(7)-C(6)-C(5)-N(3)	-53.9(8)
N(21)-Ta(2)-N(22)-C(21)	-4.9(4)
Cl(5)-Ta(2)-N(22)-C(21)	-6.7(6)
C(28)-Ta(2)-N(22)-C(21)	154.9(4)
Cl(6)-Ta(2)-N(22)-C(21)	-84.8(4)
C(32)-Ta(2)-N(22)-C(21)	-171.3(4)
C(29)-Ta(2)-N(22)-C(21)	144.0(4)
Cl(4)-Ta(2)-N(22)-C(21)	72.2(4)
C(30)-Ta(2)-N(22)-C(21)	178.3(4)
C(31)-Ta(2)-N(22)-C(21)	-155.7(4)
N(21)-Ta(2)-N(22)-C(22)	-176.1(11)
Cl(5)-Ta(2)-N(22)-C(22)	-177.9(9)
C(28)-Ta(2)-N(22)-C(22)	-16.4(10)
Cl(6)-Ta(2)-N(22)-C(22)	103.9(10)
C(32)-Ta(2)-N(22)-C(22)	17.5(10)
C(29)-Ta(2)-N(22)-C(22)	-27.2(11)
Cl(4)-Ta(2)-N(22)-C(22)	-99.0(10)
C(30)-Ta(2)-N(22)-C(22)	7.1(12)
C(31)-Ta(2)-N(22)-C(22)	33.1(11)

C(21)-Ta(2)-N(22)-C(22)	-171.2(12)
C(1)-N(3)-C(4)-C(3)	10.8(9)
C(5)-N(3)-C(4)-C(3)	-170.1(6)
N(22)-Ta(2)-N(21)-C(21)	5.0(4)
Cl(5)-Ta(2)-N(21)-C(21)	-176.0(4)
C(28)-Ta(2)-N(21)-C(21)	-30.5(6)
Cl(6)-Ta(2)-N(21)-C(21)	97.2(4)
C(32)-Ta(2)-N(21)-C(21)	28.6(6)
C(29)-Ta(2)-N(21)-C(21)	-95.0(6)
Cl(4)-Ta(2)-N(21)-C(21)	-88.1(4)
C(30)-Ta(2)-N(21)-C(21)	179.1(6)
C(31)-Ta(2)-N(21)-C(21)	91.9(6)
N(22)-Ta(2)-N(21)-C(27)	146.6(7)
Cl(5)-Ta(2)-N(21)-C(27)	-34.5(7)
C(28)-Ta(2)-N(21)-C(27)	111.0(7)
Cl(6)-Ta(2)-N(21)-C(27)	-121.3(7)
C(32)-Ta(2)-N(21)-C(27)	170.1(6)
C(29)-Ta(2)-N(21)-C(27)	46.5(9)
Cl(4)-Ta(2)-N(21)-C(27)	53.5(7)
C(30)-Ta(2)-N(21)-C(27)	-39.4(11)
C(31)-Ta(2)-N(21)-C(27)	-126.6(7)
C(21)-Ta(2)-N(21)-C(27)	141.5(9)
N(3)-C(4)-C(3)-C(2)	-42.8(8)
C(1)-N(1)-C(2)-C(3)	-41.4(8)
Ta(1)-N(1)-C(2)-C(3)	171.5(6)
C(4)-C(3)-C(2)-N(1)	58.4(8)
N(1)-Ta(1)-C(8)-C(12)	166.7(4)
N(2)-Ta(1)-C(8)-C(12)	135.3(4)
Cl(3)-Ta(1)-C(8)-C(12)	3.9(4)
Cl(1)-Ta(1)-C(8)-C(12)	-105.1(4)
Cl(2)-Ta(1)-C(8)-C(12)	80.4(4)
C(9)-Ta(1)-C(8)-C(12)	-113.0(6)
C(11)-Ta(1)-C(8)-C(12)	-37.1(4)
C(10)-Ta(1)-C(8)-C(12)	-77.0(4)
C(1)-Ta(1)-C(8)-C(12)	152.4(4)
N(1)-Ta(1)-C(8)-C(9)	-80.3(4)

N(2)-Ta(1)-C(8)-C(9)	-111.7(5)
Cl(3)-Ta(1)-C(8)-C(9)	117.0(4)
Cl(1)-Ta(1)-C(8)-C(9)	7.9(4)
Cl(2)-Ta(1)-C(8)-C(9)	-166.6(4)
C(12)-Ta(1)-C(8)-C(9)	113.0(6)
C(11)-Ta(1)-C(8)-C(9)	76.0(5)
C(10)-Ta(1)-C(8)-C(9)	36.1(4)
C(1)-Ta(1)-C(8)-C(9)	-94.6(4)
N(1)-Ta(1)-C(8)-C(13)	45.7(6)
N(2)-Ta(1)-C(8)-C(13)	14.3(8)
Cl(3)-Ta(1)-C(8)-C(13)	-117.1(6)
Cl(1)-Ta(1)-C(8)-C(13)	133.9(6)
Cl(2)-Ta(1)-C(8)-C(13)	-40.6(6)
C(12)-Ta(1)-C(8)-C(13)	-121.0(8)
C(9)-Ta(1)-C(8)-C(13)	126.0(8)
C(11)-Ta(1)-C(8)-C(13)	-158.1(7)
C(10)-Ta(1)-C(8)-C(13)	162.1(7)
C(1)-Ta(1)-C(8)-C(13)	31.4(7)
C(12)-C(8)-C(9)-C(10)	0.6(7)
C(13)-C(8)-C(9)-C(10)	170.7(6)
Ta(1)-C(8)-C(9)-C(10)	-67.0(5)
C(12)-C(8)-C(9)-C(14)	-169.3(7)
C(13)-C(8)-C(9)-C(14)	0.7(11)
Ta(1)-C(8)-C(9)-C(14)	123.1(7)
C(12)-C(8)-C(9)-Ta(1)	67.5(4)
C(13)-C(8)-C(9)-Ta(1)	-122.4(7)
N(1)-Ta(1)-C(9)-C(10)	-148.0(5)
N(2)-Ta(1)-C(9)-C(10)	-128.5(5)
Cl(3)-Ta(1)-C(9)-C(10)	25.5(5)
Cl(1)-Ta(1)-C(9)-C(10)	-58.0(4)
C(8)-Ta(1)-C(9)-C(10)	115.5(6)
Cl(2)-Ta(1)-C(9)-C(10)	129.9(4)
C(12)-Ta(1)-C(9)-C(10)	77.0(5)
C(11)-Ta(1)-C(9)-C(10)	36.2(4)
C(1)-Ta(1)-C(9)-C(10)	-142.8(4)
N(1)-Ta(1)-C(9)-C(8)	96.6(4)

N(2)-Ta(1)-C(9)-C(8)	116.0(4)
Cl(3)-Ta(1)-C(9)-C(8)	-89.9(4)
Cl(1)-Ta(1)-C(9)-C(8)	-173.4(4)
Cl(2)-Ta(1)-C(9)-C(8)	14.5(4)
C(12)-Ta(1)-C(9)-C(8)	-38.4(4)
C(11)-Ta(1)-C(9)-C(8)	-79.3(4)
C(10)-Ta(1)-C(9)-C(8)	-115.5(6)
C(1)-Ta(1)-C(9)-C(8)	101.7(4)
N(1)-Ta(1)-C(9)-C(14)	-26.6(7)
N(2)-Ta(1)-C(9)-C(14)	-7.1(9)
Cl(3)-Ta(1)-C(9)-C(14)	146.9(6)
Cl(1)-Ta(1)-C(9)-C(14)	63.5(7)
C(8)-Ta(1)-C(9)-C(14)	-123.1(9)
Cl(2)-Ta(1)-C(9)-C(14)	-108.7(7)
C(12)-Ta(1)-C(9)-C(14)	-161.5(8)
C(11)-Ta(1)-C(9)-C(14)	157.6(8)
C(10)-Ta(1)-C(9)-C(14)	121.4(9)
C(1)-Ta(1)-C(9)-C(14)	-21.4(7)
C(8)-C(9)-C(10)-C(11)	-0.1(8)
C(14)-C(9)-C(10)-C(11)	170.2(7)
Ta(1)-C(9)-C(10)-C(11)	-64.8(5)
C(8)-C(9)-C(10)-C(15)	-169.8(7)
C(14)-C(9)-C(10)-C(15)	0.5(11)
Ta(1)-C(9)-C(10)-C(15)	125.5(7)
C(8)-C(9)-C(10)-Ta(1)	64.7(4)
C(14)-C(9)-C(10)-Ta(1)	-125.0(7)
N(1)-Ta(1)-C(10)-C(9)	33.9(5)
N(2)-Ta(1)-C(10)-C(9)	101.9(5)
Cl(3)-Ta(1)-C(10)-C(9)	-161.0(4)
Cl(1)-Ta(1)-C(10)-C(9)	118.0(4)
C(8)-Ta(1)-C(10)-C(9)	-37.9(4)
Cl(2)-Ta(1)-C(10)-C(9)	-68.0(5)
C(12)-Ta(1)-C(10)-C(9)	-79.5(4)
C(11)-Ta(1)-C(10)-C(9)	-116.9(6)
C(1)-Ta(1)-C(10)-C(9)	54.1(5)
N(1)-Ta(1)-C(10)-C(11)	150.7(4)

N(2)-Ta(1)-C(10)-C(11)	-141.3(5)
Cl(3)-Ta(1)-C(10)-C(11)	-44.1(4)
Cl(1)-Ta(1)-C(10)-C(11)	-125.2(5)
C(8)-Ta(1)-C(10)-C(11)	78.9(4)
Cl(2)-Ta(1)-C(10)-C(11)	48.8(5)
C(12)-Ta(1)-C(10)-C(11)	37.4(4)
C(9)-Ta(1)-C(10)-C(11)	116.9(6)
C(1)-Ta(1)-C(10)-C(11)	170.9(4)
N(1)-Ta(1)-C(10)-C(15)	-83.9(8)
N(2)-Ta(1)-C(10)-C(15)	-15.9(11)
Cl(3)-Ta(1)-C(10)-C(15)	81.3(8)
Cl(1)-Ta(1)-C(10)-C(15)	0.2(8)
C(8)-Ta(1)-C(10)-C(15)	-155.7(9)
Cl(2)-Ta(1)-C(10)-C(15)	174.2(7)
C(12)-Ta(1)-C(10)-C(15)	162.8(9)
C(9)-Ta(1)-C(10)-C(15)	-117.7(10)
C(11)-Ta(1)-C(10)-C(15)	125.4(10)
C(1)-Ta(1)-C(10)-C(15)	-63.6(9)
C(9)-C(10)-C(11)-C(12)	-0.5(8)
C(15)-C(10)-C(11)-C(12)	168.6(7)
Ta(1)-C(10)-C(11)-C(12)	-64.7(5)
C(9)-C(10)-C(11)-C(16')	-173.8(8)
C(15)-C(10)-C(11)-C(16')	-4.8(12)
Ta(1)-C(10)-C(11)-C(16')	122.0(8)
C(9)-C(10)-C(11)-C(16)	-171.1(11)
C(15)-C(10)-C(11)-C(16)	-2.1(16)
Ta(1)-C(10)-C(11)-C(16)	124.7(11)
C(9)-C(10)-C(11)-Ta(1)	64.2(5)
C(15)-C(10)-C(11)-Ta(1)	-126.8(8)
N(1)-Ta(1)-C(11)-C(10)	-41.9(6)
N(2)-Ta(1)-C(11)-C(10)	115.8(7)
Cl(3)-Ta(1)-C(11)-C(10)	136.1(4)
Cl(1)-Ta(1)-C(11)-C(10)	51.8(4)
C(8)-Ta(1)-C(11)-C(10)	-77.6(4)
Cl(2)-Ta(1)-C(11)-C(10)	-143.5(4)
C(12)-Ta(1)-C(11)-C(10)	-115.3(6)



C(9)-Ta(1)-C(11)-C(10)	-35.9(4)
C(1)-Ta(1)-C(11)-C(10)	-31.5(14)
N(1)-Ta(1)-C(11)-C(12)	73.4(6)
N(2)-Ta(1)-C(11)-C(12)	-128.9(7)
Cl(3)-Ta(1)-C(11)-C(12)	-108.6(4)
Cl(1)-Ta(1)-C(11)-C(12)	167.2(4)
C(8)-Ta(1)-C(11)-C(12)	37.7(4)
Cl(2)-Ta(1)-C(11)-C(12)	-28.2(4)
C(9)-Ta(1)-C(11)-C(12)	79.4(4)
C(10)-Ta(1)-C(11)-C(12)	115.3(6)
C(1)-Ta(1)-C(11)-C(12)	83.8(13)
N(1)-Ta(1)-C(11)-C(16')	-150.7(9)
N(2)-Ta(1)-C(11)-C(16')	7.0(15)
Cl(3)-Ta(1)-C(11)-C(16')	27.3(10)
Cl(1)-Ta(1)-C(11)-C(16')	-56.9(10)
C(8)-Ta(1)-C(11)-C(16')	173.6(11)
Cl(2)-Ta(1)-C(11)-C(16')	107.7(10)
C(12)-Ta(1)-C(11)-C(16')	135.9(12)
C(9)-Ta(1)-C(11)-C(16')	-144.7(11)
C(10)-Ta(1)-C(11)-C(16')	-108.8(11)
C(1)-Ta(1)-C(11)-C(16')	-140.3(11)
N(1)-Ta(1)-C(11)-C(16)	-178.6(9)
N(2)-Ta(1)-C(11)-C(16)	-20.9(15)
Cl(3)-Ta(1)-C(11)-C(16)	-0.6(9)
Cl(1)-Ta(1)-C(11)-C(16)	-84.9(10)
C(8)-Ta(1)-C(11)-C(16)	145.7(11)
Cl(2)-Ta(1)-C(11)-C(16)	79.8(10)
C(12)-Ta(1)-C(11)-C(16)	108.0(11)
C(9)-Ta(1)-C(11)-C(16)	-172.6(11)
C(10)-Ta(1)-C(11)-C(16)	-136.7(11)
C(1)-Ta(1)-C(11)-C(16)	-168.2(11)
C(10)-C(11)-C(12)-C(8)	0.8(7)
C(16')-C(11)-C(12)-C(8)	171.8(11)
C(16)-C(11)-C(12)-C(8)	173.8(8)
Ta(1)-C(11)-C(12)-C(8)	-65.2(4)
C(10)-C(11)-C(12)-C(17)	-170.0(7)

C(16')-C(11)-C(12)-C(17)	1.0(15)
C(16)-C(11)-C(12)-C(17)	3.0(11)
Ta(1)-C(11)-C(12)-C(17)	124.0(7)
C(10)-C(11)-C(12)-Ta(1)	66.0(5)
C(16')-C(11)-C(12)-Ta(1)	-123.0(12)
C(16)-C(11)-C(12)-Ta(1)	-121.0(8)
C(9)-C(8)-C(12)-C(11)	-0.8(7)
C(13)-C(8)-C(12)-C(11)	-171.4(6)
Ta(1)-C(8)-C(12)-C(11)	66.9(5)
C(9)-C(8)-C(12)-C(17)	169.9(6)
C(13)-C(8)-C(12)-C(17)	-0.6(10)
Ta(1)-C(8)-C(12)-C(17)	-122.3(7)
C(9)-C(8)-C(12)-Ta(1)	-67.7(4)
C(13)-C(8)-C(12)-Ta(1)	121.7(6)
N(1)-Ta(1)-C(12)-C(11)	-130.2(4)
N(2)-Ta(1)-C(12)-C(11)	141.8(5)
Cl(3)-Ta(1)-C(12)-C(11)	68.4(4)
Cl(1)-Ta(1)-C(12)-C(11)	-15.8(5)
C(8)-Ta(1)-C(12)-C(11)	-114.9(6)
Cl(2)-Ta(1)-C(12)-C(11)	152.3(4)
C(9)-Ta(1)-C(12)-C(11)	-76.3(5)
C(10)-Ta(1)-C(12)-C(11)	-36.6(4)
C(1)-Ta(1)-C(12)-C(11)	-159.8(4)
N(1)-Ta(1)-C(12)-C(8)	-15.4(5)
N(2)-Ta(1)-C(12)-C(8)	-103.3(5)
Cl(3)-Ta(1)-C(12)-C(8)	-176.8(4)
Cl(1)-Ta(1)-C(12)-C(8)	99.1(4)
Cl(2)-Ta(1)-C(12)-C(8)	-92.8(4)
C(9)-Ta(1)-C(12)-C(8)	38.6(4)
C(11)-Ta(1)-C(12)-C(8)	114.9(6)
C(10)-Ta(1)-C(12)-C(8)	78.3(4)
C(1)-Ta(1)-C(12)-C(8)	-44.9(5)
N(1)-Ta(1)-C(12)-C(17)	106.4(7)
N(2)-Ta(1)-C(12)-C(17)	18.5(10)
Cl(3)-Ta(1)-C(12)-C(17)	-55.0(7)
Cl(1)-Ta(1)-C(12)-C(17)	-139.2(7)

C(8)-Ta(1)-C(12)-C(17)	121.8(9)
Cl(2)-Ta(1)-C(12)-C(17)	28.9(7)
C(9)-Ta(1)-C(12)-C(17)	160.4(8)
C(11)-Ta(1)-C(12)-C(17)	-123.3(9)
C(10)-Ta(1)-C(12)-C(17)	-160.0(8)
C(1)-Ta(1)-C(12)-C(17)	76.9(8)
C(10)-C(11)-C(16')-C(16)	175(2)
C(12)-C(11)-C(16')-C(16)	5(3)
Ta(1)-C(11)-C(16')-C(16)	-98(2)
C(10)-C(11)-C(16)-C(16')	-6(3)
C(12)-C(11)-C(16)-C(16')	-177(2)
Ta(1)-C(11)-C(16)-C(16')	99(2)
C(27)-N(21)-C(21)-N(23)	16.6(10)
Ta(2)-N(21)-C(21)-N(23)	170.4(6)
C(27)-N(21)-C(21)-N(22)	-161.0(6)
Ta(2)-N(21)-C(21)-N(22)	-7.2(5)
C(27)-N(21)-C(21)-Ta(2)	-153.7(6)
C(24)-N(23)-C(21)-N(21)	-176.4(8)
C(25)-N(23)-C(21)-N(21)	1.4(11)
C(24)-N(23)-C(21)-N(22)	0.8(11)
C(25)-N(23)-C(21)-N(22)	178.6(6)
C(24)-N(23)-C(21)-Ta(2)	-72(4)
C(25)-N(23)-C(21)-Ta(2)	106(3)
C(22)-N(22)-C(21)-N(21)	-177.9(7)
Ta(2)-N(22)-C(21)-N(21)	7.5(5)
C(22)-N(22)-C(21)-N(23)	4.5(10)
Ta(2)-N(22)-C(21)-N(23)	-170.2(6)
C(22)-N(22)-C(21)-Ta(2)	174.7(8)
N(22)-Ta(2)-C(21)-N(21)	-171.5(6)
Cl(5)-Ta(2)-C(21)-N(21)	4.3(4)
C(28)-Ta(2)-C(21)-N(21)	160.5(4)
Cl(6)-Ta(2)-C(21)-N(21)	-78.6(4)
C(32)-Ta(2)-C(21)-N(21)	-161.9(4)
C(29)-Ta(2)-C(21)-N(21)	132.5(4)
Cl(4)-Ta(2)-C(21)-N(21)	85.0(4)
C(30)-Ta(2)-C(21)-N(21)	-178.1(12)

C(31)-Ta(2)-C(21)-N(21)	-134.6(4)
N(22)-Ta(2)-C(21)-N(23)	78(3)
N(21)-Ta(2)-C(21)-N(23)	-110(4)
Cl(5)-Ta(2)-C(21)-N(23)	-106(3)
C(28)-Ta(2)-C(21)-N(23)	50(3)
Cl(6)-Ta(2)-C(21)-N(23)	171(3)
C(32)-Ta(2)-C(21)-N(23)	88(3)
C(29)-Ta(2)-C(21)-N(23)	22(4)
Cl(4)-Ta(2)-C(21)-N(23)	-25(3)
C(30)-Ta(2)-C(21)-N(23)	72(4)
C(31)-Ta(2)-C(21)-N(23)	115(3)
N(21)-Ta(2)-C(21)-N(22)	171.5(6)
Cl(5)-Ta(2)-C(21)-N(22)	175.8(4)
C(28)-Ta(2)-C(21)-N(22)	-28.0(5)
Cl(6)-Ta(2)-C(21)-N(22)	92.9(4)
C(32)-Ta(2)-C(21)-N(22)	9.6(5)
C(29)-Ta(2)-C(21)-N(22)	-56.0(5)
Cl(4)-Ta(2)-C(21)-N(22)	-103.5(4)
C(30)-Ta(2)-C(21)-N(22)	-6.6(15)
C(31)-Ta(2)-C(21)-N(22)	36.9(5)
C(21)-N(22)-C(22)-C(23)	-33.2(11)
Ta(2)-N(22)-C(22)-C(23)	136.9(9)
N(22)-C(22)-C(23)-C(24)	55.6(12)
C(21)-N(23)-C(24)-C(23)	22.0(12)
C(25)-N(23)-C(24)-C(23)	-155.8(8)
C(22)-C(23)-C(24)-N(23)	-49.5(12)
C(21)-N(23)-C(25)-C(26)	11.4(10)
C(24)-N(23)-C(25)-C(26)	-170.7(7)
N(23)-C(25)-C(26)-C(27)	-39.7(8)
C(21)-N(21)-C(27)-C(26)	-44.0(8)
Ta(2)-N(21)-C(27)-C(26)	-179.8(5)
C(25)-C(26)-C(27)-N(21)	55.6(7)
N(22)-Ta(2)-C(28)-C(29)	-162.7(4)
N(21)-Ta(2)-C(28)-C(29)	-131.7(4)
Cl(5)-Ta(2)-C(28)-C(29)	4.1(5)
Cl(6)-Ta(2)-C(28)-C(29)	112.4(4)

C(32)-Ta(2)-C(28)-C(29)	112.7(6)
Cl(4)-Ta(2)-C(28)-C(29)	-75.6(4)
C(30)-Ta(2)-C(28)-C(29)	36.1(4)
C(31)-Ta(2)-C(28)-C(29)	76.0(4)
C(21)-Ta(2)-C(28)-C(29)	-148.3(4)
N(22)-Ta(2)-C(28)-C(32)	84.6(4)
N(21)-Ta(2)-C(28)-C(32)	115.6(4)
Cl(5)-Ta(2)-C(28)-C(32)	-108.6(4)
Cl(6)-Ta(2)-C(28)-C(32)	-0.3(4)
C(29)-Ta(2)-C(28)-C(32)	-112.7(6)
Cl(4)-Ta(2)-C(28)-C(32)	171.7(4)
C(30)-Ta(2)-C(28)-C(32)	-76.6(4)
C(31)-Ta(2)-C(28)-C(32)	-36.7(4)
C(21)-Ta(2)-C(28)-C(32)	98.9(4)
N(22)-Ta(2)-C(28)-C(33)	-41.0(7)
N(21)-Ta(2)-C(28)-C(33)	-9.9(8)
Cl(5)-Ta(2)-C(28)-C(33)	125.8(6)
Cl(6)-Ta(2)-C(28)-C(33)	-125.9(6)
C(32)-Ta(2)-C(28)-C(33)	-125.6(8)
C(29)-Ta(2)-C(28)-C(33)	121.7(8)
Cl(4)-Ta(2)-C(28)-C(33)	46.1(6)
C(30)-Ta(2)-C(28)-C(33)	157.8(7)
C(31)-Ta(2)-C(28)-C(33)	-162.3(7)
C(21)-Ta(2)-C(28)-C(33)	-26.6(7)
C(32)-C(28)-C(29)-C(30)	0.3(7)
C(33)-C(28)-C(29)-C(30)	171.9(6)
Ta(2)-C(28)-C(29)-C(30)	-67.3(5)
C(32)-C(28)-C(29)-C(34)	-172.1(7)
C(33)-C(28)-C(29)-C(34)	-0.5(11)
Ta(2)-C(28)-C(29)-C(34)	120.3(7)
C(32)-C(28)-C(29)-Ta(2)	67.6(4)
C(33)-C(28)-C(29)-Ta(2)	-120.8(7)
N(22)-Ta(2)-C(29)-C(30)	135.8(4)
N(21)-Ta(2)-C(29)-C(30)	-139.1(5)
Cl(5)-Ta(2)-C(29)-C(30)	-60.7(4)
C(28)-Ta(2)-C(29)-C(30)	116.0(6)

Cl(6)-Ta(2)-C(29)-C(30)	25.8(5)
C(32)-Ta(2)-C(29)-C(30)	77.2(4)
Cl(4)-Ta(2)-C(29)-C(30)	-146.2(4)
C(31)-Ta(2)-C(29)-C(30)	37.0(4)
C(21)-Ta(2)-C(29)-C(30)	165.8(4)
N(22)-Ta(2)-C(29)-C(28)	19.8(5)
N(21)-Ta(2)-C(29)-C(28)	104.9(5)
Cl(5)-Ta(2)-C(29)-C(28)	-176.7(4)
Cl(6)-Ta(2)-C(29)-C(28)	-90.2(4)
C(32)-Ta(2)-C(29)-C(28)	-38.8(4)
Cl(4)-Ta(2)-C(29)-C(28)	97.8(4)
C(30)-Ta(2)-C(29)-C(28)	-116.0(6)
C(31)-Ta(2)-C(29)-C(28)	-79.0(4)
C(21)-Ta(2)-C(29)-C(28)	49.8(5)
N(22)-Ta(2)-C(29)-C(34)	-102.1(7)
N(21)-Ta(2)-C(29)-C(34)	-17.0(10)
Cl(5)-Ta(2)-C(29)-C(34)	61.4(7)
C(28)-Ta(2)-C(29)-C(34)	-121.9(9)
Cl(6)-Ta(2)-C(29)-C(34)	147.9(6)
C(32)-Ta(2)-C(29)-C(34)	-160.7(8)
Cl(4)-Ta(2)-C(29)-C(34)	-24.0(7)
C(30)-Ta(2)-C(29)-C(34)	122.1(9)
C(31)-Ta(2)-C(29)-C(34)	159.1(8)
C(21)-Ta(2)-C(29)-C(34)	-72.1(8)
C(28)-C(29)-C(30)-C(31)	-1.2(8)
C(34)-C(29)-C(30)-C(31)	171.4(7)
Ta(2)-C(29)-C(30)-C(31)	-66.0(5)
C(28)-C(29)-C(30)-C(35)	-171.5(7)
C(34)-C(29)-C(30)-C(35)	1.1(12)
Ta(2)-C(29)-C(30)-C(35)	123.7(8)
C(28)-C(29)-C(30)-Ta(2)	64.8(5)
C(34)-C(29)-C(30)-Ta(2)	-122.6(7)
N(22)-Ta(2)-C(30)-C(29)	-66.3(5)
N(21)-Ta(2)-C(30)-C(29)	121.7(6)
Cl(5)-Ta(2)-C(30)-C(29)	116.7(4)
C(28)-Ta(2)-C(30)-C(29)	-37.4(4)

Cl(6)-Ta(2)-C(30)-C(29)	-159.5(4)
C(32)-Ta(2)-C(30)-C(29)	-79.0(4)
Cl(4)-Ta(2)-C(30)-C(29)	33.7(4)
C(31)-Ta(2)-C(30)-C(29)	-115.0(6)
C(21)-Ta(2)-C(30)-C(29)	-61.1(14)
N(22)-Ta(2)-C(30)-C(31)	48.8(6)
N(21)-Ta(2)-C(30)-C(31)	-123.3(6)
Cl(5)-Ta(2)-C(30)-C(31)	-128.3(4)
C(28)-Ta(2)-C(30)-C(31)	77.7(4)
Cl(6)-Ta(2)-C(30)-C(31)	-44.5(4)
C(32)-Ta(2)-C(30)-C(31)	36.0(4)
C(29)-Ta(2)-C(30)-C(31)	115.0(6)
Cl(4)-Ta(2)-C(30)-C(31)	148.7(4)
C(21)-Ta(2)-C(30)-C(31)	53.9(15)
N(22)-Ta(2)-C(30)-C(35)	170.1(7)
N(21)-Ta(2)-C(30)-C(35)	-1.9(12)
Cl(5)-Ta(2)-C(30)-C(35)	-6.9(8)
C(28)-Ta(2)-C(30)-C(35)	-161.0(9)
Cl(6)-Ta(2)-C(30)-C(35)	76.9(8)
C(32)-Ta(2)-C(30)-C(35)	157.4(9)
C(29)-Ta(2)-C(30)-C(35)	-123.6(10)
Cl(4)-Ta(2)-C(30)-C(35)	-89.9(8)
C(31)-Ta(2)-C(30)-C(35)	121.4(10)
C(21)-Ta(2)-C(30)-C(35)	175.3(11)
C(29)-C(30)-C(31)-C(32)	1.6(8)
C(35)-C(30)-C(31)-C(32)	172.3(7)
Ta(2)-C(30)-C(31)-C(32)	-62.8(5)
C(29)-C(30)-C(31)-C(36)	-168.1(6)
C(35)-C(30)-C(31)-C(36)	2.6(11)
Ta(2)-C(30)-C(31)-C(36)	127.5(6)
C(29)-C(30)-C(31)-Ta(2)	64.4(5)
C(35)-C(30)-C(31)-Ta(2)	-124.9(8)
N(22)-Ta(2)-C(31)-C(32)	-29.9(5)
N(21)-Ta(2)-C(31)-C(32)	-104.0(5)
Cl(5)-Ta(2)-C(31)-C(32)	167.7(4)
C(28)-Ta(2)-C(31)-C(32)	38.2(4)

Cl(6)-Ta(2)-C(31)-C(32)	-109.4(4)
C(29)-Ta(2)-C(31)-C(32)	80.0(4)
Cl(4)-Ta(2)-C(31)-C(32)	76.0(4)
C(30)-Ta(2)-C(31)-C(32)	117.1(6)
C(21)-Ta(2)-C(31)-C(32)	-50.1(5)
N(22)-Ta(2)-C(31)-C(30)	-147.0(4)
N(21)-Ta(2)-C(31)-C(30)	138.9(5)
Cl(5)-Ta(2)-C(31)-C(30)	50.6(4)
C(28)-Ta(2)-C(31)-C(30)	-78.9(4)
Cl(6)-Ta(2)-C(31)-C(30)	133.5(4)
C(32)-Ta(2)-C(31)-C(30)	-117.1(6)
C(29)-Ta(2)-C(31)-C(30)	-37.1(4)
Cl(4)-Ta(2)-C(31)-C(30)	-41.1(5)
C(21)-Ta(2)-C(31)-C(30)	-167.1(4)
N(22)-Ta(2)-C(31)-C(36)	91.6(7)
N(21)-Ta(2)-C(31)-C(36)	17.5(10)
Cl(5)-Ta(2)-C(31)-C(36)	-70.9(7)
C(28)-Ta(2)-C(31)-C(36)	159.7(8)
Cl(6)-Ta(2)-C(31)-C(36)	12.1(7)
C(32)-Ta(2)-C(31)-C(36)	121.5(9)
C(29)-Ta(2)-C(31)-C(36)	-158.6(8)
Cl(4)-Ta(2)-C(31)-C(36)	-162.6(6)
C(30)-Ta(2)-C(31)-C(36)	-121.5(9)
C(21)-Ta(2)-C(31)-C(36)	71.4(8)
C(30)-C(31)-C(32)-C(28)	-1.4(7)
C(36)-C(31)-C(32)-C(28)	168.1(6)
Ta(2)-C(31)-C(32)-C(28)	-65.7(4)
C(30)-C(31)-C(32)-C(37)	-170.4(6)
C(36)-C(31)-C(32)-C(37)	-0.9(10)
Ta(2)-C(31)-C(32)-C(37)	125.3(7)
C(30)-C(31)-C(32)-Ta(2)	64.3(5)
C(36)-C(31)-C(32)-Ta(2)	-126.2(7)
C(29)-C(28)-C(32)-C(31)	0.7(7)
C(33)-C(28)-C(32)-C(31)	-170.6(7)
Ta(2)-C(28)-C(32)-C(31)	68.5(4)
C(29)-C(28)-C(32)-C(37)	168.8(7)



C(33)-C(28)-C(32)-C(37)	-2.4(12)
Ta(2)-C(28)-C(32)-C(37)	-123.4(7)
C(29)-C(28)-C(32)-Ta(2)	-67.8(4)
C(33)-C(28)-C(32)-Ta(2)	121.0(7)
N(22)-Ta(2)-C(32)-C(31)	152.6(4)
N(21)-Ta(2)-C(32)-C(31)	131.7(4)
Cl(5)-Ta(2)-C(32)-C(31)	-15.9(5)
C(28)-Ta(2)-C(32)-C(31)	-114.6(6)
Cl(6)-Ta(2)-C(32)-C(31)	65.1(4)
C(29)-Ta(2)-C(32)-C(31)	-76.0(4)
Cl(4)-Ta(2)-C(32)-C(31)	-123.8(4)
C(30)-Ta(2)-C(32)-C(31)	-35.9(4)
C(21)-Ta(2)-C(32)-C(31)	147.5(4)
N(22)-Ta(2)-C(32)-C(28)	-92.8(4)
N(21)-Ta(2)-C(32)-C(28)	-113.6(5)
Cl(5)-Ta(2)-C(32)-C(28)	98.7(4)
Cl(6)-Ta(2)-C(32)-C(28)	179.7(4)
C(29)-Ta(2)-C(32)-C(28)	38.6(4)
Cl(4)-Ta(2)-C(32)-C(28)	-9.2(4)
C(30)-Ta(2)-C(32)-C(28)	78.7(4)
C(31)-Ta(2)-C(32)-C(28)	114.6(6)
C(21)-Ta(2)-C(32)-C(28)	-97.9(4)
N(22)-Ta(2)-C(32)-C(37)	32.2(7)
N(21)-Ta(2)-C(32)-C(37)	11.4(9)
Cl(5)-Ta(2)-C(32)-C(37)	-136.3(6)
C(28)-Ta(2)-C(32)-C(37)	125.0(8)
Cl(6)-Ta(2)-C(32)-C(37)	-55.3(6)
C(29)-Ta(2)-C(32)-C(37)	163.6(8)
Cl(4)-Ta(2)-C(32)-C(37)	115.8(6)
C(30)-Ta(2)-C(32)-C(37)	-156.3(8)
C(31)-Ta(2)-C(32)-C(37)	-120.4(8)
C(21)-Ta(2)-C(32)-C(37)	27.1(7)

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Observed and calculated structure factors for (C<sub>5</sub>Me<sub>5</sub>)Ta(hpp)Cl<sub>3</sub>

h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s
2	0	0	0	153	1	20	4	0	17	16	15	6	10	0	112	103	3	36	2	1	160	156	5	-15	5	1	314	316	2
4	0	0	34	1658	11	22	4	0	165	170	2	8	10	0	214	207	3	38	2	1	40	10	18	-13	5	1	104	109	2
6	0	0	93	84	1	24	4	0	48	52	7	10	10	0	114	108	4	-37	3	1	32	45	31	-11	5	1	218	222	1
8	0	0	727	665	5	26	4	0	87	89	4	12	10	0	127	127	4	-35	3	1	123	121	3	-9	5	1	24	37	3
10	0	0	126	138	1	28	4	0	38	41	17	14	10	0	93	77	10	-33	3	1	16	17	16	-7	5	1	174	174	1
12	0	0	678	655	4	30	4	0	77	81	5	16	10	0	103	104	5	-31	3	1	107	112	5	-5	5	1	152	162	1
14	0	0	190	196	2	32	4	0	36	41	15	18	10	0	81	80	7	-29	3	1	0	10	1	-3	5	1	255	256	1
16	0	0	571	571	4	34	4	0	98	99	4	1	11	0	98	93	7	-27	3	1	171	162	4	-1	5	1	9	18	9
18	0	0	70	64	3	36	4	0	38	37	29	3	11	0	124	117	5	-25	3	1	45	41	4	1	5	1	198	197	1
20	0	0	413	415	3	1	5	0	174	175	1	5	11	0	72	62	6	-23	3	1	199	193	2	3	5	1	119	124	1
22	0	0	37	29	9	3	5	0	4	20	4	7	11	0	117	109	4	-21	3	1	18	3	17	5	5	1	157	154	1
24	0	0	346	343	5	5	5	0	154	153	1	9	11	0	15	28	15	-19	3	1	269	260	1	7	5	1	64	61	3
26	0	0	155	162	3	7	5	0	133	137	2	-37	1	1	17	9	16	-17	3	1	111	104	2	9	5	1	190	188	2
28	0	0	71	88	7	9	5	0	161	162	1	-35	1	1	177	185	3	-15	3	1	463	446	2	11	5	1	25	32	4
30	0	0	20	50	20	11	5	0	141	150	1	-33	1	1	32	25	12	-13	3	1	260	258	1	13	5	1	153	154	1
32	0	0	47	46	8	13	5	0	244	239	2	-31	1	1	307	288	3	-11	3	1	516	502	3	15	5	1	100	111	3
34	0	0	54	54	10	15	5	0	274	276	1	-29	1	1	42	42	6	-9	3	1	182	189	1	17	5	1	64	64	2
36	0	0	64	53	7	17	5	0	289	295	2	-27	1	1	517	491	3	-7	3	1	393	390	2	19	5	1	160	161	2
38	0	0	39	40	38	19	5	0	391	379	3	-25	1	1	94	88	2	-5	3	1	35	34	1	21	5	1	137	143	2
1	1	0	8	50	4	21	5	0	328	323	2	-23	1	1	596	561	3	-3	3	1	57	59	1	23	5	1	29	16	11
3	1	0	225	226	7	23	5	0	385	366	3	-21	1	1	196	187	1	-1	3	1	124	128	1	25	5	1	143	137	3
5	1	0	241	224	1	25	5	0	199	196	3	-19	1	1	712	677	3	1	3	1	133	133	1	27	5	1	28	44	16
7	1	0	133	156	1	27	5	0	404	380	5	-17	1	1	202	194	1	3	3	1	134	145	1	29	5	1	90	89	4
9	1	0	24	33	2	29	5	0	65	72	6	-15	1	1	875	812	4	5	3	1	113	128	1	31	5	1	115	124	5
11	1	0	26	30	2	31	5	0	359	338	3	-13	1	1	232	231	1	7	3	1	196	182	2	33	5	1	28	21	28
13	1	0	81	73	1	33	5	0	27	25	27	-11	1	1	703	668	3	9	3	1	11	10	10	-32	6	1	126	129	4
15	1	0	100	116	1	0	6	0	251	233	2	-9	1	1	254	249	1	11	3	1	168	164	1	-30	6	1	56	49	7
17	1	0	185	179	1	2	6	0	44	39	2	-7	1	1	653	618	3	13	3	1	132	130	1	-28	6	1	137	142	3
19	1	0	286	281	1	4	6	0	218	225	1	-5	1	1	322	298	2	15	3	1	64	65	2	-26	6	1	38	37	10
21	1	0	100	95	2	6	6	0	55	62	2	-3	1	1	1038	882	49	17	3	1	136	133	2	-24	6	1	156	164	2
23	1	0	231	239	2	8	6	0	263	266	1	-1	1	1	0	635	1	19	3	1	69	70	2	-22	6	1	20	6	19
25	1	0	110	116	3	10	6	0	62	72	3	1	1	1	0	851	1	21	3	1	58	62	3	-20	6	1	116	118	2
27	1	0	174	170	2	12	6	0	456	449	4	3	1	1	584	535	6	23	3	1	19	24	19	-18	6	1	82	88	4
29	1	0	144	136	2	14	6	0	19	12	13	5	1	1	204	206	1	25	3	1	55	61	4	-16	6	1	50	49	3
31	1	0	154	145	4	16	6	0	283	280	2	7	1	1	88	75	1	27	3	1	32	25	10	-14	6	1	197	205	3
33	1	0	75	70	4	18	6	0	88	81	3	9	1	1	348	344	1	29	3	1	75	81	3	-12	6	1	57	62	6
35	1	0	135	134	3	20	6	0	113	113	2	11	1	1	82	76	1	31	3	1	90	94	3	-10	6	1	247	243	2
37	1	0	27	47	27	22	6	0	87	95	4	13	1	1	132	133	2	33	3	1	83	89	5	-8	6	1	115	126	1
0	2	0	344	348	2	24	6	0	133	133	3	15	1	1	38	47	2	35	3	1	120	127	4	-6	6	1	197	205	1
2	2	0	160	147	1	26	6	0	77	84	5	17	1	1	90	96	3	37	3	1	70	66	7	-4	6	1	247	255	2
4	2	0	676	617	5	28	6	0	127	117	5	19	1	1	169	169	1	-36	4	1	137	145	6	-2	6	1	180	188	1
6	2	0	134	148	1	30	6	0	117	120	5	21	1	1	134	132	1	-34	4	1	234	229	3	0	6	1	414	404	2
8	2	0	686	655	4	32	6	0	38	45	16	23	1	1	147	157	2	-32	4	1	138	133	5	2	6	1	223	208	1
10	2	0	173	179	1	1	7	0	72	69	3	25	1	1	116	119	2	-30	4	1	217	214	3	4	6	1	408	405	2
12	2	0	677	639	3	3	7	0	95	94	2	27	1	1	219	219	2	-28	4	1	190	195	3	6	6	1	149	151	1
14	2	0	146	154	1	5	7	0	241	233	4	29	1	1	188	186	2	-26	4	1	61	61	4	8	6	1	354	350	2
16	2	0	449	445	2	7	7	0	95	94	3	31	1	1	329	324	2	-24	4	1	113	115	3	10	6	1	35	44	3
18	2	0	172	172	1	9	7	0	242	232	4	33	1	1	104	115	3	-22	4	1	23	30	9	12	6	1	286	278	2
20	2	0	196	202	1	11	7	0	46	48	4	35	1	1	263	253	3	-20	4	1	32	29	4	14	6	1	16	27	15
22	2	0	102	104	2	13	7	0	168	163	2	37	1	1	93	97	5	-18	4	1	56	56	2	16	6	1	375	364	3
24	2	0	150	155	2	15	7	0	62	70	4	-38	2	1	134	135	4	-16	4	1	0	13	1	18	6	1	58	63	4
26	2	0	104	109	2	17	7	0	133	137	5	-36	2	1	92	86	5	-14	4	1	17	18	7	20	6	1	358	352	3
28	2	0	35	42	7	19	7	0	135	135	2	-34	2	1	134	139	4	-12	4	1	58	61	2	22	6	1	83	83	5
30	2	0	14	12	14	21	7	0	133	131	5	-32	2	1	136	133	3	-10	4	1	37	45	2	24	6	1	297	287	2
32	2	0	22	24	22	23	7	0	173	166	3	-30	2	1	142	150	3	-8	4	1	330	320	1	26	6	1	123	123	3
34	2	0	24	29	23	25	7	0	59	53	6	-28	2	1	219	216	2	-6	4	1	42	46	2	28	6	1	221	212	3
36	2	0	58	60	8	27	7	0	129	131	4	-26	2	1	173	179	2	-4	4	1	377	379	2	30	6	1	101	105	5
38	2	0	0	10	1	29	7	0	0	41	1	-24	2	1	97	99	3	-2	4	1	280	275	1	32	6	1	146	149	4
1	3	0	96	96	1	0	8	0	520	494	5	-22	2	1	288	284	2	0	4	1	570	55							

h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s
0	4	0	139	143	1	11	9	0	33	31	10	16	2	1	534	515	4	-35	5	1	32	37	19	9	7	1	273	262	4
2	4	0	18	12	5	13	9	0	59	62	5	18	2	1	259	263	1	-33	5	1	128	126	4	11	7	1	52	57	4
4	4	0	24	12	7	15	9	0	59	45	6	20	2	1	320	316	2	-31	5	1	69	68	5	13	7	1	190	189	4
6	4	0	32	39	4	17	9	0	95	89	5	22	2	1	49	59	5	-29	5	1	66	70	7	15	7	1	51	51	4
8	4	0	124	124	2	19	9	0	109	113	4	24	2	1	314	310	2	-27	5	1	97	97	6	17	7	1	51	45	4
10	4	0	76	92	2	21	9	0	72	74	6	26	2	1	81	87	3	-25	5	1	37	47	9	19	7	1	109	109	3
12	4	0	19	7	5	23	9	0	99	102	6	28	2	1	262	255	2	-23	5	1	35	40	7	21	7	1	54	56	5
14	4	0	117	115	1	0	10	0	223	212	4	30	2	1	2	16	1	-21	5	1	116	114	3	23	7	1	130	133	3
16	4	0	15	15	14	2	10	0	3	35	3	32	2	1	177	172	5	-19	5	1	109	107	2	25	7	1	0	24	1
18	4	0	184	178	1	4	10	0	255	235	3	34	2	1	32	9	26	-17	5	1	100	106	3	27	7	1	171	175	4
29	7	1	36	43	20	-28	0	2	327	314	5	-8	2	2	44	50	2	16	4	2	53	49	3	-15	7	2	308	299	2
-26	8	1	70	74	10	-26	0	2	465	447	8	-6	2	2	50	45	1	18	4	2	12	11	12	-13	7	2	93	92	2
-24	8	1	92	85	5	-24	0	2	463	454	4	-4	2	2	42	41	1	20	4	2	46	52	3	-11	7	2	333	324	5
-22	8	1	159	157	4	-22	0	2	349	343	3	-2	2	2	126	135	1	22	4	2	54	56	4	-9	7	2	30	27	8
-20	8	1	120	107	4	-20	0	2	410	413	3	0	2	2	204	205	1	24	4	2	119	120	3	-7	7	2	212	203	2
-18	8	1	176	171	4	-18	0	2	558	542	6	2	2	2	301	297	1	26	4	2	22	6	21	-5	7	2	143	147	2
-16	8	1	41	31	6	-16	0	2	530	503	3	4	2	2	239	239	1	28	4	2	0	18	1	-3	7	2	264	260	2
-14	8	1	151	151	4	-14	0	2	374	355	2	6	2	2	317	309	1	30	4	2	23	19	23	-1	7	2	134	129	2
-12	8	1	26	14	12	-12	0	2	164	185	1	8	2	2	131	145	1	32	4	2	25	27	24	1	7	2	338	333	3
-10	8	1	201	195	2	-10	0	2	155	164	2	10	2	2	231	240	1	34	4	2	28	40	28	3	7	2	35	32	4
-8	8	1	36	33	5	-8	0	2	384	379	2	12	2	2	484	466	2	36	4	2	0	44	1	5	7	2	319	311	3
-6	8	1	168	160	3	-6	0	2	119	116	2	14	2	2	151	154	1	-35	5	2	40	32	15	7	7	2	189	184	2
-4	8	1	4	1	4	-4	0	2	0	58	1	16	2	2	663	638	3	-33	5	2	27	5	24	9	7	2	124	131	5
-2	8	1	98	96	3	-2	0	2	0	11	1	18	2	2	132	145	3	-31	5	2	50	47	9	11	7	2	295	289	2
0	8	1	22	17	16	0	0	2	0	49	1	20	2	2	417	410	2	-29	5	2	46	51	6	13	7	2	0	11	1
2	8	1	186	185	2	2	0	2	8	123	8	22	2	2	10	16	10	-27	5	2	162	172	3	15	7	2	240	232	3
4	8	1	24	7	11	4	0	2	429	427	8	24	2	2	243	243	2	-25	5	2	0	26	1	17	7	2	33	35	9
6	8	1	208	205	3	6	0	2	42	14	3	26	2	2	28	25	8	-23	5	2	338	332	3	19	7	2	189	184	3
8	8	1	114	122	2	8	0	2	156	143	2	28	2	2	336	322	2	-21	5	2	20	29	15	21	7	2	67	73	4
10	8	1	0	8	1	10	0	2	123	127	1	30	2	2	41	41	6	-19	5	2	351	346	3	23	7	2	151	152	3
12	8	1	145	136	2	12	0	2	277	288	2	32	2	2	322	306	4	-17	5	2	41	44	10	25	7	2	76	57	6
14	8	1	58	57	5	14	0	2	130	132	4	34	2	2	54	63	6	-15	5	2	328	330	2	27	7	2	111	102	7
16	8	1	91	89	3	16	0	2	543	553	4	36	2	2	243	241	3	-13	5	2	20	13	5	29	7	2	16	22	15
18	8	1	48	54	8	18	0	2	25	41	7	-37	3	2	88	79	7	-11	5	2	462	447	2	-28	8	2	175	167	7
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-21	5	31	66	75	9	-26	2	32	27	13	26	-2	6	32	36	52	36	-17	5	33	54	45	10	-21	3	34	68	43	16
-19	5	31	76	86	8	-24	2	32	212	208	4	0	6	32	21	7	20	-15	5	33	0	54	1	-19	3	34	232	223	5
-17	5	31	84	88	5	-22	2	32	31	8	30	-2	6	32	86	76	14	-13	5	33	105	106	9	-17	3	34	48	55	8
-15	5	31	21	40	20	-20	2	32	248	248	5	4	6	32	24	41	24	-11	5	33	51	61	7	-15	3	34	172	174	3
-13	5	31	33	34	18	-18	2	32	38	4	22	6	6	32	111	115	19	-9	5	33	110	116	9	-13	3	34	28	31	17
-11	5	31	20	46	19	-16	2	32	201	192	4	-25	1	33	0	14	1	-7	5	33	52	51	10	-11	3	34	77	82	9
-9	5	31	41	8	12	-14	2	32	59	45	7	-23	1	33	58	68	9	-5	5	33	43	62	10	-9	3	34	31	21	31
-7	5	31	120	120	4	-12	2	32	111	109	3	-21	1</																



	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s	h	k	l	Fo	Fc	s		
	2	6	31	20	12	19	-15	3	32	236	226	6	-22	2	33	69	68	6	-16	0	34	199	200	6	0	4	34	52	53	8		
	4	6	31	177	178	8	-13	3	32	226	212	5	-20	2	33	188	185	3	-14	0	34	114	120	6	2	4	34	16	6	16		
	6	6	31	62	63	7	-11	3	32	190	186	3	-18	2	33	50	50	8	-12	0	34	345	331	6	4	4	34	6	15	5		
	8	6	31	176	173	11	-9	3	32	181	173	5	-16	2	33	255	240	3	-10	0	34	123	121	3	6	4	34	59	39	38		
-11	7	31	73	96	25		-7	3	32	155	155	3	-14	2	33	25	13	24	-8	0	34	460	435	15	8	4	34	26	17	26		
-9	7	31	166	172	7		-5	3	32	228	226	3	-12	2	33	220	209	3	-6	0	34	207	199	3	10	4	34	11	40	10		
-7	7	31	101	113	9		-3	3	32	162	164	3	-10	2	33	14	22	14	-4	0	34	443	411	5	-17	5	34	81	86	11		
-5	7	31	149	156	5		-1	3	32	318	302	4	-8	2	33	218	212	3	-2	0	34	137	139	6	-15	5	34	121	131	7		
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	1	7	31	82	93	8		5	3	32	137	135	6	-2	2	33	0	20	1	4	0	34	297	282	4	-9	5	34	49	65	10	
	3	7	31	84	99	10		7	3	32	258	238	3	0	2	33	138	138	3	6	0	34	42	25	10	-7	5	34	20	2	19	
-5	5	34	30	21	30		-1	3	35	133	137	7	-1	1	36	22	32	22	-11	1	37	200	202	3	-17	1	38	14	17	14		
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	1	5	34	42	61	13		5	3	35	15	31	14		5	1	36	34	6	12	-5	1	37	4	46	3	-11	1	38	66	72	6
	3	5	34	22	55	22		7	3	35	38	39	17		7	1	36	46	51	7	-3	1	37	234	228	5	-9	1	38	7	27	6
	5	5	34	91	102	7		9	3	35	0	12	1		9	1	36	57	44	11	-1	1	37	30	17	26	-7	1	38	58	60	7
	7	5	34	0	47	1		11	3	35	30	10	21		11	1	36	30	46	14	1	1	37	284	271	4	-5	1	38	58	52	7
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-6	6	34	46	31	19		-16	4	35	49	36	10		-18	2	36	114	117	7	5	1	37	291	284	4	-1	1	38	10	8	10	
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-2	6	34	30	32	29		-12	4	35	113	113	4		-14	2	36	62	65	7	9	1	37	275	271	4	3	1	38	19	19	18	
-23	1	35	271	256	6		-10	4	35	106	110	4		-12	2	36	0	30	1	-18	2	37	166	168	4	5	1	38	91	93	6	
-21	1	35	0	7	1		-8	4	35	135	138	6		-10	2	36	0	19	1	-16	2	37	79	83	7	7	1	38	26	27	26	
-19	1	35	305	283	4		-6	4	35	92	100	7		-8	2	36	9	37	8	-14	2	37	168	164	4	-16	2	38	65	36	9	
-17	1	35	30	45	30		-4	4	35	189	194	4		-6	2	36	60	64	6	-12	2	37	113	106	8	-14	2	38	199	192	6	
-15	1	35	310	289	4		-2	4	35	83	90	6		-4	2	36	0	35	1	-10	2	37	159	159	4	-12	2	38	122	110	7	
-13	1	35	115	116	3		0	4	35	273	269	8		-2	2	36	117	109	4	-8	2	37	116	110	9	-10	2	38	207	196	4	
-11	1	35	283	267	3		2	4	35	55	70	28		0	2	36	15	26	14	-6	2	37	201	198	4	-8	2	38	127	131	5	
-9	1	35	76	71	7		4	4	35	269	255	5		2	2	36	103	86	6	-4	2	37	138	129	4	-6	2	38	203	197	4	
-7	1	35	305	292	3		6	4	35	124	128	6		4	2	36	94	100	4	-2	2	37	120	124	5	-4	2	38	134	131	5	
-5	1	35	59	62	7		8	4	35	297	274	5		6	2	36	54	50	13	0	2	37	33	53	16	-2	2	38	185	169	6	
-3	1	35	233	222	4		-13	5	35	0	27	1		8	2	36	143	143	4	2	2	37	31	11	18	0	2	38	178	166	5	
-1	1	35	92	86	15		-11	5	35	29	36	28		10	2	36	70	80	5	4	2	37	42	27	41	2	2	38	125	128	6	
	1	35	123	119	3		-9	5	35	91	71	7		-19	3	36	175	173	7	6	2	37	40	9	40	4	2	38	114	122	9	
	3	1	35	20	24	20		-7	5	35	25	36	24		-17	3	36	0	29	1	8	2	37	35	26	17	-13	3	38	31	20	31
	5	1	35	93	93	3		-5	5	35	0	47	1		-15	3	36	278	270	6	-17	3	37	41	47	40	-11	3	38	0	13	1
	7	1	35	0	6	1		-3	5	35	40	57	19		-13	3	36	44	3	10	-15	3	37	64	67	13	-9	3	38	0	11	1
	9	1	35	23	29	23		-1	5	35	49	24	49		-11	3	36	283	259	3	-13	3	37	44	37	10	-7	3	38	25	6	25
	11	1	35	32	36	13		1	5	35	76	69	13		-9	3	36	23	25	23	-11	3	37	95	105	6	-5	3	38	34	13	33
	13	1	35	54	55	15		3	5	35	15	24	15		-7	3	36	258	248	4	-9	3	37	37	45	17	-3	3	38	37	43	22
-22	2	35	32	19	31		-22	0	36	122	139	12		-5	3	36	37	36	37	-7	3	37	125	126	6	-1	3	38	23	29	23	
-20	2	35	18	17	17		-20	0	36	104	106	9		-3	3	36	311	295	4	-5	3	37	68	75	14	1	3	38	116	122	6	
-18	2	35	0	30	1		-18	0	36	66	87	28		-1	3	36	79	73	11	-3	3	37	161	170	4	-13	1	39	96	101	33	
-16	2	35	59	74	9		-16	0	36	250	220	8		1	3	36	230	230	5	-1	3	37	52	55	11	-11	1	39	245	215	7	
-14	2	35	63	66	6		-14	0	36	37	19	25		-3	3	36	33	53	33	1	3	37	138	141	6	-9	1	39	0	46	1	
-12	2	35	95	104	6		-12	0	36	125	122	10		5	3	36	202	193	4	3	3	37	41	42	17	-7	1	39	141	147	5	
-10	2	35	12	19	12		-10	0	36	0	17	1		7	3	36	52	13	21	5	3	37	132	138	5	-5	1	39	52	48	9	
-8	2	35	23	56	23		-8	0	36	0	11	1		9	3	36	251	223	6	-12	4	37	147	130	5	-3	1	39	126	120	6	
-6	2	35	10	13	10		-6	0	36	73	59	7		-16	4	36	0	17	1	-10	4	37	133	128	5	-1	1	39	0	20	1	
-4	2	35	112	117	4		-4	0	36	22	20	21		-14	4	36	55	54	9	-8	4	37	82	92	13	1	1	39	79	72	15	
-2	2	35	80	84	6		-2	0	36	0	21	1		-12	4	36	30	40	30	-6	4	37	94	96	10	3	1	39	37	22	36	
	0	2	35	217	207	9		0	0	36	85	91	5		-10	4	36	38	50	15	-4	4	37	90	86	7	-12	2	39	15	28	15
	2	2	35	81	78	7		2	0	36	59	40	8		-8	4	36	74	71	14	-2	4	37	99	94	6	-10	2	39	78	84	6
	4	2	35	176	172	9		4	0	36	193	196	14		-6	4	36	90	74	6	0	4	37	95	89	6	-8	2	39	49	36	9
	6	2	35	0	16	1		6	0	36	35	44																				

## APPENDIX F

CRYSTALLOGRAPHIC DATA FOR  $(C_5Me_5)Ta(hpp)Cl_2$

Identification code	mes1022	
Empirical formula	C17 H27 Cl2 N3 Ta	
Formula weight	525.27	
Temperature	190(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P1	
Unit cell dimensions	a = 8.7191(10) Å	$\alpha = 83.847(5)^\circ$ .
	b = 9.2140(10) Å	$\beta = 73.764(5)^\circ$ .
	c = 13.0122(14) Å	$\gamma = 71.654(5)^\circ$ .
Volume	952.46(18) Å <sup>3</sup>	
Z	2	
Density (calculated)	1.832 Mg/m <sup>3</sup>	
Absorption coefficient	6.053 mm <sup>-1</sup>	
F(000)	514	
Crystal size	0.12 x 0.07 x 0.03 mm <sup>3</sup>	
Theta range for data collection	1.63 to 27.91°.	
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -17 ≤ l ≤ 16	
Reflections collected	8387	
Independent reflections	8387 [R(int) = 0.0000]	
Completeness to theta = 27.91°	99.4 %	
Max. and min. transmission	0.8393 and 0.5303	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	8387 / 3 / 341	
Goodness-of-fit on F <sup>2</sup>	1.099	
Final R indices [I > 2σ(I)]	R1 = 0.0416, wR2 = 0.1070	
R indices (all data)	R1 = 0.0634, wR2 = 0.1478	
Absolute structure parameter	0.50(3)	
Largest diff. peak and hole	2.610 and -2.591 e.Å <sup>-3</sup>	

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1022.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

	x	y	z	$U(\text{eq})$
Ta(1)	3088(1)	8217(1)	3030(1)	24(1)
Cl(1)	2522(15)	5820(11)	3558(8)	38(2)
N(1)	5180(30)	8560(30)	1870(20)	26(5)
C(1)	6250(30)	7520(30)	2342(19)	16(5)
Ta(2)	227(1)	3724(1)	8304(1)	25(1)
Cl(2)	1914(13)	8278(13)	1536(8)	41(3)
C(2)	6500(20)	6010(20)	3862(13)	39(4)
N(2)	5520(30)	7040(30)	3220(20)	19(4)
Cl(3)	786(16)	6167(13)	7807(8)	39(3)
C(3)	8247(16)	5705(16)	3506(11)	27(3)
N(3)	7960(40)	7010(30)	1800(20)	28(7)
Cl(4)	1455(14)	3616(11)	9773(7)	38(2)
C(4)	9030(40)	5860(40)	2430(30)	69(11)
C(5)	8520(40)	7570(30)	750(20)	26(6)
C(6)	7210(30)	8050(30)	130(20)	20(4)
C(7)	5580(40)	9190(30)	820(30)	28(7)
C(8)	2990(50)	10180(30)	3990(30)	25(7)
C(9)	1860(30)	10830(30)	3270(20)	14(4)
C(10)	540(30)	10260(40)	3660(30)	32(8)
C(11)	680(40)	9370(40)	4540(30)	29(8)
C(12)	2240(40)	9360(30)	4800(30)	45(8)
C(13)	4400(40)	10730(40)	4060(30)	41(7)
C(14)	2130(40)	12010(30)	2450(30)	54(11)
C(15)	-1020(20)	10870(20)	3298(16)	42(5)
C(16)	-640(50)	8640(40)	5250(30)	60(10)
C(17)	3070(50)	8500(40)	5660(20)	55(11)
N(21)	-1770(40)	3240(30)	9450(20)	30(8)
C(21)	-3040(40)	4310(30)	9100(20)	35(7)
N(22)	-2160(40)	4710(40)	8020(20)	40(7)
C(22)	-3073(18)	6313(18)	7584(12)	28(3)
N(23)	-4550(30)	4910(30)	9530(20)	31(7)

C(23)	-4810(30)	6750(30)	8017(17)	65(5)
C(24)	-5650(30)	6100(30)	9050(20)	34(6)
C(25)	-5260(50)	4250(50)	10620(30)	54(11)
C(26)	-3930(50)	3720(40)	11190(30)	66(10)
C(27)	-2390(50)	2760(40)	10610(30)	46(11)
C(28)	350(40)	1620(40)	7350(30)	29(7)
C(29)	1410(50)	1020(40)	7910(30)	46(8)
C(30)	2880(50)	1650(40)	7590(30)	27(9)
C(31)	2560(40)	2750(30)	6720(20)	31(7)
C(32)	1020(30)	2780(30)	6574(19)	15(4)
C(33)	-1300(40)	1320(40)	7480(30)	34(7)
C(34)	1280(40)	-80(30)	8890(20)	43(9)
C(35)	4380(20)	1440(20)	8052(14)	33(4)
C(36)	3690(30)	3580(30)	6140(30)	26(5)
C(37)	370(40)	3540(30)	5660(20)	33(6)

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Bond lengths [Å] and angles [°] for mes1022.

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Ta(1)-N(2)	2.13(3)
Ta(1)-N(1)	2.10(3)
Ta(1)-C(8)	2.28(3)
Ta(1)-C(9)	2.33(3)
Ta(1)-Cl(1)	2.400(10)
Ta(1)-Cl(2)	2.425(9)
Ta(1)-C(10)	2.42(3)
Ta(1)-C(12)	2.46(3)
Ta(1)-C(11)	2.47(3)
Ta(1)-C(1)	2.54(2)
N(1)-C(1)	1.35(3)
N(1)-C(7)	1.42(4)
C(1)-N(2)	1.25(4)
C(1)-N(3)	1.41(4)
Ta(2)-N(21)	2.08(3)
Ta(2)-N(22)	2.12(3)
Ta(2)-C(32)	2.34(2)
Ta(2)-C(28)	2.37(3)
Ta(2)-Cl(4)	2.419(9)
Ta(2)-C(29)	2.43(3)
Ta(2)-Cl(3)	2.434(11)
Ta(2)-C(31)	2.47(3)
Ta(2)-C(30)	2.50(4)
Ta(2)-C(21)	2.64(3)
C(2)-C(3)	1.41(2)
C(2)-N(2)	1.43(3)
C(2)-H(2A)	0.9500
C(3)-C(4)	1.39(4)
C(3)-H(3A)	0.9500
N(3)-C(5)	1.42(4)
N(3)-C(4)	1.51(4)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.51(3)

C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-C(7)	1.58(4)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(8)-C(12)	1.36(5)
C(8)-C(13)	1.49(5)
C(8)-C(9)	1.50(4)
C(9)-C(10)	1.37(4)
C(9)-C(14)	1.46(4)
C(10)-C(11)	1.34(5)
C(10)-C(15)	1.48(3)
C(11)-C(12)	1.49(4)
C(11)-C(16)	1.55(5)
C(12)-C(17)	1.51(5)
C(13)-H(13A)	0.9800
C(13)-H(13B)	0.9800
C(13)-H(13C)	0.9800
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
N(21)-C(21)	1.38(4)
N(21)-C(27)	1.53(4)
C(21)-N(23)	1.25(4)
C(21)-N(22)	1.48(4)

N(22)-C(22)	1.56(4)
C(22)-C(23)	1.40(3)
C(22)-H(22A)	0.9900
C(22)-H(22B)	0.9900
N(23)-C(24)	1.43(4)
N(23)-C(25)	1.53(4)
C(23)-C(24)	1.50(3)
C(23)-H(23A)	0.9500
C(24)-H(24A)	0.9500
C(25)-C(26)	1.47(5)
C(25)-H(25A)	0.9900
C(25)-H(25B)	0.9900
C(26)-C(27)	1.41(5)
C(26)-H(26A)	0.9900
C(26)-H(26B)	0.9900
C(27)-H(27A)	0.9900
C(27)-H(27B)	0.9900
C(28)-C(29)	1.28(5)
C(28)-C(33)	1.51(4)
C(28)-C(32)	1.52(4)
C(29)-C(30)	1.50(5)
C(29)-C(34)	1.55(4)
C(30)-C(31)	1.47(5)
C(30)-C(35)	1.54(4)
C(31)-C(32)	1.40(4)
C(31)-C(36)	1.44(4)
C(32)-C(37)	1.48(4)
C(33)-H(33A)	0.9800
C(33)-H(33B)	0.9800
C(33)-H(33C)	0.9800
C(34)-H(34A)	0.9800
C(34)-H(34B)	0.9800
C(34)-H(34C)	0.9800
C(35)-H(35A)	0.9800
C(35)-H(35B)	0.9800
C(35)-H(35C)	0.9800



C(36)-H(36A)	0.9800
C(36)-H(36B)	0.9800
C(36)-H(36C)	0.9800
C(37)-H(37A)	0.9800
C(37)-H(37B)	0.9800
C(37)-H(37C)	0.9800
N(2)-Ta(1)-N(1)	61.1(10)
N(2)-Ta(1)-C(8)	89.5(12)
N(1)-Ta(1)-C(8)	90.2(11)
N(2)-Ta(1)-C(9)	123.0(9)
N(1)-Ta(1)-C(9)	91.7(9)
C(8)-Ta(1)-C(9)	38.0(10)
N(2)-Ta(1)-Cl(1)	85.1(7)
N(1)-Ta(1)-Cl(1)	126.7(7)
C(8)-Ta(1)-Cl(1)	132.2(9)
C(9)-Ta(1)-Cl(1)	141.4(7)
N(2)-Ta(1)-Cl(2)	130.0(8)
N(1)-Ta(1)-Cl(2)	85.6(7)
C(8)-Ta(1)-Cl(2)	129.5(9)
C(9)-Ta(1)-Cl(2)	91.7(7)
Cl(1)-Ta(1)-Cl(2)	87.2(4)
N(2)-Ta(1)-C(10)	145.8(10)
N(1)-Ta(1)-C(10)	122.8(11)
C(8)-Ta(1)-C(10)	57.8(12)
C(9)-Ta(1)-C(10)	33.4(9)
Cl(1)-Ta(1)-C(10)	108.5(9)
Cl(2)-Ta(1)-C(10)	82.9(8)
N(2)-Ta(1)-C(12)	91.3(11)
N(1)-Ta(1)-C(12)	119.7(10)
C(8)-Ta(1)-C(12)	33.1(11)
C(9)-Ta(1)-C(12)	58.2(10)
Cl(1)-Ta(1)-C(12)	99.4(8)
Cl(2)-Ta(1)-C(12)	138.7(8)
C(10)-Ta(1)-C(12)	56.2(11)
N(2)-Ta(1)-C(11)	123.2(10)

N(1)-Ta(1)-C(11)	145.2(9)
C(8)-Ta(1)-C(11)	56.9(12)
C(9)-Ta(1)-C(11)	55.8(10)
Cl(1)-Ta(1)-C(11)	87.3(8)
Cl(2)-Ta(1)-C(11)	105.6(8)
C(10)-Ta(1)-C(11)	31.9(11)
C(12)-Ta(1)-C(11)	35.2(10)
N(2)-Ta(1)-C(1)	29.3(9)
N(1)-Ta(1)-C(1)	32.0(9)
C(8)-Ta(1)-C(1)	92.1(11)
C(9)-Ta(1)-C(1)	112.0(8)
Cl(1)-Ta(1)-C(1)	104.8(6)
Cl(2)-Ta(1)-C(1)	108.7(6)
C(10)-Ta(1)-C(1)	145.2(9)
C(12)-Ta(1)-C(1)	108.7(10)
C(11)-Ta(1)-C(1)	144.0(9)
C(1)-N(1)-C(7)	126(3)
C(1)-N(1)-Ta(1)	92.2(18)
C(7)-N(1)-Ta(1)	139(2)
N(2)-C(1)-N(1)	112(2)
N(2)-C(1)-N(3)	128(3)
N(1)-C(1)-N(3)	120(2)
N(2)-C(1)-Ta(1)	56.7(15)
N(1)-C(1)-Ta(1)	55.8(15)
N(3)-C(1)-Ta(1)	169(2)
N(21)-Ta(2)-N(22)	64.0(11)
N(21)-Ta(2)-C(32)	119.9(10)
N(22)-Ta(2)-C(32)	85.0(10)
N(21)-Ta(2)-C(28)	86.9(12)
N(22)-Ta(2)-C(28)	85.0(12)
C(32)-Ta(2)-C(28)	37.7(10)
N(21)-Ta(2)-Cl(4)	86.1(8)
N(22)-Ta(2)-Cl(4)	136.3(9)
C(32)-Ta(2)-Cl(4)	138.7(6)
C(28)-Ta(2)-Cl(4)	126.6(9)
N(21)-Ta(2)-C(29)	89.2(13)

N(22)-Ta(2)-C(29)	113.6(12)
C(32)-Ta(2)-C(29)	56.6(9)
C(28)-Ta(2)-C(29)	31.0(11)
Cl(4)-Ta(2)-C(29)	96.0(8)
N(21)-Ta(2)-Cl(3)	129.3(9)
N(22)-Ta(2)-Cl(3)	88.7(9)
C(32)-Ta(2)-Cl(3)	97.4(6)
C(28)-Ta(2)-Cl(3)	135.0(9)
Cl(4)-Ta(2)-Cl(3)	86.7(3)
C(29)-Ta(2)-Cl(3)	141.5(9)
N(21)-Ta(2)-C(31)	144.7(11)
N(22)-Ta(2)-C(31)	115.9(10)
C(32)-Ta(2)-C(31)	33.6(8)
C(28)-Ta(2)-C(31)	58.8(12)
Cl(4)-Ta(2)-C(31)	106.9(7)
C(29)-Ta(2)-C(31)	57.5(11)
Cl(3)-Ta(2)-C(31)	84.9(8)
N(21)-Ta(2)-C(30)	120.8(12)
N(22)-Ta(2)-C(30)	139.3(12)
C(32)-Ta(2)-C(30)	56.8(10)
C(28)-Ta(2)-C(30)	56.8(12)
Cl(4)-Ta(2)-C(30)	82.8(9)
C(29)-Ta(2)-C(30)	35.4(11)
Cl(3)-Ta(2)-C(30)	107.9(9)
C(31)-Ta(2)-C(30)	34.5(11)
N(21)-Ta(2)-C(21)	31.2(10)
N(22)-Ta(2)-C(21)	33.9(10)
C(32)-Ta(2)-C(21)	109.9(8)
C(28)-Ta(2)-C(21)	91.5(11)
Cl(4)-Ta(2)-C(21)	107.9(6)
C(29)-Ta(2)-C(21)	108.3(11)
Cl(3)-Ta(2)-C(21)	107.3(7)
C(31)-Ta(2)-C(21)	143.6(9)
C(30)-Ta(2)-C(21)	143.7(11)
C(3)-C(2)-N(2)	117.0(16)
C(3)-C(2)-H(2A)	121.5

N(2)-C(2)-H(2A)	121.5
C(1)-N(2)-C(2)	119(2)
C(1)-N(2)-Ta(1)	94.1(19)
C(2)-N(2)-Ta(1)	146.7(17)
C(4)-C(3)-C(2)	121.1(18)
C(4)-C(3)-H(3A)	119.7
C(2)-C(3)-H(3A)	119.2
C(1)-N(3)-C(5)	119(3)
C(1)-N(3)-C(4)	114(3)
C(5)-N(3)-C(4)	126(3)
C(3)-C(4)-N(3)	115(2)
C(3)-C(4)-H(4A)	108.8
N(3)-C(4)-H(4A)	109.3
C(3)-C(4)-H(4B)	108.0
N(3)-C(4)-H(4B)	107.7
H(4A)-C(4)-H(4B)	107.5
N(3)-C(5)-C(6)	114(3)
N(3)-C(5)-H(5A)	108.8
C(6)-C(5)-H(5A)	109.6
N(3)-C(5)-H(5B)	108.2
C(6)-C(5)-H(5B)	108.2
H(5A)-C(5)-H(5B)	107.7
C(5)-C(6)-C(7)	109(2)
C(5)-C(6)-H(6A)	109.4
C(7)-C(6)-H(6A)	109.1
C(5)-C(6)-H(6B)	110.3
C(7)-C(6)-H(6B)	110.8
H(6A)-C(6)-H(6B)	108.5
N(1)-C(7)-C(6)	109(2)
N(1)-C(7)-H(7A)	110.4
C(6)-C(7)-H(7A)	108.8
N(1)-C(7)-H(7B)	110.2
C(6)-C(7)-H(7B)	110.3
H(7A)-C(7)-H(7B)	108.2
C(12)-C(8)-C(13)	123(3)
C(12)-C(8)-C(9)	109(3)

C(13)-C(8)-C(9)	126(3)
C(12)-C(8)-Ta(1)	80.7(18)
C(13)-C(8)-Ta(1)	128(2)
C(9)-C(8)-Ta(1)	72.6(16)
C(10)-C(9)-C(14)	132(3)
C(10)-C(9)-C(8)	105(3)
C(14)-C(9)-C(8)	123(2)
C(10)-C(9)-Ta(1)	77.0(18)
C(14)-C(9)-Ta(1)	125(2)
C(8)-C(9)-Ta(1)	69.4(15)
C(11)-C(10)-C(9)	112(3)
C(11)-C(10)-C(15)	125(3)
C(9)-C(10)-C(15)	122(3)
C(11)-C(10)-Ta(1)	76.3(19)
C(9)-C(10)-Ta(1)	69.6(16)
C(15)-C(10)-Ta(1)	133(2)
C(10)-C(11)-C(12)	108(3)
C(10)-C(11)-C(16)	127(3)
C(12)-C(11)-C(16)	125(3)
C(10)-C(11)-Ta(1)	71.8(19)
C(12)-C(11)-Ta(1)	71.8(17)
C(16)-C(11)-Ta(1)	128(2)
C(8)-C(12)-C(17)	123(3)
C(8)-C(12)-C(11)	105(3)
C(17)-C(12)-C(11)	131(3)
C(8)-C(12)-Ta(1)	66.2(18)
C(17)-C(12)-Ta(1)	119(2)
C(11)-C(12)-Ta(1)	72.9(17)
C(8)-C(13)-H(13A)	109.8
C(8)-C(13)-H(13B)	109.4
H(13A)-C(13)-H(13B)	109.5
C(8)-C(13)-H(13C)	109.2
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(9)-C(14)-H(14A)	109.9
C(9)-C(14)-H(14B)	109.3

H(14A)-C(14)-H(14B)	109.5
C(9)-C(14)-H(14C)	109.2
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(10)-C(15)-H(15A)	110.5
C(10)-C(15)-H(15B)	108.9
H(15A)-C(15)-H(15B)	109.5
C(10)-C(15)-H(15C)	109.1
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(11)-C(16)-H(16A)	108.1
C(11)-C(16)-H(16B)	110.7
H(16A)-C(16)-H(16B)	109.5
C(11)-C(16)-H(16C)	109.6
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(12)-C(17)-H(17A)	109.8
C(12)-C(17)-H(17B)	109.8
H(17A)-C(17)-H(17B)	109.5
C(12)-C(17)-H(17C)	108.8
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(21)-N(21)-C(27)	111(3)
C(21)-N(21)-Ta(2)	97(2)
C(27)-N(21)-Ta(2)	145(2)
N(23)-C(21)-N(21)	133(3)
N(23)-C(21)-N(22)	124(3)
N(21)-C(21)-N(22)	102(3)
N(23)-C(21)-Ta(2)	165(3)
N(21)-C(21)-Ta(2)	51.5(17)
N(22)-C(21)-Ta(2)	53.2(16)
C(21)-N(22)-C(22)	114(2)
C(21)-N(22)-Ta(2)	93(2)
C(22)-N(22)-Ta(2)	134.2(19)
C(23)-C(22)-N(22)	113.0(18)
C(23)-C(22)-H(22A)	109.0

N(22)-C(22)-H(22A)	109.0
C(23)-C(22)-H(22B)	109.0
N(22)-C(22)-H(22B)	108.9
H(22A)-C(22)-H(22B)	107.8
C(21)-N(23)-C(24)	125(3)
C(21)-N(23)-C(25)	116(3)
C(24)-N(23)-C(25)	119(3)
C(22)-C(23)-C(24)	123(2)
C(22)-C(23)-H(23A)	118.4
C(24)-C(23)-H(23A)	118.2
N(23)-C(24)-C(23)	115(2)
N(23)-C(24)-H(24A)	122.6
C(23)-C(24)-H(24A)	122.6
C(26)-C(25)-N(23)	109(3)
C(26)-C(25)-H(25A)	108.8
N(23)-C(25)-H(25A)	109.5
C(26)-C(25)-H(25B)	110.8
N(23)-C(25)-H(25B)	110.7
H(25A)-C(25)-H(25B)	108.2
C(27)-C(26)-C(25)	116(3)
C(27)-C(26)-H(26A)	109.3
C(25)-C(26)-H(26A)	109.2
C(27)-C(26)-H(26B)	107.4
C(25)-C(26)-H(26B)	107.7
H(26A)-C(26)-H(26B)	107.3
C(26)-C(27)-N(21)	116(3)
C(26)-C(27)-H(27A)	109.6
N(21)-C(27)-H(27A)	107.8
C(26)-C(27)-H(27B)	107.6
N(21)-C(27)-H(27B)	108.3
H(27A)-C(27)-H(27B)	107.5
C(29)-C(28)-C(33)	127(3)
C(29)-C(28)-C(32)	107(3)
C(33)-C(28)-C(32)	126(3)
C(29)-C(28)-Ta(2)	77(2)
C(33)-C(28)-Ta(2)	115(2)

C(32)-C(28)-Ta(2)	70.0(15)
C(28)-C(29)-C(30)	112(3)
C(28)-C(29)-C(34)	129(4)
C(30)-C(29)-C(34)	118(3)
C(28)-C(29)-Ta(2)	72(2)
C(30)-C(29)-Ta(2)	75(2)
C(34)-C(29)-Ta(2)	115(2)
C(31)-C(30)-C(29)	105(3)
C(31)-C(30)-C(35)	121(3)
C(29)-C(30)-C(35)	134(3)
C(31)-C(30)-Ta(2)	71.5(18)
C(29)-C(30)-Ta(2)	69.6(19)
C(35)-C(30)-Ta(2)	119(2)
C(32)-C(31)-C(30)	107(3)
C(32)-C(31)-C(36)	129(3)
C(30)-C(31)-C(36)	124(3)
C(32)-C(31)-Ta(2)	68.1(16)
C(30)-C(31)-Ta(2)	74.0(19)
C(36)-C(31)-Ta(2)	124(2)
C(31)-C(32)-C(37)	124(2)
C(31)-C(32)-C(28)	109(2)
C(37)-C(32)-C(28)	126(2)
C(31)-C(32)-Ta(2)	78.3(16)
C(37)-C(32)-Ta(2)	126.5(17)
C(28)-C(32)-Ta(2)	72.2(16)
C(28)-C(33)-H(33A)	109.1
C(28)-C(33)-H(33B)	109.7
H(33A)-C(33)-H(33B)	109.5
C(28)-C(33)-H(33C)	109.6
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
C(29)-C(34)-H(34A)	109.0
C(29)-C(34)-H(34B)	109.8
H(34A)-C(34)-H(34B)	109.5
C(29)-C(34)-H(34C)	109.6
H(34A)-C(34)-H(34C)	109.5



H(34B)-C(34)-H(34C)	109.5
C(30)-C(35)-H(35A)	109.3
C(30)-C(35)-H(35B)	109.5
H(35A)-C(35)-H(35B)	109.5
C(30)-C(35)-H(35C)	109.6
H(35A)-C(35)-H(35C)	109.5
H(35B)-C(35)-H(35C)	109.5
C(31)-C(36)-H(36A)	111.3
C(31)-C(36)-H(36B)	109.3
H(36A)-C(36)-H(36B)	109.5
C(31)-C(36)-H(36C)	107.7
H(36A)-C(36)-H(36C)	109.5
H(36B)-C(36)-H(36C)	109.5
C(32)-C(37)-H(37A)	109.0
C(32)-C(37)-H(37B)	110.3
H(37A)-C(37)-H(37B)	109.5
C(32)-C(37)-H(37C)	109.1
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5

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Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for mes1022. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Ta(1)	24(1)	26(1)	23(1)	3(1)	-7(1)	-8(1)
Cl(1)	49(6)	28(5)	40(5)	6(4)	-7(5)	-20(4)
Ta(2)	26(1)	26(1)	23(1)	3(1)	-8(1)	-7(1)
Cl(2)	35(5)	60(7)	40(6)	3(5)	-19(4)	-23(5)
N(2)	11(8)	22(8)	20(9)	4(7)	-3(6)	-3(6)
Cl(3)	43(7)	38(5)	35(5)	6(4)	-8(5)	-15(5)
N(3)	20(15)	22(11)	38(17)	-2(11)	-2(13)	-4(10)
Cl(4)	52(6)	43(5)	25(4)	4(4)	-20(4)	-17(4)
C(4)	41(15)	78(19)	61(19)	18(14)	-31(13)	30(12)
C(8)	37(11)	17(9)	20(9)	-7(7)	-9(6)	-5(7)
C(10)	7(7)	49(11)	32(10)	-4(6)	-3(5)	0(5)
C(11)	18(14)	30(16)	34(16)	-11(12)	10(11)	-13(12)
C(12)	61(14)	41(14)	39(10)	-19(9)	-18(8)	-10(10)
C(13)	34(11)	32(11)	44(16)	-19(9)	16(9)	-10(8)
C(14)	42(16)	51(18)	50(20)	14(14)	-18(15)	17(13)
C(16)	80(20)	70(20)	37(14)	-10(13)	12(15)	-46(18)
C(17)	50(20)	80(20)	23(14)	-8(13)	-23(14)	3(17)
N(21)	23(9)	40(12)	26(9)	18(6)	-9(6)	-10(7)
C(21)	49(13)	40(12)	19(10)	9(7)	-10(8)	-21(8)
N(22)	40(13)	45(14)	23(10)	0(8)	-6(8)	3(9)
N(23)	13(11)	52(17)	19(14)	5(12)	-2(10)	-3(10)
C(24)	23(11)	60(15)	22(8)	14(8)	-6(7)	-20(11)
C(25)	36(11)	80(20)	34(11)	9(9)	8(7)	-26(10)
C(26)	69(15)	80(20)	33(10)	1(11)	7(8)	-26(13)
C(27)	44(15)	57(15)	41(14)	30(10)	-15(10)	-26(10)
C(29)	62(13)	26(10)	38(16)	1(9)	-8(10)	-2(7)
C(31)	44(15)	28(11)	19(9)	10(7)	-18(9)	-3(10)
C(33)	26(13)	51(14)	30(12)	-11(9)	-12(11)	-12(11)
C(34)	60(20)	16(12)	41(17)	-1(11)	2(14)	-18(12)
C(36)	12(6)	30(9)	25(10)	-9(7)	11(6)	-2(6)
C(37)	36(13)	24(10)	31(13)	1(8)	0(10)	-8(9)

Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1022.

	x	y	z	U(eq)
H(2A)	5990	5558	4503	47
H(3A)	8902	5381	4009	33
H(4A)	9410	4855	2098	82
H(4B)	10038	6175	2385	82
H(5A)	9502	6772	352	31
H(5B)	8901	8465	798	31
H(6A)	6956	7136	-35	23
H(6B)	7633	8549	-559	23
H(7A)	5768	10189	845	34
H(7B)	4642	9357	487	34
H(13A)	3963	11600	4533	61
H(13B)	4948	11035	3342	61
H(13C)	5204	9897	4340	61
H(14A)	1475	13019	2745	81
H(14B)	1773	11853	1831	81
H(14C)	3320	11938	2229	81
H(15A)	-1810	11694	3770	63
H(15B)	-1517	10051	3320	63
H(15C)	-750	11276	2563	63
H(16A)	-1439	9381	5783	91
H(16B)	-82	7728	5618	91
H(16C)	-1234	8359	4806	91
H(17A)	2603	9088	6316	82
H(17B)	4280	8360	5412	82
H(17C)	2874	7502	5792	82
H(22A)	-2621	7091	7755	33
H(22B)	-2828	6282	6795	33
H(23A)	-5486	7504	7639	78

H(24A)	-6826	6456	9355	41
H(25A)	-6221	5039	11041	64
H(25B)	-5646	3379	10533	64
H(26A)	-3727	4632	11403	79
H(26B)	-4342	3174	11856	79
H(27A)	-2500	1717	10620	56
H(27B)	-1529	2691	10987	56
H(33A)	-1127	451	7048	51
H(33B)	-2080	2232	7244	51
H(33C)	-1765	1097	8236	51
H(34A)	1783	-1144	8653	64
H(34B)	100	78	9271	64
H(34C)	1875	115	9372	64
H(35A)	5309	567	7716	49
H(35B)	4059	1254	8827	49
H(35C)	4736	2368	7906	49
H(36A)	4509	2969	5544	39
H(36B)	4283	3788	6623	39
H(36C)	3067	4550	5864	39
H(37A)	923	2896	5028	49
H(37B)	609	4528	5502	49
H(37C)	-837	3713	5833	49

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Torsion angles [°] for mes1022.

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N(2)-Ta(1)-N(1)-C(1)	4.2(15)
C(8)-Ta(1)-N(1)-C(1)	93.5(17)
C(9)-Ta(1)-N(1)-C(1)	131.5(15)
Cl(1)-Ta(1)-N(1)-C(1)	-53.7(17)
Cl(2)-Ta(1)-N(1)-C(1)	-136.9(15)
C(10)-Ta(1)-N(1)-C(1)	144.4(15)
C(12)-Ta(1)-N(1)-C(1)	77.6(18)
C(11)-Ta(1)-N(1)-C(1)	112(2)
N(2)-Ta(1)-N(1)-C(7)	166(3)
C(8)-Ta(1)-N(1)-C(7)	-105(3)
C(9)-Ta(1)-N(1)-C(7)	-67(3)
Cl(1)-Ta(1)-N(1)-C(7)	108(3)
Cl(2)-Ta(1)-N(1)-C(7)	25(3)
C(10)-Ta(1)-N(1)-C(7)	-54(3)
C(12)-Ta(1)-N(1)-C(7)	-121(3)
C(11)-Ta(1)-N(1)-C(7)	-86(3)
C(1)-Ta(1)-N(1)-C(7)	162(4)
C(7)-N(1)-C(1)-N(2)	-172(3)
Ta(1)-N(1)-C(1)-N(2)	-7(2)
C(7)-N(1)-C(1)-N(3)	3(4)
Ta(1)-N(1)-C(1)-N(3)	168(2)
C(7)-N(1)-C(1)-Ta(1)	-165(3)
N(1)-Ta(1)-C(1)-N(2)	172(3)
C(8)-Ta(1)-C(1)-N(2)	85(2)
C(9)-Ta(1)-C(1)-N(2)	118.6(19)
Cl(1)-Ta(1)-C(1)-N(2)	-49.4(19)
Cl(2)-Ta(1)-C(1)-N(2)	-141.5(17)
C(10)-Ta(1)-C(1)-N(2)	113(2)
C(12)-Ta(1)-C(1)-N(2)	56(2)
C(11)-Ta(1)-C(1)-N(2)	57(3)
N(2)-Ta(1)-C(1)-N(1)	-172(3)
C(8)-Ta(1)-C(1)-N(1)	-87.2(17)
C(9)-Ta(1)-C(1)-N(1)	-53.9(17)
Cl(1)-Ta(1)-C(1)-N(1)	138.1(14)

Cl(2)-Ta(1)-C(1)-N(1)	46.0(16)
C(10)-Ta(1)-C(1)-N(1)	-59(2)
C(12)-Ta(1)-C(1)-N(1)	-116.4(17)
C(11)-Ta(1)-C(1)-N(1)	-115.6(19)
N(2)-Ta(1)-C(1)-N(3)	119(11)
N(1)-Ta(1)-C(1)-N(3)	-69(10)
C(8)-Ta(1)-C(1)-N(3)	-156(10)
C(9)-Ta(1)-C(1)-N(3)	-123(10)
Cl(1)-Ta(1)-C(1)-N(3)	69(10)
Cl(2)-Ta(1)-C(1)-N(3)	-23(10)
C(10)-Ta(1)-C(1)-N(3)	-128(10)
C(12)-Ta(1)-C(1)-N(3)	175(10)
C(11)-Ta(1)-C(1)-N(3)	176(10)
N(1)-C(1)-N(2)-C(2)	-175(2)
N(3)-C(1)-N(2)-C(2)	10(4)
Ta(1)-C(1)-N(2)-C(2)	178(3)
N(1)-C(1)-N(2)-Ta(1)	7(2)
N(3)-C(1)-N(2)-Ta(1)	-168(3)
C(3)-C(2)-N(2)-C(1)	5(3)
C(3)-C(2)-N(2)-Ta(1)	-178(3)
N(1)-Ta(1)-N(2)-C(1)	-4.5(17)
C(8)-Ta(1)-N(2)-C(1)	-95(2)
C(9)-Ta(1)-N(2)-C(1)	-76(2)
Cl(1)-Ta(1)-N(2)-C(1)	132.5(18)
Cl(2)-Ta(1)-N(2)-C(1)	50(2)
C(10)-Ta(1)-N(2)-C(1)	-111(2)
C(12)-Ta(1)-N(2)-C(1)	-128.1(19)
C(11)-Ta(1)-N(2)-C(1)	-143.9(17)
N(1)-Ta(1)-N(2)-C(2)	179(4)
C(8)-Ta(1)-N(2)-C(2)	88(3)
C(9)-Ta(1)-N(2)-C(2)	107(3)
Cl(1)-Ta(1)-N(2)-C(2)	-44(3)
Cl(2)-Ta(1)-N(2)-C(2)	-127(3)
C(10)-Ta(1)-N(2)-C(2)	72(4)
C(12)-Ta(1)-N(2)-C(2)	55(3)
C(11)-Ta(1)-N(2)-C(2)	39(4)

C(1)-Ta(1)-N(2)-C(2)	-177(5)
N(2)-C(2)-C(3)-C(4)	-24(3)
N(2)-C(1)-N(3)-C(5)	173(3)
N(1)-C(1)-N(3)-C(5)	-2(4)
Ta(1)-C(1)-N(3)-C(5)	61(11)
N(2)-C(1)-N(3)-C(4)	-7(4)
N(1)-C(1)-N(3)-C(4)	179(3)
Ta(1)-C(1)-N(3)-C(4)	-119(10)
C(2)-C(3)-C(4)-N(3)	27(4)
C(1)-N(3)-C(4)-C(3)	-11(4)
C(5)-N(3)-C(4)-C(3)	169(3)
C(1)-N(3)-C(5)-C(6)	-28(3)
C(4)-N(3)-C(5)-C(6)	152(3)
N(3)-C(5)-C(6)-C(7)	52(3)
C(1)-N(1)-C(7)-C(6)	23(4)
Ta(1)-N(1)-C(7)-C(6)	-134(2)
C(5)-C(6)-C(7)-N(1)	-49(3)
N(2)-Ta(1)-C(8)-C(12)	-93(2)
N(1)-Ta(1)-C(8)-C(12)	-154(2)
C(9)-Ta(1)-C(8)-C(12)	113(3)
Cl(1)-Ta(1)-C(8)-C(12)	-10(3)
Cl(2)-Ta(1)-C(8)-C(12)	121.3(19)
C(10)-Ta(1)-C(8)-C(12)	76(2)
C(11)-Ta(1)-C(8)-C(12)	38(2)
C(1)-Ta(1)-C(8)-C(12)	-122(2)
N(2)-Ta(1)-C(8)-C(13)	31(3)
N(1)-Ta(1)-C(8)-C(13)	-30(3)
C(9)-Ta(1)-C(8)-C(13)	-122(4)
Cl(1)-Ta(1)-C(8)-C(13)	114(3)
Cl(2)-Ta(1)-C(8)-C(13)	-114(3)
C(10)-Ta(1)-C(8)-C(13)	-159(4)
C(12)-Ta(1)-C(8)-C(13)	124(4)
C(11)-Ta(1)-C(8)-C(13)	163(4)
C(1)-Ta(1)-C(8)-C(13)	2(3)
N(2)-Ta(1)-C(8)-C(9)	153.5(18)
N(1)-Ta(1)-C(8)-C(9)	92.4(18)

Cl(1)-Ta(1)-C(8)-C(9)	-123.5(15)
Cl(2)-Ta(1)-C(8)-C(9)	8(2)
C(10)-Ta(1)-C(8)-C(9)	-37.2(17)
C(12)-Ta(1)-C(8)-C(9)	-113(3)
C(11)-Ta(1)-C(8)-C(9)	-75.2(19)
C(1)-Ta(1)-C(8)-C(9)	124.3(17)
C(12)-C(8)-C(9)-C(10)	-4(3)
C(13)-C(8)-C(9)-C(10)	-165(3)
Ta(1)-C(8)-C(9)-C(10)	70(2)
C(12)-C(8)-C(9)-C(14)	168(3)
C(13)-C(8)-C(9)-C(14)	6(5)
Ta(1)-C(8)-C(9)-C(14)	-119(3)
C(12)-C(8)-C(9)-Ta(1)	-73(2)
C(13)-C(8)-C(9)-Ta(1)	125(3)
N(2)-Ta(1)-C(9)-C(10)	-143.8(19)
N(1)-Ta(1)-C(9)-C(10)	160.0(19)
C(8)-Ta(1)-C(9)-C(10)	-112(3)
Cl(1)-Ta(1)-C(9)-C(10)	-13(2)
Cl(2)-Ta(1)-C(9)-C(10)	74.3(18)
C(12)-Ta(1)-C(9)-C(10)	-75.6(19)
C(11)-Ta(1)-C(9)-C(10)	-33.4(18)
C(1)-Ta(1)-C(9)-C(10)	-174.6(17)
N(2)-Ta(1)-C(9)-C(14)	84(2)
N(1)-Ta(1)-C(9)-C(14)	28(2)
C(8)-Ta(1)-C(9)-C(14)	116(3)
Cl(1)-Ta(1)-C(9)-C(14)	-145(2)
Cl(2)-Ta(1)-C(9)-C(14)	-58(2)
C(10)-Ta(1)-C(9)-C(14)	-132(3)
C(12)-Ta(1)-C(9)-C(14)	152(3)
C(11)-Ta(1)-C(9)-C(14)	-165(3)
C(1)-Ta(1)-C(9)-C(14)	53(2)
N(2)-Ta(1)-C(9)-C(8)	-32(2)
N(1)-Ta(1)-C(9)-C(8)	-88.2(19)
Cl(1)-Ta(1)-C(9)-C(8)	98.4(18)
Cl(2)-Ta(1)-C(9)-C(8)	-173.9(18)
C(10)-Ta(1)-C(9)-C(8)	112(3)



C(12)-Ta(1)-C(9)-C(8)	36.1(18)
C(11)-Ta(1)-C(9)-C(8)	78(2)
C(1)-Ta(1)-C(9)-C(8)	-62.9(19)
C(14)-C(9)-C(10)-C(11)	-170(3)
C(8)-C(9)-C(10)-C(11)	1(4)
Ta(1)-C(9)-C(10)-C(11)	65(3)
C(14)-C(9)-C(10)-C(15)	-3(5)
C(8)-C(9)-C(10)-C(15)	168(3)
Ta(1)-C(9)-C(10)-C(15)	-128(3)
C(14)-C(9)-C(10)-Ta(1)	126(3)
C(8)-C(9)-C(10)-Ta(1)	-64.1(18)
N(2)-Ta(1)-C(10)-C(11)	-59(3)
N(1)-Ta(1)-C(10)-C(11)	-144.3(17)
C(8)-Ta(1)-C(10)-C(11)	-78(2)
C(9)-Ta(1)-C(10)-C(11)	-120(3)
Cl(1)-Ta(1)-C(10)-C(11)	50.9(19)
Cl(2)-Ta(1)-C(10)-C(11)	135.5(18)
C(12)-Ta(1)-C(10)-C(11)	-38.2(17)
C(1)-Ta(1)-C(10)-C(11)	-112(2)
N(2)-Ta(1)-C(10)-C(9)	62(3)
N(1)-Ta(1)-C(10)-C(9)	-24(2)
C(8)-Ta(1)-C(10)-C(9)	42.5(18)
Cl(1)-Ta(1)-C(10)-C(9)	171.3(15)
Cl(2)-Ta(1)-C(10)-C(9)	-104.1(17)
C(12)-Ta(1)-C(10)-C(9)	82.1(19)
C(11)-Ta(1)-C(10)-C(9)	120(3)
C(1)-Ta(1)-C(10)-C(9)	9(3)
N(2)-Ta(1)-C(10)-C(15)	176(2)
N(1)-Ta(1)-C(10)-C(15)	91(3)
C(8)-Ta(1)-C(10)-C(15)	157(4)
C(9)-Ta(1)-C(10)-C(15)	115(4)
Cl(1)-Ta(1)-C(10)-C(15)	-74(3)
Cl(2)-Ta(1)-C(10)-C(15)	10(3)
C(12)-Ta(1)-C(10)-C(15)	-163(3)
C(11)-Ta(1)-C(10)-C(15)	-125(4)
C(1)-Ta(1)-C(10)-C(15)	123(2)

C(9)-C(10)-C(11)-C(12)	2(4)
C(15)-C(10)-C(11)-C(12)	-164(3)
Ta(1)-C(10)-C(11)-C(12)	63(2)
C(9)-C(10)-C(11)-C(16)	174(3)
C(15)-C(10)-C(11)-C(16)	8(5)
Ta(1)-C(10)-C(11)-C(16)	-125(3)
C(9)-C(10)-C(11)-Ta(1)	-61(2)
C(15)-C(10)-C(11)-Ta(1)	133(3)
N(2)-Ta(1)-C(11)-C(10)	145.0(18)
N(1)-Ta(1)-C(11)-C(10)	59(3)
C(8)-Ta(1)-C(11)-C(10)	81(2)
C(9)-Ta(1)-C(11)-C(10)	35.1(18)
Cl(1)-Ta(1)-C(11)-C(10)	-132.6(19)
Cl(2)-Ta(1)-C(11)-C(10)	-46.2(19)
C(12)-Ta(1)-C(11)-C(10)	117(3)
C(1)-Ta(1)-C(11)-C(10)	116(2)
N(2)-Ta(1)-C(11)-C(12)	28(2)
N(1)-Ta(1)-C(11)-C(12)	-58(3)
C(8)-Ta(1)-C(11)-C(12)	-35.8(18)
C(9)-Ta(1)-C(11)-C(12)	-81.9(18)
Cl(1)-Ta(1)-C(11)-C(12)	110.5(17)
Cl(2)-Ta(1)-C(11)-C(12)	-163.1(16)
C(10)-Ta(1)-C(11)-C(12)	-117(3)
C(1)-Ta(1)-C(11)-C(12)	-1(3)
N(2)-Ta(1)-C(11)-C(16)	-92(3)
N(1)-Ta(1)-C(11)-C(16)	-178(3)
C(8)-Ta(1)-C(11)-C(16)	-156(4)
C(9)-Ta(1)-C(11)-C(16)	158(3)
Cl(1)-Ta(1)-C(11)-C(16)	-10(3)
Cl(2)-Ta(1)-C(11)-C(16)	76(3)
C(10)-Ta(1)-C(11)-C(16)	123(4)
C(12)-Ta(1)-C(11)-C(16)	-120(4)
C(1)-Ta(1)-C(11)-C(16)	-122(3)
C(13)-C(8)-C(12)-C(17)	-20(5)
C(9)-C(8)-C(12)-C(17)	178(3)
Ta(1)-C(8)-C(12)-C(17)	110(3)

C(13)-C(8)-C(12)-C(11)	167(3)
C(9)-C(8)-C(12)-C(11)	5(3)
Ta(1)-C(8)-C(12)-C(11)	-63(2)
C(13)-C(8)-C(12)-Ta(1)	-130(3)
C(9)-C(8)-C(12)-Ta(1)	68(2)
C(10)-C(11)-C(12)-C(8)	-4(3)
C(16)-C(11)-C(12)-C(8)	-177(3)
Ta(1)-C(11)-C(12)-C(8)	59(2)
C(10)-C(11)-C(12)-C(17)	-176(3)
C(16)-C(11)-C(12)-C(17)	11(5)
Ta(1)-C(11)-C(12)-C(17)	-113(3)
C(10)-C(11)-C(12)-Ta(1)	-63(2)
C(16)-C(11)-C(12)-Ta(1)	125(3)
N(2)-Ta(1)-C(12)-C(8)	87(2)
N(1)-Ta(1)-C(12)-C(8)	30(2)
C(9)-Ta(1)-C(12)-C(8)	-42(2)
Cl(1)-Ta(1)-C(12)-C(8)	172(2)
Cl(2)-Ta(1)-C(12)-C(8)	-91(2)
C(10)-Ta(1)-C(12)-C(8)	-82(2)
C(11)-Ta(1)-C(12)-C(8)	-116(3)
C(1)-Ta(1)-C(12)-C(8)	63(2)
N(2)-Ta(1)-C(12)-C(17)	-29(3)
N(1)-Ta(1)-C(12)-C(17)	-86(3)
C(8)-Ta(1)-C(12)-C(17)	-116(4)
C(9)-Ta(1)-C(12)-C(17)	-157(3)
Cl(1)-Ta(1)-C(12)-C(17)	57(3)
Cl(2)-Ta(1)-C(12)-C(17)	153(2)
C(10)-Ta(1)-C(12)-C(17)	163(3)
C(11)-Ta(1)-C(12)-C(17)	128(4)
C(1)-Ta(1)-C(12)-C(17)	-53(3)
N(2)-Ta(1)-C(12)-C(11)	-156.8(18)
N(1)-Ta(1)-C(12)-C(11)	146.2(17)
C(8)-Ta(1)-C(12)-C(11)	116(3)
C(9)-Ta(1)-C(12)-C(11)	74.4(18)
Cl(1)-Ta(1)-C(12)-C(11)	-71.5(18)
Cl(2)-Ta(1)-C(12)-C(11)	25(2)

C(10)-Ta(1)-C(12)-C(11)	34.5(18)
C(1)-Ta(1)-C(12)-C(11)	179.3(17)
N(22)-Ta(2)-N(21)-C(21)	-12.1(19)
C(32)-Ta(2)-N(21)-C(21)	-79(2)
C(28)-Ta(2)-N(21)-C(21)	-98(2)
Cl(4)-Ta(2)-N(21)-C(21)	135(2)
C(29)-Ta(2)-N(21)-C(21)	-129(2)
Cl(3)-Ta(2)-N(21)-C(21)	52(2)
C(31)-Ta(2)-N(21)-C(21)	-111(2)
C(30)-Ta(2)-N(21)-C(21)	-145.9(19)
N(22)-Ta(2)-N(21)-C(27)	-157(5)
C(32)-Ta(2)-N(21)-C(27)	136(4)
C(28)-Ta(2)-N(21)-C(27)	117(4)
Cl(4)-Ta(2)-N(21)-C(27)	-10(4)
C(29)-Ta(2)-N(21)-C(27)	86(4)
Cl(3)-Ta(2)-N(21)-C(27)	-92(4)
C(31)-Ta(2)-N(21)-C(27)	104(4)
C(30)-Ta(2)-N(21)-C(27)	69(4)
C(21)-Ta(2)-N(21)-C(27)	-145(5)
C(27)-N(21)-C(21)-N(23)	-2(6)
Ta(2)-N(21)-C(21)-N(23)	-162(4)
C(27)-N(21)-C(21)-N(22)	175(3)
Ta(2)-N(21)-C(21)-N(22)	16(3)
C(27)-N(21)-C(21)-Ta(2)	159(3)
N(21)-Ta(2)-C(21)-N(23)	115(9)
N(22)-Ta(2)-C(21)-N(23)	-85(9)
C(32)-Ta(2)-C(21)-N(23)	-130(8)
C(28)-Ta(2)-C(21)-N(23)	-163(8)
Cl(4)-Ta(2)-C(21)-N(23)	67(8)
C(29)-Ta(2)-C(21)-N(23)	170(8)
Cl(3)-Ta(2)-C(21)-N(23)	-25(9)
C(31)-Ta(2)-C(21)-N(23)	-130(8)
C(30)-Ta(2)-C(21)-N(23)	170(8)
N(22)-Ta(2)-C(21)-N(21)	160(3)
C(32)-Ta(2)-C(21)-N(21)	115(2)
C(28)-Ta(2)-C(21)-N(21)	82(2)

Cl(4)-Ta(2)-C(21)-N(21)	-48(2)
C(29)-Ta(2)-C(21)-N(21)	55(2)
Cl(3)-Ta(2)-C(21)-N(21)	-140(2)
C(31)-Ta(2)-C(21)-N(21)	115(2)
C(30)-Ta(2)-C(21)-N(21)	55(3)
N(21)-Ta(2)-C(21)-N(22)	-160(3)
C(32)-Ta(2)-C(21)-N(22)	-45(2)
C(28)-Ta(2)-C(21)-N(22)	-79(2)
Cl(4)-Ta(2)-C(21)-N(22)	151.8(17)
C(29)-Ta(2)-C(21)-N(22)	-105(2)
Cl(3)-Ta(2)-C(21)-N(22)	59.7(19)
C(31)-Ta(2)-C(21)-N(22)	-46(3)
C(30)-Ta(2)-C(21)-N(22)	-106(2)
N(23)-C(21)-N(22)-C(22)	20(4)
N(21)-C(21)-N(22)-C(22)	-158(2)
Ta(2)-C(21)-N(22)-C(22)	-142(3)
N(23)-C(21)-N(22)-Ta(2)	162(3)
N(21)-C(21)-N(22)-Ta(2)	-16(3)
N(21)-Ta(2)-N(22)-C(21)	11.2(19)
C(32)-Ta(2)-N(22)-C(21)	138.0(19)
C(28)-Ta(2)-N(22)-C(21)	100(2)
Cl(4)-Ta(2)-N(22)-C(21)	-41(2)
C(29)-Ta(2)-N(22)-C(21)	88(2)
Cl(3)-Ta(2)-N(22)-C(21)	-124.5(18)
C(31)-Ta(2)-N(22)-C(21)	151.8(17)
C(30)-Ta(2)-N(22)-C(21)	119(2)
N(21)-Ta(2)-N(22)-C(22)	140(3)
C(32)-Ta(2)-N(22)-C(22)	-94(3)
C(28)-Ta(2)-N(22)-C(22)	-132(3)
Cl(4)-Ta(2)-N(22)-C(22)	88(3)
C(29)-Ta(2)-N(22)-C(22)	-144(2)
Cl(3)-Ta(2)-N(22)-C(22)	4(3)
C(31)-Ta(2)-N(22)-C(22)	-80(3)
C(30)-Ta(2)-N(22)-C(22)	-113(3)
C(21)-Ta(2)-N(22)-C(22)	128(4)
C(21)-N(22)-C(22)-C(23)	-28(3)

Ta(2)-N(22)-C(22)-C(23)	-149(2)
N(21)-C(21)-N(23)-C(24)	175(3)
N(22)-C(21)-N(23)-C(24)	-2(5)
Ta(2)-C(21)-N(23)-C(24)	72(9)
N(21)-C(21)-N(23)-C(25)	-7(6)
N(22)-C(21)-N(23)-C(25)	176(3)
Ta(2)-C(21)-N(23)-C(25)	-110(8)
N(22)-C(22)-C(23)-C(24)	21(3)
C(21)-N(23)-C(24)-C(23)	-7(4)
C(25)-N(23)-C(24)-C(23)	175(3)
C(22)-C(23)-C(24)-N(23)	-4(4)
C(21)-N(23)-C(25)-C(26)	32(4)
C(24)-N(23)-C(25)-C(26)	-150(3)
N(23)-C(25)-C(26)-C(27)	-51(4)
C(25)-C(26)-C(27)-N(21)	44(4)
C(21)-N(21)-C(27)-C(26)	-16(4)
Ta(2)-N(21)-C(27)-C(26)	126(4)
N(21)-Ta(2)-C(28)-C(29)	-94(2)
N(22)-Ta(2)-C(28)-C(29)	-158(3)
C(32)-Ta(2)-C(28)-C(29)	114(3)
Cl(4)-Ta(2)-C(28)-C(29)	-11(3)
Cl(3)-Ta(2)-C(28)-C(29)	119(2)
C(31)-Ta(2)-C(28)-C(29)	78(2)
C(30)-Ta(2)-C(28)-C(29)	37(2)
C(21)-Ta(2)-C(28)-C(29)	-124(2)
N(21)-Ta(2)-C(28)-C(33)	31(3)
N(22)-Ta(2)-C(28)-C(33)	-33(3)
C(32)-Ta(2)-C(28)-C(33)	-121(3)
Cl(4)-Ta(2)-C(28)-C(33)	114(2)
C(29)-Ta(2)-C(28)-C(33)	125(4)
Cl(3)-Ta(2)-C(28)-C(33)	-116(2)
C(31)-Ta(2)-C(28)-C(33)	-157(3)
C(30)-Ta(2)-C(28)-C(33)	162(3)
C(21)-Ta(2)-C(28)-C(33)	0(3)
N(21)-Ta(2)-C(28)-C(32)	152.5(17)
N(22)-Ta(2)-C(28)-C(32)	88.3(17)

Cl(4)-Ta(2)-C(28)-C(32)	-124.7(13)
C(29)-Ta(2)-C(28)-C(32)	-114(3)
Cl(3)-Ta(2)-C(28)-C(32)	5(2)
C(31)-Ta(2)-C(28)-C(32)	-36.1(14)
C(30)-Ta(2)-C(28)-C(32)	-77.1(18)
C(21)-Ta(2)-C(28)-C(32)	121.6(15)
C(33)-C(28)-C(29)-C(30)	-177(3)
C(32)-C(28)-C(29)-C(30)	0(4)
Ta(2)-C(28)-C(29)-C(30)	-65(3)
C(33)-C(28)-C(29)-C(34)	-4(6)
C(32)-C(28)-C(29)-C(34)	172(3)
Ta(2)-C(28)-C(29)-C(34)	108(3)
C(33)-C(28)-C(29)-Ta(2)	-112(4)
C(32)-C(28)-C(29)-Ta(2)	64(2)
N(21)-Ta(2)-C(29)-C(28)	85(2)
N(22)-Ta(2)-C(29)-C(28)	24(3)
C(32)-Ta(2)-C(29)-C(28)	-42(2)
Cl(4)-Ta(2)-C(29)-C(28)	171(2)
Cl(3)-Ta(2)-C(29)-C(28)	-96(2)
C(31)-Ta(2)-C(29)-C(28)	-82(2)
C(30)-Ta(2)-C(29)-C(28)	-120(3)
C(21)-Ta(2)-C(29)-C(28)	60(2)
N(21)-Ta(2)-C(29)-C(30)	-155(2)
N(22)-Ta(2)-C(29)-C(30)	144(2)
C(32)-Ta(2)-C(29)-C(30)	78(2)
C(28)-Ta(2)-C(29)-C(30)	120(3)
Cl(4)-Ta(2)-C(29)-C(30)	-69(2)
Cl(3)-Ta(2)-C(29)-C(30)	24(3)
C(31)-Ta(2)-C(29)-C(30)	38(2)
C(21)-Ta(2)-C(29)-C(30)	-179.6(19)
N(21)-Ta(2)-C(29)-C(34)	-40(3)
N(22)-Ta(2)-C(29)-C(34)	-101(2)
C(32)-Ta(2)-C(29)-C(34)	-168(3)
C(28)-Ta(2)-C(29)-C(34)	-126(4)
Cl(4)-Ta(2)-C(29)-C(34)	46(2)
Cl(3)-Ta(2)-C(29)-C(34)	138(2)

C(31)-Ta(2)-C(29)-C(34)	152(3)
C(30)-Ta(2)-C(29)-C(34)	114(3)
C(21)-Ta(2)-C(29)-C(34)	-66(3)
C(28)-C(29)-C(30)-C(31)	-1(4)
C(34)-C(29)-C(30)-C(31)	-174(3)
Ta(2)-C(29)-C(30)-C(31)	-63(2)
C(28)-C(29)-C(30)-C(35)	174(3)
C(34)-C(29)-C(30)-C(35)	0(6)
Ta(2)-C(29)-C(30)-C(35)	111(4)
C(28)-C(29)-C(30)-Ta(2)	63(3)
C(34)-C(29)-C(30)-Ta(2)	-111(3)
N(21)-Ta(2)-C(30)-C(31)	144.2(18)
N(22)-Ta(2)-C(30)-C(31)	59(3)
C(32)-Ta(2)-C(30)-C(31)	36.5(16)
C(28)-Ta(2)-C(30)-C(31)	82(2)
Cl(4)-Ta(2)-C(30)-C(31)	-134.8(19)
C(29)-Ta(2)-C(30)-C(31)	114(3)
Cl(3)-Ta(2)-C(30)-C(31)	-50.6(19)
C(21)-Ta(2)-C(30)-C(31)	115(2)
N(21)-Ta(2)-C(30)-C(29)	30(2)
N(22)-Ta(2)-C(30)-C(29)	-55(3)
C(32)-Ta(2)-C(30)-C(29)	-78(2)
C(28)-Ta(2)-C(30)-C(29)	-32.2(19)
Cl(4)-Ta(2)-C(30)-C(29)	111(2)
Cl(3)-Ta(2)-C(30)-C(29)	-164.8(18)
C(31)-Ta(2)-C(30)-C(29)	-114(3)
C(21)-Ta(2)-C(30)-C(29)	1(3)
N(21)-Ta(2)-C(30)-C(35)	-99(2)
N(22)-Ta(2)-C(30)-C(35)	175.6(19)
C(32)-Ta(2)-C(30)-C(35)	153(3)
C(28)-Ta(2)-C(30)-C(35)	-162(3)
Cl(4)-Ta(2)-C(30)-C(35)	-18(2)
C(29)-Ta(2)-C(30)-C(35)	-130(4)
Cl(3)-Ta(2)-C(30)-C(35)	66(3)
C(31)-Ta(2)-C(30)-C(35)	116(3)
C(21)-Ta(2)-C(30)-C(35)	-129(2)



C(29)-C(30)-C(31)-C(32)	2(3)
C(35)-C(30)-C(31)-C(32)	-174(3)
Ta(2)-C(30)-C(31)-C(32)	-60.5(19)
C(29)-C(30)-C(31)-C(36)	-177(3)
C(35)-C(30)-C(31)-C(36)	8(5)
Ta(2)-C(30)-C(31)-C(36)	121(3)
C(29)-C(30)-C(31)-Ta(2)	62(2)
C(35)-C(30)-C(31)-Ta(2)	-113(3)
N(21)-Ta(2)-C(31)-C(32)	56(2)
N(22)-Ta(2)-C(31)-C(32)	-25(2)
C(28)-Ta(2)-C(31)-C(32)	40.6(17)
Cl(4)-Ta(2)-C(31)-C(32)	163.6(14)
C(29)-Ta(2)-C(31)-C(32)	77.3(18)
Cl(3)-Ta(2)-C(31)-C(32)	-111.4(15)
C(30)-Ta(2)-C(31)-C(32)	116(3)
C(21)-Ta(2)-C(31)-C(32)	1(3)
N(21)-Ta(2)-C(31)-C(30)	-61(3)
N(22)-Ta(2)-C(31)-C(30)	-141.5(19)
C(32)-Ta(2)-C(31)-C(30)	-116(3)
C(28)-Ta(2)-C(31)-C(30)	-76(2)
Cl(4)-Ta(2)-C(31)-C(30)	47(2)
C(29)-Ta(2)-C(31)-C(30)	-38.9(19)
Cl(3)-Ta(2)-C(31)-C(30)	132.4(19)
C(21)-Ta(2)-C(31)-C(30)	-115(2)
N(21)-Ta(2)-C(31)-C(36)	179(2)
N(22)-Ta(2)-C(31)-C(36)	98(3)
C(32)-Ta(2)-C(31)-C(36)	124(3)
C(28)-Ta(2)-C(31)-C(36)	164(3)
Cl(4)-Ta(2)-C(31)-C(36)	-73(2)
C(29)-Ta(2)-C(31)-C(36)	-159(3)
Cl(3)-Ta(2)-C(31)-C(36)	12(2)
C(30)-Ta(2)-C(31)-C(36)	-120(3)
C(21)-Ta(2)-C(31)-C(36)	125(2)
C(30)-C(31)-C(32)-C(37)	-169(3)
C(36)-C(31)-C(32)-C(37)	9(5)
Ta(2)-C(31)-C(32)-C(37)	126(2)

C(30)-C(31)-C(32)-C(28)	-2(3)
C(36)-C(31)-C(32)-C(28)	177(3)
Ta(2)-C(31)-C(32)-C(28)	-66.4(19)
C(30)-C(31)-C(32)-Ta(2)	64(2)
C(36)-C(31)-C(32)-Ta(2)	-117(3)
C(29)-C(28)-C(32)-C(31)	2(4)
C(33)-C(28)-C(32)-C(31)	178(3)
Ta(2)-C(28)-C(32)-C(31)	70.4(19)
C(29)-C(28)-C(32)-C(37)	169(3)
C(33)-C(28)-C(32)-C(37)	-15(5)
Ta(2)-C(28)-C(32)-C(37)	-123(2)
C(29)-C(28)-C(32)-Ta(2)	-69(3)
C(33)-C(28)-C(32)-Ta(2)	107(3)
N(21)-Ta(2)-C(32)-C(31)	-146.6(16)
N(22)-Ta(2)-C(32)-C(31)	157.3(18)
C(28)-Ta(2)-C(32)-C(31)	-114(2)
Cl(4)-Ta(2)-C(32)-C(31)	-24(2)
C(29)-Ta(2)-C(32)-C(31)	-80.1(19)
Cl(3)-Ta(2)-C(32)-C(31)	69.2(16)
C(30)-Ta(2)-C(32)-C(31)	-37.4(17)
C(21)-Ta(2)-C(32)-C(31)	-179.4(16)
N(21)-Ta(2)-C(32)-C(37)	90(2)
N(22)-Ta(2)-C(32)-C(37)	34(2)
C(28)-Ta(2)-C(32)-C(37)	122(3)
Cl(4)-Ta(2)-C(32)-C(37)	-147.8(18)
C(29)-Ta(2)-C(32)-C(37)	156(3)
Cl(3)-Ta(2)-C(32)-C(37)	-54(2)
C(31)-Ta(2)-C(32)-C(37)	-124(3)
C(30)-Ta(2)-C(32)-C(37)	-161(3)
C(21)-Ta(2)-C(32)-C(37)	57(2)
N(21)-Ta(2)-C(32)-C(28)	-32(2)
N(22)-Ta(2)-C(32)-C(28)	-88.3(18)
Cl(4)-Ta(2)-C(32)-C(28)	90.3(17)
C(29)-Ta(2)-C(32)-C(28)	34.3(18)
Cl(3)-Ta(2)-C(32)-C(28)	-176.3(16)
C(31)-Ta(2)-C(32)-C(28)	114(2)

C(30)-Ta(2)-C(32)-C(28)	77.0(19)
C(21)-Ta(2)-C(32)-C(28)	-64.9(18)

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Observed and calculated structure factors for (C<sub>5</sub>Me<sub>5</sub>)Ta(hpp)Cl<sub>2</sub>.

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
-4	-2	-17	378	378	17	-4	-2	-15	362	383	16	2	-4	-14	365	345	35	-6	-8	-13	0	52	1	1	-1	-13	195	152	28
-3	-2	-17	320	315	19	-3	-2	-15	318	340	17	-8	-3	-14	321	306	33	-5	-8	-13	223	241	26	2	-1	-13	282	259	21
-4	-1	-17	137	164	50	-2	-2	-15	79	52	50	-7	-3	-14	459	443	28	-4	-8	-13	156	192	31	3	-1	-13	104	185	74
-3	-1	-17	76	129	76	-1	-2	-15	306	293	20	-6	-3	-14	211	201	16	-3	-8	-13	0	36	1	4	-1	-13	46	69	45
-2	-1	-17	310	303	29	0	-2	-15	406	394	17	-5	-3	-14	0	38	1	-2	-8	-13	0	186	1	-8	0	-13	50	35	49
-3	0	-17	322	315	27	1	-2	-15	208	215	22	-4	-3	-14	360	373	16	-8	-7	-13	124	149	37	-7	0	-13	335	338	25
-5	-5	-16	217	225	26	-7	-1	-15	0	33	1	-3	-3	-14	334	318	15	-7	-7	-13	129	115	33	-6	0	-13	370	357	25
-4	-5	-16	373	360	21	-6	-1	-15	321	316	27	-2	-3	-14	145	88	32	-6	-7	-13	230	231	21	-5	0	-13	68	93	67
-3	-5	-16	190	169	26	-5	-1	-15	432	411	28	-1	-3	-14	386	391	16	-5	-7	-13	61	124	61	-4	0	-13	226	214	10
-6	-4	-16	286	272	18	-4	-1	-15	362	384	14	0	-3	-14	518	521	27	-4	-7	-13	162	222	28	-3	0	-13	621	603	13
-5	-4	-16	323	325	20	-3	-1	-15	82	74	40	1	-3	-14	172	214	38	-3	-7	-13	333	338	30	-2	0	-13	271	281	12
-4	-4	-16	0	37	1	-2	-1	-15	247	280	19	2	-3	-14	173	126	41	-2	-7	-13	362	345	29	-1	0	-13	146	150	16
-3	-4	-16	254	210	22	-1	-1	-15	280	284	18	-8	-2	-14	416	381	26	-1	-7	-13	0	49	1	0	0	-13	480	464	14
-2	-4	-16	250	265	23	0	-1	-15	74	56	73	-7	-2	-14	74	45	73	0	-7	-13	131	222	69	1	0	-13	474	454	14
-1	-4	-16	116	95	44	1	-1	-15	313	311	19	-6	-2	-14	353	357	24	-9	-6	-13	98	147	39	2	0	-13	35	112	35
-6	-3	-16	65	89	64	2	-1	-15	355	375	26	-5	-2	-14	367	390	15	-8	-6	-13	304	298	19	3	0	-13	296	275	25
-5	-3	-16	146	188	24	-7	0	-15	313	338	34	-4	-2	-14	245	268	16	-7	-6	-13	346	352	18	-8	-1	-13	401	406	29
-4	-3	-16	417	445	19	-6	0	-15	463	391	28	-3	-2	-14	214	239	18	-6	-6	-13	0	99	1	-7	1	-13	316	303	31
-3	-3	-16	275	293	19	-5	0	-15	110	92	110	-2	-2	-14	493	502	15	-5	-6	-13	192	221	25	-6	1	-13	0	87	1
-2	-3	-16	0	74	1	-4	0	-15	237	232	31	-1	-2	-14	381	382	23	-4	-6	-13	345	359	18	-5	1	-13	348	332	23
-1	-3	-16	258	265	20	-3	0	-15	368	382	15	0	-2	-14	0	34	1	-3	-6	-13	179	206	23	-4	1	-13	256	277	21
-6	-2	-16	362	354	32	-2	0	-15	89	70	35	1	-2	-14	286	280	18	-2	-6	-13	84	113	84	-3	1	-13	65	58	44
-5	-2	-16	440	429	30	-1	0	-15	225	224	19	-2	-2	-14	387	391	18	-1	-6	-13	422	398	28	-2	1	-13	555	543	14
-4	-2	-16	229	220	18	0	0	-15	383	380	17	3	-2	-14	191	171	38	0	-6	-13	271	287	39	-1	1	-13	617	598	15
-3	-2	-16	149	182	24	1	0	-15	315	302	31	-8	-1	-14	219	186	32	1	-6	-13	134	139	60	0	1	-13	460	447	13
-2	-2	-16	340	334	18	-6	-1	-15	266	214	34	-7	-1	-14	470	479	25	-9	-5	-13	380	388	17	1	1	-13	58	106	57
-1	-2	-16	244	250	22	-5	-1	-15	430	408	29	-6	-1	-14	442	414	26	-8	-5	-13	238	264	19	2	1	-13	388	375	23
0	-2	-16	102	55	52	-4	-1	-15	370	343	26	-5	-1	-14	0	138	1	-7	-5	-13	58	23	57	3	1	-13	384	379	27
-6	-1	-16	199	240	38	-3	-1	-15	58	14	57	-4	-1	-14	267	258	12	-6	-5	-13	370	351	15	4	1	-13	76	15	76
-5	-1	-16	0	29	1	-2	-1	-15	356	327	15	-3	-1	-14	381	374	15	-5	-5	-13	367	357	17	-6	2	-13	381	375	28
-4	-1	-16	330	323	28	-1	-1	-15	423	410	15	-2	-1	-14	65	62	64	-4	-5	-13	58	39	57	-5	2	-13	199	197	27
-3	-1	-16	313	328	19	0	-1	-15	192	195	18	-1	-1	-14	185	184	19	-3	-5	-13	275	264	18	-4	2	-13	114	186	46
-2	-1	-16	71	74	71	1	-1	-15	192	190	42	0	-1	-14	447	431	15	-2	-5	-13	475	436	23	-3	2	-13	538	536	12
-1	-1	-16	156	156	33	2	-1	-15	394	439	30	1	-1	-14	232	238	23	-1	-5	-13	305	240	29	-2	2	-13	586	562	14
0	-1	-16	260	251	20	-6	-2	-15	485	494	27	2	-1	-14	20	44	20	0	-5	-13	0	158	1	-1	2	-13	94	46	26
-6	0	-16	223	255	33	-5	-2	-15	193	178	33	3	-1	-14	305	332	30	1	-5	-13	124	237	63	0	2	-13	407	377	13
-5	0	-16	359	341	28	-4	-2	-15	0	126	1	-8	0	-14	441	410	26	2	-5	-13	211	247	40	1	2	-13	608	581	14
-4	0	-16	306	281	26	-3	-2	-15	444	440	25	-7	0	-14	234	200	28	-9	-4	-13	0	56	1	2	2	-13	321	337	15
-3	0	-16	100	68	32	-2	-2	-15	280	305	16	-6	0	-14	169	167	45	-8	-4	-13	245	245	14	3	2	-13	0	44	1
-2	0	-16	323	304	17	-1	-2	-15	50	47	49	-5	0	-14	359	348	24	-7	-4	-13	460	447	16	4	2	-13	274	259	22
-1	0	-16	203	238	23	0	-2	-15	318	317	24	-4	0	-14	305	315	11	-6	-4	-13	254	251	18	-5	3	-13	285	297	26
0	0	-16	41	40	40	1	-2	-15	485	405	23	-3	0	-14	20	74	20	-5	-4	-13	0	48	1	-4	3	-13	425	410	23
-5	1	-16	46	92	45	2	-2	-15	152	154	60	-2	0	-14	378	377	15	-4	-4	-13	348	349	14	-3	3	-13	221	191	23
-4	1	-16	274	255	30	-5	-3	-15	393	361	28	-1	0	-14	353	360	15	-3	-4	-13	279	279	18	-2	3	-13	224	233	14
-3	1	-16	403	400	26	-4	-3	-15	459	408	28	0	0	-14	199	173	16	-2	-4	-13	0	35	1	-1	3	-13	533	486	14
-2	1	-16	239	258	20	-3	-3	-15	177	182	32	1	0	-14	225	217	19	-1	-4	-13	306	341	27	0	3	-13	406	384	11
-1	1	-16	102	117	41	-2	-3	-15	215	203	26	2	0	-14	408	380	27	0	-4	-13	403	425	25	1	3	-13	129	122	44
0	1	-16	310	304	32	-1	-3	-15	356	359	22	3	0	-14	238	250	34	1	-4	-13	123	131	55	2	3	-13	219	225	26
-4	2	-16	455	426	30	0	3	-15	191	174	25	-7	-1	-14	355	344	31	2	-4	-13	121	147	59	3	3	-13	338	344	24
-3	2	-16	0	85	1	1	3	-15	0	97	1	-6	-1	-14	403	415	29	3	-4	-13	292	339	30	4	3	-13	50	83	49
-2	2	-16	190	170	44	-1	-4	-15	0	46	1	-5	-1	-14	171	133	43	-9	-3	-13	330	366	29	-2	4	-13	389	391	15
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4	2	11	647	672	14	4	-6	12	423	424	16	5	1	12	0	44	1	1	-5	13	470	442	24	-3	4	13	346	339	34
5	2	11	247	237	11	-2	-5	12	447	433	22	6	1	12	429	435	15	2	-5	13	102	99	81	-2	4	13	87	147	86
6	2	11	333	342	24	-1	-5	12	461	444	21	7	1	12	442	441	15	3	-5	13	239	219	34	-1	4	13	94	131	93
7	2	11	315	374	26	0	-5	12	119	106	61	8	1	12	0	44	1	4	-5	13	284	305	22	0	4	13	453	425	24
8	2	11	136	122	48	1	-5	12	389	340	21	9	1	12	183	237	27	5	-5	13	0	101	1	1	4	13	318	341	24
-5	3	11	314	329	32	2	-5	12	593	553	25	-4	2	12	0	58	1	-1	-4	13	399	425	24	2	4	13	35	35	35
-4	3	11	121	180	59	3	-5	12	319	337	19	-3	2	12	242	248	35	0	-4	13	256	222	23	3	4	13	252	279	27
-3	3	11	250	229	33	4	-5	12	58	38	57	-2	2	12	321	315	24	1	-4	13	87	114	86	4	4	13	337	349	24
-2	3	11	366	404	22	5	-5	12	288	300	18	-1	2	12	177	175	28	2	-4	13	384	391	24	5	4	13	0	48	1
-1	3	11	372	413	29	6	-5	12	259	282	20	0	2	12	265	286	22	3	-4	13	324	295	24	6	4	13	272	251	26
0	3	11	89	89	53	-5	-4	12	55	97	54	1	2	12	731	674	20	4	-4	13	0	43	1	7	4	13	474	447	26
1	3	11	643	657	21	-4	-4	12	169	205	51	2	2	12	725	711	25	5	-4	13	292	316	18	8	4	13	256	245	37
2	3	11	626	603	12	-1	-4	12	119	121	42	3	2	12	256	257	18	6	-4	13	257	279	20	-2	5	13	216	247	38
3	3	11	281	303	11	0	-4	12	404	415	19	5	2	12	505	528	20	0	-3	13	412	384	21	-1	5	13	203	237	40
4	3	11	280	305	13	1	-4	12	472	480	23	6	2	12	351	306	22	1	-3	13	546	486	23	0	5	13	123	158	101
5	3	11	613	608	15	2	-4	12	0	68	1	7	2	12	0	104	1	2	-3	13	226	232	25	1	5	13	234	240	28
6	3	11	390	358	20	3	-4	12	305	330	16	8	2	12	383	364	24	3	-3	13	172	190	32	2	5	13	454	436	27
7	3	11	0	15	1	4	-4	12	378	398	15	-4	3	12	319	324	31	4	-3	13	400	410	17	3	5	13	230	265	23
8	3	11	288	311	32	5	-4	12	150	157	22	-3	3	12	338	340	29	5	-3	13	282	297	15	4	5	13	0	39	1
-4	4	11	317	343	31	6	-4	12	110	164	39	-2	3	12	112	50	61	6	-3	13	58	82	57	5	5	13	391	357	20
-3	4	11	366	393	28	7	-4	12	324	353	19	-1	3	12	238	250	25	7	-3	13	370	353	17	6	5	13	395	351	22
-2	4	11	98	115	68	-5	-3	12	195	210	36	0	3	12	578	574	25	-4	-2	13	228	259	31	7	5	13	0	23	1
-1	4	11	46	73	45	-4	-3	12	364	337	25	1	3	12	351	361	19	-3	-2	13	0	44	1	8	5	13	226	264	30
0	4	11	481	489	24	-3	-3	12	0	80	1	2	3	12	175	206	25	-2	-2	13	318	337	24	-1	6				



h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
4	-4	14	234	263	24	-1	1	14	221	238	35	5	4	14	382	333	24	5	-3	15	346	361	23	7	5	15	234	193	32
5	-4	14	208	218	28	5	1	14	195	138	32	6	4	14	330	332	29	4	-2	15	0	126	1	5	-1	16	116	92	115
1	-3	14	65	35	64	6	1	14	396	414	26	7	4	14	0	53	1	5	-2	15	165	178	32	5	0	16	315	341	33
2	-3	14	277	312	25	7	1	14	481	479	19	8	4	14	250	277	40	6	-2	15	489	494	22	6	0	16	223	255	36
3	-3	14	310	336	27	8	1	14	161	186	29	-1	5	14	61	115	61	5	-1	15	331	408	34	5	1	16	118	29	66
4	-3	14	0	57	1	-3	2	14	169	171	51	0	5	14	228	261	35	6	-1	15	206	214	42	6	1	16	277	240	36
5	-3	14	266	260	24	-2	2	14	396	391	31	1	5	14	340	361	34	5	0	15	84	92	84	5	2	16	375	429	28
6	-3	14	349	373	23	-1	2	14	260	280	30	4	5	14	294	314	24	6	0	15	406	391	32	6	2	16	349	354	31
2	-2	14	353	370	23	0	2	14	0	34	1	5	5	14	102	121	102	7	0	15	331	338	34	5	3	16	184	188	56
3	-2	14	74	116	73	5	2	14	401	390	25	6	5	14	210	200	30	5	1	15	378	411	33	6	3	16	162	89	64
4	-2	14	351	401	24	6	2	14	348	357	28	7	5	14	507	454	27	6	1	15	302	316	35	5	4	16	345	325	34
5	-2	14	399	422	21	7	2	14	41	45	40	8	5	14	336	331	31	7	1	15	0	33	1	6	4	16	257	272	44
6	-2	14	106	68	52	8	2	14	362	381	19	0	6	14	173	178	47	5	2	15	0	35	1	4	5	16	329	360	27
7	-2	14	155	201	40	-2	3	14	71	126	71	1	6	14	0	76	1	6	2	15	274	282	30	5	5	16	210	225	36
4	-1	14	217	211	29	-1	3	14	204	214	38	2	6	14	327	327	36	7	2	15	505	485	30	7	2	15	505	485	30
5	-1	14	71	133	71	0	3	14	532	521	27	5	6	14	293	230	29	8	2	15	388	332	31	8	2	15	388	332	31
6	-1	14	403	415	20	1	3	14	380	391	24	6	6	14	325	317	27	5	3	15	393	402	26	5	3	15	393	402	26

## APPENDIX G

CRYSTALLOGRAPHIC DATA FOR  $(C_5Me_4Et)Ta(hpp)Cl_2$

Identification code	mes1010	
Empirical formula	C <sub>37</sub> H <sub>41</sub> Cl <sub>14</sub> N <sub>6</sub> Ta <sub>2</sub>	
Formula weight	1073.46	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P1	
Unit cell dimensions	a = 9.0682(14) Å	α = 85.403(5)°.
	b = 9.3280(14) Å	β = 73.210(5)°.
	c = 12.5226(19) Å	γ = 74.062(5)°.
Volume	975.1(3) Å <sup>3</sup>	
Z	1	
Density (calculated)	1.828 Mg/m <sup>3</sup>	
Absorption coefficient	5.915 mm <sup>-1</sup>	
F(000)	519	
Crystal size	0.16 x 0.13 x 0.05 mm <sup>3</sup>	
Theta range for data collection	2.96 to 27.98°.	
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 12, -16 ≤ l ≤ 16	
Reflections collected	8346	
Independent reflections	4630 [R(int) = 0.0352]	
Completeness to theta = 27.98°	98.6 %	
Max. and min. transmission	0.7564 and 0.4512	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	4630 / 0 / 218	
Goodness-of-fit on F <sup>2</sup>	1.108	
Final R indices [I > 2σ(I)]	R1 = 0.0474, wR2 = 0.1438	
R indices (all data)	R1 = 0.0611, wR2 = 0.1518	
Extinction coefficient	0.0000(10)	
Largest diff. peak and hole	3.224 and -2.067 e.Å <sup>-3</sup>	

Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1010.  $U(\text{eq})$  is defined as one third of the trace of the orthogonalized  $U^{ij}$  tensor.

	x	y	z	U(eq)
Ta(1)	3546(1)	7857(1)	7702(1)	26(1)
Cl(1)	4163(3)	10226(3)	7232(2)	34(1)
Cl(2)	4514(3)	7852(3)	9304(2)	34(1)
N(2)	1507(9)	7450(9)	8837(6)	29(2)
N(1)	1320(9)	8869(9)	7364(6)	30(2)
C(8)	5799(11)	6902(10)	6053(8)	27(2)
C(7)	486(11)	10122(11)	6791(9)	32(2)
C(2)	929(12)	6863(12)	9922(9)	36(2)
C(1)	485(11)	8426(10)	8328(8)	29(2)
N(3)	-1084(9)	8838(10)	8768(7)	31(2)
C(12)	6046(11)	6016(11)	6987(8)	32(2)
C(11)	4774(11)	5316(10)	7394(7)	27(2)
C(4)	-1821(12)	8276(13)	9859(9)	37(2)
C(3)	-655(12)	7897(13)	10562(9)	38(2)
C(5)	-2080(12)	9912(13)	8161(9)	38(2)
C(6)	-1179(12)	9970(13)	6954(9)	39(2)
C(10)	3742(11)	5801(11)	6701(8)	29(2)
C(9)	4373(11)	6778(10)	5883(7)	27(2)
C(15)	2311(12)	5246(12)	6756(9)	37(2)
C(13)	6876(12)	7802(12)	5362(9)	36(2)
C(17)	7498(11)	5698(12)	7405(9)	35(2)
C(14)	3665(12)	7477(13)	4957(8)	37(2)
C(16)	4633(14)	4182(13)	8312(10)	42(2)
C(18)	8720(13)	4262(13)	6881(13)	51(3)

Bond lengths [Å] and angles [°] for mes1010.

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Ta(1)-N(2)	2.089(7)
Ta(1)-N(1)	2.128(8)
Ta(1)-C(10)	2.314(9)
Ta(1)-C(11)	2.336(9)
Ta(1)-C(9)	2.394(9)
Ta(1)-Cl(2)	2.410(2)
Ta(1)-Cl(1)	2.413(2)
Ta(1)-C(12)	2.414(9)
Ta(1)-C(8)	2.468(9)
Ta(1)-C(1)	2.569(9)
N(2)-C(1)	1.371(12)
N(2)-C(2)	1.431(12)
N(1)-C(1)	1.328(12)
N(1)-C(7)	1.466(12)
C(8)-C(9)	1.406(13)
C(8)-C(12)	1.413(14)
C(8)-C(13)	1.500(13)
C(7)-C(6)	1.509(14)
C(7)-H(7A)	0.9900
C(7)-H(7B)	0.9900
C(2)-C(3)	1.528(15)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(1)-N(3)	1.325(12)
N(3)-C(4)	1.462(13)
N(3)-C(5)	1.475(13)
C(12)-C(11)	1.427(14)
C(12)-C(17)	1.501(13)
C(11)-C(10)	1.417(12)
C(11)-C(16)	1.501(14)
C(4)-C(3)	1.517(14)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(3)-H(3A)	0.9900

C(3)-H(3B)	0.9900
C(5)-C(6)	1.500(15)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(10)-C(9)	1.417(13)
C(10)-C(15)	1.506(13)
C(9)-C(14)	1.504(12)
C(15)-H(13A)	0.9800
C(15)-H(13B)	0.9800
C(15)-H(13C)	0.9800
C(13)-H(16A)	0.9800
C(13)-H(16B)	0.9800
C(13)-H(16C)	0.9800
C(17)-C(18)	1.535(15)
C(17)-H(15A)	0.9900
C(17)-H(15B)	0.9900
C(14)-H(17A)	0.9800
C(14)-H(17B)	0.9800
C(14)-H(17C)	0.9800
C(16)-H(14A)	0.9800
C(16)-H(14B)	0.9800
C(16)-H(14C)	0.9800
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
N(2)-Ta(1)-N(1)	62.9(3)
N(2)-Ta(1)-C(10)	88.4(3)
N(1)-Ta(1)-C(10)	88.3(3)
N(2)-Ta(1)-C(11)	92.5(3)
N(1)-Ta(1)-C(11)	121.3(3)
C(10)-Ta(1)-C(11)	35.5(3)
N(2)-Ta(1)-C(9)	117.8(3)
N(1)-Ta(1)-C(9)	86.0(3)

C(10)-Ta(1)-C(9)	35.0(3)
C(11)-Ta(1)-C(9)	58.1(3)
N(2)-Ta(1)-Cl(2)	85.8(2)
N(1)-Ta(1)-Cl(2)	133.4(2)
C(10)-Ta(1)-Cl(2)	126.7(2)
C(11)-Ta(1)-Cl(2)	91.9(2)
C(9)-Ta(1)-Cl(2)	140.5(2)
N(2)-Ta(1)-Cl(1)	127.6(2)
N(1)-Ta(1)-Cl(1)	86.9(2)
C(10)-Ta(1)-Cl(1)	135.1(2)
C(11)-Ta(1)-Cl(1)	139.5(2)
C(9)-Ta(1)-Cl(1)	100.1(2)
Cl(2)-Ta(1)-Cl(1)	86.42(9)
N(2)-Ta(1)-C(12)	125.6(3)
N(1)-Ta(1)-C(12)	141.9(3)
C(10)-Ta(1)-C(12)	57.7(3)
C(11)-Ta(1)-C(12)	34.9(3)
C(9)-Ta(1)-C(12)	56.5(3)
Cl(2)-Ta(1)-C(12)	84.1(2)
Cl(1)-Ta(1)-C(12)	104.9(3)
N(2)-Ta(1)-C(8)	145.2(3)
N(1)-Ta(1)-C(8)	115.2(3)
C(10)-Ta(1)-C(8)	57.1(3)
C(11)-Ta(1)-C(8)	57.4(3)
C(9)-Ta(1)-C(8)	33.6(3)
Cl(2)-Ta(1)-C(8)	110.1(2)
Cl(1)-Ta(1)-C(8)	85.3(2)
C(12)-Ta(1)-C(8)	33.6(3)
N(2)-Ta(1)-C(1)	32.2(3)
N(1)-Ta(1)-C(1)	31.1(3)
C(10)-Ta(1)-C(1)	91.3(3)
C(11)-Ta(1)-C(1)	112.4(3)
C(9)-Ta(1)-C(1)	105.9(3)
Cl(2)-Ta(1)-C(1)	109.4(2)
Cl(1)-Ta(1)-C(1)	106.2(2)
C(12)-Ta(1)-C(1)	146.7(3)

C(8)-Ta(1)-C(1)	139.4(3)
C(1)-N(2)-C(2)	121.0(8)
C(1)-N(2)-Ta(1)	93.6(6)
C(2)-N(2)-Ta(1)	143.2(7)
C(1)-N(1)-C(7)	117.7(8)
C(1)-N(1)-Ta(1)	93.2(6)
C(7)-N(1)-Ta(1)	143.5(6)
C(9)-C(8)-C(12)	107.7(8)
C(9)-C(8)-C(13)	126.3(9)
C(12)-C(8)-C(13)	126.0(8)
C(9)-C(8)-Ta(1)	70.3(5)
C(12)-C(8)-Ta(1)	71.1(5)
C(13)-C(8)-Ta(1)	124.5(7)
N(1)-C(7)-C(6)	107.6(8)
N(1)-C(7)-H(7A)	110.2
C(6)-C(7)-H(7A)	110.2
N(1)-C(7)-H(7B)	110.2
C(6)-C(7)-H(7B)	110.2
H(7A)-C(7)-H(7B)	108.5
N(2)-C(2)-C(3)	110.8(8)
N(2)-C(2)-H(2A)	109.5
C(3)-C(2)-H(2A)	109.5
N(2)-C(2)-H(2B)	109.5
C(3)-C(2)-H(2B)	109.5
H(2A)-C(2)-H(2B)	108.1
N(3)-C(1)-N(1)	127.3(8)
N(3)-C(1)-N(2)	123.4(9)
N(1)-C(1)-N(2)	109.3(8)
N(3)-C(1)-Ta(1)	171.6(7)
N(1)-C(1)-Ta(1)	55.8(5)
N(2)-C(1)-Ta(1)	54.2(4)
C(1)-N(3)-C(4)	120.6(8)
C(1)-N(3)-C(5)	119.2(8)
C(4)-N(3)-C(5)	120.1(8)
C(8)-C(12)-C(11)	108.7(8)
C(8)-C(12)-C(17)	126.1(9)



C(11)-C(12)-C(17)	124.7(9)
C(8)-C(12)-Ta(1)	75.3(5)
C(11)-C(12)-Ta(1)	69.6(5)
C(17)-C(12)-Ta(1)	126.8(7)
C(10)-C(11)-C(12)	106.7(8)
C(10)-C(11)-C(16)	126.5(9)
C(12)-C(11)-C(16)	126.6(9)
C(10)-C(11)-Ta(1)	71.4(5)
C(12)-C(11)-Ta(1)	75.5(5)
C(16)-C(11)-Ta(1)	122.4(7)
N(3)-C(4)-C(3)	110.2(8)
N(3)-C(4)-H(4A)	109.6
C(3)-C(4)-H(4A)	109.6
N(3)-C(4)-H(4B)	109.6
C(3)-C(4)-H(4B)	109.6
H(4A)-C(4)-H(4B)	108.1
C(4)-C(3)-C(2)	110.4(9)
C(4)-C(3)-H(3A)	109.6
C(2)-C(3)-H(3A)	109.6
C(4)-C(3)-H(3B)	109.6
C(2)-C(3)-H(3B)	109.6
H(3A)-C(3)-H(3B)	108.1
N(3)-C(5)-C(6)	110.3(8)
N(3)-C(5)-H(5A)	109.6
C(6)-C(5)-H(5A)	109.6
N(3)-C(5)-H(5B)	109.6
C(6)-C(5)-H(5B)	109.6
H(5A)-C(5)-H(5B)	108.1
C(5)-C(6)-C(7)	112.9(9)
C(5)-C(6)-H(6A)	109.0
C(7)-C(6)-H(6A)	109.0
C(5)-C(6)-H(6B)	109.0
C(7)-C(6)-H(6B)	109.0
H(6A)-C(6)-H(6B)	107.8
C(9)-C(10)-C(11)	108.5(8)
C(9)-C(10)-C(15)	125.8(9)

C(11)-C(10)-C(15)	125.5(9)
C(9)-C(10)-Ta(1)	75.6(5)
C(11)-C(10)-Ta(1)	73.1(5)
C(15)-C(10)-Ta(1)	122.0(7)
C(8)-C(9)-C(10)	108.4(8)
C(8)-C(9)-C(14)	126.7(9)
C(10)-C(9)-C(14)	124.9(9)
C(8)-C(9)-Ta(1)	76.1(5)
C(10)-C(9)-Ta(1)	69.4(5)
C(14)-C(9)-Ta(1)	123.0(7)
C(10)-C(15)-H(13A)	109.5
C(10)-C(15)-H(13B)	109.5
H(13A)-C(15)-H(13B)	109.5
C(10)-C(15)-H(13C)	109.5
H(13A)-C(15)-H(13C)	109.5
H(13B)-C(15)-H(13C)	109.5
C(8)-C(13)-H(16A)	109.5
C(8)-C(13)-H(16B)	109.5
H(16A)-C(13)-H(16B)	109.5
C(8)-C(13)-H(16C)	109.5
H(16A)-C(13)-H(16C)	109.5
H(16B)-C(13)-H(16C)	109.5
C(12)-C(17)-C(18)	110.7(9)
C(12)-C(17)-H(15A)	109.5
C(18)-C(17)-H(15A)	109.5
C(12)-C(17)-H(15B)	109.5
C(18)-C(17)-H(15B)	109.5
H(15A)-C(17)-H(15B)	108.1
C(9)-C(14)-H(17A)	109.5
C(9)-C(14)-H(17B)	109.5
H(17A)-C(14)-H(17B)	109.5
C(9)-C(14)-H(17C)	109.5
H(17A)-C(14)-H(17C)	109.5
H(17B)-C(14)-H(17C)	109.5
C(11)-C(16)-H(14A)	109.5
C(11)-C(16)-H(14B)	109.5

H(14A)-C(16)-H(14B)	109.5
C(11)-C(16)-H(14C)	109.5
H(14A)-C(16)-H(14C)	109.5
H(14B)-C(16)-H(14C)	109.5
C(17)-C(18)-H(18A)	109.5
C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5

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Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for mes1010. The anisotropic displacement factor exponent takes the form:  $-2\pi^2 [ h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12} ]$

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{23}$	$U^{13}$	$U^{12}$
Ta(1)	24(1)	28(1)	25(1)	4(1)	-9(1)	-7(1)
Cl(1)	39(1)	31(1)	34(1)	5(1)	-10(1)	-13(1)
Cl(2)	35(1)	42(1)	31(1)	3(1)	-16(1)	-10(1)
N(2)	27(4)	36(4)	25(4)	9(3)	-6(3)	-11(3)
N(1)	24(4)	39(5)	24(4)	0(3)	-6(3)	-5(3)
C(8)	25(4)	26(4)	31(5)	-1(4)	-9(4)	-5(3)
C(7)	32(5)	26(5)	37(5)	7(4)	-17(4)	1(4)
C(2)	33(5)	38(6)	39(5)	10(4)	-9(4)	-18(4)
C(1)	26(4)	27(5)	35(5)	2(4)	-15(4)	-4(4)
N(3)	26(4)	38(5)	27(4)	1(3)	-9(3)	-6(3)
C(12)	24(4)	37(5)	35(5)	-6(4)	-10(4)	-3(4)
C(11)	29(4)	26(4)	23(4)	1(3)	-10(3)	0(4)
C(4)	27(5)	43(6)	36(5)	-4(4)	-1(4)	-8(4)
C(3)	36(5)	49(6)	31(5)	5(4)	-11(4)	-15(5)
C(5)	27(5)	43(6)	45(6)	8(5)	-13(4)	-10(4)
C(6)	36(5)	50(6)	35(5)	12(5)	-21(4)	-11(5)
C(10)	31(5)	29(5)	29(5)	0(4)	-12(4)	-8(4)
C(9)	30(4)	26(4)	24(4)	4(3)	-12(4)	-3(4)
C(15)	36(5)	36(5)	43(6)	2(4)	-15(4)	-14(4)
C(13)	33(5)	40(6)	36(5)	6(4)	-5(4)	-19(4)
C(17)	28(5)	36(5)	42(6)	-3(4)	-17(4)	0(4)
C(14)	35(5)	50(6)	29(5)	3(4)	-16(4)	-6(5)
C(16)	40(6)	40(6)	43(6)	2(5)	-10(5)	-11(5)
C(18)	34(6)	34(6)	88(10)	-5(6)	-26(6)	-2(5)

Hydrogen coordinates ( $\times 10^4$ ) and isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )  
for mes1010.

	x	y	z	U(eq)
H(7A)	1056	10102	5986	39
H(7B)	440	11079	7106	39
H(2A)	1727	6746	10342	43
H(2B)	780	5867	9849	43
H(4A)	-2144	7375	9760	44
H(4B)	-2791	9041	10244	44
H(3A)	-1115	7404	11261	46
H(3B)	-469	8824	10763	46
H(5A)	-2390	10914	8496	45
H(5B)	-3067	9611	8223	45
H(6A)	-1777	10825	6594	47
H(6B)	-1111	9049	6580	47
H(13A)	2643	4369	6275	55
H(13B)	1520	6033	6501	55
H(13C)	1839	4975	7527	55
H(16A)	7680	7183	4756	54
H(16B)	7409	8144	5832	54
H(16C)	6243	8666	5047	54
H(15A)	7181	5592	8227	42
H(15B)	7990	6544	7217	42
H(17A)	4172	6852	4289	56
H(17B)	3847	8470	4791	56
H(17C)	2515	7566	5187	56
H(14A)	5233	3188	8009	62
H(14B)	3506	4202	8639	62
H(14C)	5066	4416	8889	62
H(18A)	9657	4075	7163	76
H(18B)	9045	4373	6068	76
H(18C)	8237	3423	7077	76

Torsion angles [°] for mes1010.

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N(1)-Ta(1)-N(2)-C(1)	-6.2(5)
C(10)-Ta(1)-N(2)-C(1)	-95.0(6)
C(11)-Ta(1)-N(2)-C(1)	-130.3(6)
C(9)-Ta(1)-N(2)-C(1)	-75.3(6)
Cl(2)-Ta(1)-N(2)-C(1)	138.0(6)
Cl(1)-Ta(1)-N(2)-C(1)	55.7(6)
C(12)-Ta(1)-N(2)-C(1)	-142.3(6)
C(8)-Ta(1)-N(2)-C(1)	-102.1(7)
N(1)-Ta(1)-N(2)-C(2)	-167.5(13)
C(10)-Ta(1)-N(2)-C(2)	103.6(12)
C(11)-Ta(1)-N(2)-C(2)	68.4(12)
C(9)-Ta(1)-N(2)-C(2)	123.4(11)
Cl(2)-Ta(1)-N(2)-C(2)	-23.4(11)
Cl(1)-Ta(1)-N(2)-C(2)	-105.6(11)
C(12)-Ta(1)-N(2)-C(2)	56.3(13)
C(8)-Ta(1)-N(2)-C(2)	96.5(12)
C(1)-Ta(1)-N(2)-C(2)	-161.4(15)
N(2)-Ta(1)-N(1)-C(1)	6.3(6)
C(10)-Ta(1)-N(1)-C(1)	95.4(6)
C(11)-Ta(1)-N(1)-C(1)	81.7(6)
C(9)-Ta(1)-N(1)-C(1)	130.4(6)
Cl(2)-Ta(1)-N(1)-C(1)	-47.2(7)
Cl(1)-Ta(1)-N(1)-C(1)	-129.3(6)
C(12)-Ta(1)-N(1)-C(1)	120.4(7)
C(8)-Ta(1)-N(1)-C(1)	147.4(6)
N(2)-Ta(1)-N(1)-C(7)	155.2(12)
C(10)-Ta(1)-N(1)-C(7)	-115.7(11)
C(11)-Ta(1)-N(1)-C(7)	-129.4(11)
C(9)-Ta(1)-N(1)-C(7)	-80.8(11)
Cl(2)-Ta(1)-N(1)-C(7)	101.7(10)
Cl(1)-Ta(1)-N(1)-C(7)	19.6(11)
C(12)-Ta(1)-N(1)-C(7)	-90.7(12)
C(8)-Ta(1)-N(1)-C(7)	-63.7(11)
C(1)-Ta(1)-N(1)-C(7)	148.9(14)

N(2)-Ta(1)-C(8)-C(9)	46.3(8)
N(1)-Ta(1)-C(8)-C(9)	-32.0(6)
C(10)-Ta(1)-C(8)-C(9)	37.8(5)
C(11)-Ta(1)-C(8)-C(9)	80.3(6)
Cl(2)-Ta(1)-C(8)-C(9)	159.3(5)
Cl(1)-Ta(1)-C(8)-C(9)	-116.3(5)
C(12)-Ta(1)-C(8)-C(9)	117.6(8)
C(1)-Ta(1)-C(8)-C(9)	-6.7(8)
N(2)-Ta(1)-C(8)-C(12)	-71.3(8)
N(1)-Ta(1)-C(8)-C(12)	-149.6(5)
C(10)-Ta(1)-C(8)-C(12)	-79.8(6)
C(11)-Ta(1)-C(8)-C(12)	-37.3(5)
C(9)-Ta(1)-C(8)-C(12)	-117.6(8)
Cl(2)-Ta(1)-C(8)-C(12)	41.6(6)
Cl(1)-Ta(1)-C(8)-C(12)	126.1(5)
C(1)-Ta(1)-C(8)-C(12)	-124.4(6)
N(2)-Ta(1)-C(8)-C(13)	167.4(7)
N(1)-Ta(1)-C(8)-C(13)	89.1(8)
C(10)-Ta(1)-C(8)-C(13)	158.9(9)
C(11)-Ta(1)-C(8)-C(13)	-158.6(9)
C(9)-Ta(1)-C(8)-C(13)	121.1(10)
Cl(2)-Ta(1)-C(8)-C(13)	-79.6(8)
Cl(1)-Ta(1)-C(8)-C(13)	4.8(8)
C(12)-Ta(1)-C(8)-C(13)	-121.3(10)
C(1)-Ta(1)-C(8)-C(13)	114.4(8)
C(1)-N(1)-C(7)-C(6)	-37.6(12)
Ta(1)-N(1)-C(7)-C(6)	178.0(8)
C(1)-N(2)-C(2)-C(3)	-23.7(13)
Ta(1)-N(2)-C(2)-C(3)	134.4(10)
C(7)-N(1)-C(1)-N(3)	10.7(15)
Ta(1)-N(1)-C(1)-N(3)	170.4(9)
C(7)-N(1)-C(1)-N(2)	-168.8(8)
Ta(1)-N(1)-C(1)-N(2)	-9.1(8)
C(7)-N(1)-C(1)-Ta(1)	-159.7(9)
C(2)-N(2)-C(1)-N(3)	-3.1(15)
Ta(1)-N(2)-C(1)-N(3)	-170.2(9)

C(2)-N(2)-C(1)-N(1)	176.4(9)
Ta(1)-N(2)-C(1)-N(1)	9.3(8)
C(2)-N(2)-C(1)-Ta(1)	167.1(10)
N(2)-Ta(1)-C(1)-N(3)	76(5)
N(1)-Ta(1)-C(1)-N(3)	-114(5)
C(10)-Ta(1)-C(1)-N(3)	161(5)
C(11)-Ta(1)-C(1)-N(3)	132(5)
C(9)-Ta(1)-C(1)-N(3)	-167(5)
Cl(2)-Ta(1)-C(1)-N(3)	31(5)
Cl(1)-Ta(1)-C(1)-N(3)	-61(5)
C(12)-Ta(1)-C(1)-N(3)	141(5)
C(8)-Ta(1)-C(1)-N(3)	-163(5)
N(2)-Ta(1)-C(1)-N(1)	-169.3(9)
C(10)-Ta(1)-C(1)-N(1)	-84.5(6)
C(11)-Ta(1)-C(1)-N(1)	-113.8(6)
C(9)-Ta(1)-C(1)-N(1)	-52.2(6)
Cl(2)-Ta(1)-C(1)-N(1)	145.6(5)
Cl(1)-Ta(1)-C(1)-N(1)	53.6(6)
C(12)-Ta(1)-C(1)-N(1)	-104.6(7)
C(8)-Ta(1)-C(1)-N(1)	-48.4(8)
N(1)-Ta(1)-C(1)-N(2)	169.3(9)
C(10)-Ta(1)-C(1)-N(2)	84.9(6)
C(11)-Ta(1)-C(1)-N(2)	55.5(6)
C(9)-Ta(1)-C(1)-N(2)	117.1(6)
Cl(2)-Ta(1)-C(1)-N(2)	-45.1(6)
Cl(1)-Ta(1)-C(1)-N(2)	-137.0(5)
C(12)-Ta(1)-C(1)-N(2)	64.7(8)
C(8)-Ta(1)-C(1)-N(2)	121.0(6)
N(1)-C(1)-N(3)-C(4)	-178.3(10)
N(2)-C(1)-N(3)-C(4)	1.1(15)
Ta(1)-C(1)-N(3)-C(4)	-70(5)
N(1)-C(1)-N(3)-C(5)	-0.3(15)
N(2)-C(1)-N(3)-C(5)	179.2(9)
Ta(1)-C(1)-N(3)-C(5)	108(5)
C(9)-C(8)-C(12)-C(11)	0.8(11)
C(13)-C(8)-C(12)-C(11)	-178.7(9)



Ta(1)-C(8)-C(12)-C(11)	62.0(6)
C(9)-C(8)-C(12)-C(17)	173.7(9)
C(13)-C(8)-C(12)-C(17)	-5.8(16)
Ta(1)-C(8)-C(12)-C(17)	-125.2(10)
C(9)-C(8)-C(12)-Ta(1)	-61.1(7)
C(13)-C(8)-C(12)-Ta(1)	119.4(10)
N(2)-Ta(1)-C(12)-C(8)	138.3(5)
N(1)-Ta(1)-C(12)-C(8)	47.9(8)
C(10)-Ta(1)-C(12)-C(8)	77.9(6)
C(11)-Ta(1)-C(12)-C(8)	116.9(8)
C(9)-Ta(1)-C(12)-C(8)	36.0(5)
Cl(2)-Ta(1)-C(12)-C(8)	-141.1(5)
Cl(1)-Ta(1)-C(12)-C(8)	-56.4(5)
C(1)-Ta(1)-C(12)-C(8)	102.0(7)
N(2)-Ta(1)-C(12)-C(11)	21.4(7)
N(1)-Ta(1)-C(12)-C(11)	-69.0(8)
C(10)-Ta(1)-C(12)-C(11)	-39.0(5)
C(9)-Ta(1)-C(12)-C(11)	-80.9(6)
Cl(2)-Ta(1)-C(12)-C(11)	102.0(5)
Cl(1)-Ta(1)-C(12)-C(11)	-173.3(5)
C(8)-Ta(1)-C(12)-C(11)	-116.9(8)
C(1)-Ta(1)-C(12)-C(11)	-14.9(9)
N(2)-Ta(1)-C(12)-C(17)	-97.3(9)
N(1)-Ta(1)-C(12)-C(17)	172.3(8)
C(10)-Ta(1)-C(12)-C(17)	-157.7(10)
C(11)-Ta(1)-C(12)-C(17)	-118.7(11)
C(9)-Ta(1)-C(12)-C(17)	160.4(10)
Cl(2)-Ta(1)-C(12)-C(17)	-16.7(9)
Cl(1)-Ta(1)-C(12)-C(17)	68.0(9)
C(8)-Ta(1)-C(12)-C(17)	124.4(12)
C(1)-Ta(1)-C(12)-C(17)	-133.6(8)
C(8)-C(12)-C(11)-C(10)	-0.6(11)
C(17)-C(12)-C(11)-C(10)	-173.6(9)
Ta(1)-C(12)-C(11)-C(10)	65.1(6)
C(8)-C(12)-C(11)-C(16)	174.5(9)
C(17)-C(12)-C(11)-C(16)	1.5(16)

Ta(1)-C(12)-C(11)-C(16)	-119.9(10)
C(8)-C(12)-C(11)-Ta(1)	-65.6(7)
C(17)-C(12)-C(11)-Ta(1)	121.3(9)
N(2)-Ta(1)-C(11)-C(10)	83.7(6)
N(1)-Ta(1)-C(11)-C(10)	24.1(6)
C(9)-Ta(1)-C(11)-C(10)	-37.8(5)
Cl(2)-Ta(1)-C(11)-C(10)	169.6(5)
Cl(1)-Ta(1)-C(11)-C(10)	-103.6(6)
C(12)-Ta(1)-C(11)-C(10)	-113.6(8)
C(8)-Ta(1)-C(11)-C(10)	-77.7(6)
C(1)-Ta(1)-C(11)-C(10)	57.6(6)
N(2)-Ta(1)-C(11)-C(12)	-162.7(6)
N(1)-Ta(1)-C(11)-C(12)	137.7(5)
C(10)-Ta(1)-C(11)-C(12)	113.6(8)
C(9)-Ta(1)-C(11)-C(12)	75.8(6)
Cl(2)-Ta(1)-C(11)-C(12)	-76.8(5)
Cl(1)-Ta(1)-C(11)-C(12)	10.0(7)
C(8)-Ta(1)-C(11)-C(12)	35.9(5)
C(1)-Ta(1)-C(11)-C(12)	171.2(5)
N(2)-Ta(1)-C(11)-C(16)	-38.3(8)
N(1)-Ta(1)-C(11)-C(16)	-97.9(8)
C(10)-Ta(1)-C(11)-C(16)	-122.0(10)
C(9)-Ta(1)-C(11)-C(16)	-159.7(9)
Cl(2)-Ta(1)-C(11)-C(16)	47.6(8)
Cl(1)-Ta(1)-C(11)-C(16)	134.4(7)
C(12)-Ta(1)-C(11)-C(16)	124.4(10)
C(8)-Ta(1)-C(11)-C(16)	160.3(9)
C(1)-Ta(1)-C(11)-C(16)	-64.4(8)
C(1)-N(3)-C(4)-C(3)	27.4(13)
C(5)-N(3)-C(4)-C(3)	-150.6(9)
N(3)-C(4)-C(3)-C(2)	-52.4(12)
N(2)-C(2)-C(3)-C(4)	50.8(12)
C(1)-N(3)-C(5)-C(6)	19.1(13)
C(4)-N(3)-C(5)-C(6)	-162.9(9)
N(3)-C(5)-C(6)-C(7)	-47.9(13)
N(1)-C(7)-C(6)-C(5)	56.9(12)

C(12)-C(11)-C(10)-C(9)	0.1(11)
C(16)-C(11)-C(10)-C(9)	-175.0(9)
Ta(1)-C(11)-C(10)-C(9)	68.0(7)
C(12)-C(11)-C(10)-C(15)	174.4(9)
C(16)-C(11)-C(10)-C(15)	-0.7(16)
Ta(1)-C(11)-C(10)-C(15)	-117.7(10)
C(12)-C(11)-C(10)-Ta(1)	-67.9(6)
C(16)-C(11)-C(10)-Ta(1)	117.0(10)
N(2)-Ta(1)-C(10)-C(9)	148.6(5)
N(1)-Ta(1)-C(10)-C(9)	85.6(5)
C(11)-Ta(1)-C(10)-C(9)	-114.8(8)
Cl(2)-Ta(1)-C(10)-C(9)	-127.9(5)
Cl(1)-Ta(1)-C(10)-C(9)	1.8(7)
C(12)-Ta(1)-C(10)-C(9)	-76.4(6)
C(8)-Ta(1)-C(10)-C(9)	-36.3(5)
C(1)-Ta(1)-C(10)-C(9)	116.5(5)
N(2)-Ta(1)-C(10)-C(11)	-96.6(6)
N(1)-Ta(1)-C(10)-C(11)	-159.6(6)
C(9)-Ta(1)-C(10)-C(11)	114.8(8)
Cl(2)-Ta(1)-C(10)-C(11)	-13.0(6)
Cl(1)-Ta(1)-C(10)-C(11)	116.6(5)
C(12)-Ta(1)-C(10)-C(11)	38.4(5)
C(8)-Ta(1)-C(10)-C(11)	78.5(6)
C(1)-Ta(1)-C(10)-C(11)	-128.7(6)
N(2)-Ta(1)-C(10)-C(15)	25.1(8)
N(1)-Ta(1)-C(10)-C(15)	-37.9(8)
C(11)-Ta(1)-C(10)-C(15)	121.7(10)
C(9)-Ta(1)-C(10)-C(15)	-123.5(10)
Cl(2)-Ta(1)-C(10)-C(15)	108.7(7)
Cl(1)-Ta(1)-C(10)-C(15)	-121.7(7)
C(12)-Ta(1)-C(10)-C(15)	160.1(9)
C(8)-Ta(1)-C(10)-C(15)	-159.8(9)
C(1)-Ta(1)-C(10)-C(15)	-7.0(8)
C(12)-C(8)-C(9)-C(10)	-0.8(11)
C(13)-C(8)-C(9)-C(10)	178.7(9)
Ta(1)-C(8)-C(9)-C(10)	-62.4(6)

C(12)-C(8)-C(9)-C(14)	-177.3(9)
C(13)-C(8)-C(9)-C(14)	2.2(16)
Ta(1)-C(8)-C(9)-C(14)	121.1(10)
C(12)-C(8)-C(9)-Ta(1)	61.6(7)
C(13)-C(8)-C(9)-Ta(1)	-118.9(10)
C(11)-C(10)-C(9)-C(8)	0.4(11)
C(15)-C(10)-C(9)-C(8)	-173.9(9)
Ta(1)-C(10)-C(9)-C(8)	66.7(7)
C(11)-C(10)-C(9)-C(14)	177.1(9)
C(15)-C(10)-C(9)-C(14)	2.8(16)
Ta(1)-C(10)-C(9)-C(14)	-116.6(9)
C(11)-C(10)-C(9)-Ta(1)	-66.3(6)
C(15)-C(10)-C(9)-Ta(1)	119.4(10)
N(2)-Ta(1)-C(9)-C(8)	-152.2(5)
N(1)-Ta(1)-C(9)-C(8)	151.3(6)
C(10)-Ta(1)-C(9)-C(8)	-116.1(8)
C(11)-Ta(1)-C(9)-C(8)	-77.8(6)
Cl(2)-Ta(1)-C(9)-C(8)	-31.5(7)
Cl(1)-Ta(1)-C(9)-C(8)	65.2(5)
C(12)-Ta(1)-C(9)-C(8)	-36.0(5)
C(1)-Ta(1)-C(9)-C(8)	175.4(5)
N(2)-Ta(1)-C(9)-C(10)	-36.1(6)
N(1)-Ta(1)-C(9)-C(10)	-92.6(6)
C(11)-Ta(1)-C(9)-C(10)	38.3(5)
Cl(2)-Ta(1)-C(9)-C(10)	84.6(6)
Cl(1)-Ta(1)-C(9)-C(10)	-178.7(5)
C(12)-Ta(1)-C(9)-C(10)	80.1(6)
C(8)-Ta(1)-C(9)-C(10)	116.1(8)
C(1)-Ta(1)-C(9)-C(10)	-68.5(6)
N(2)-Ta(1)-C(9)-C(14)	82.9(8)
N(1)-Ta(1)-C(9)-C(14)	26.3(8)
C(10)-Ta(1)-C(9)-C(14)	119.0(10)
C(11)-Ta(1)-C(9)-C(14)	157.3(9)
Cl(2)-Ta(1)-C(9)-C(14)	-156.5(6)
Cl(1)-Ta(1)-C(9)-C(14)	-59.8(8)
C(12)-Ta(1)-C(9)-C(14)	-161.0(9)

C(8)-Ta(1)-C(9)-C(14)	-125.0(10)
C(1)-Ta(1)-C(9)-C(14)	50.5(8)
C(8)-C(12)-C(17)-C(18)	-91.8(13)
C(11)-C(12)-C(17)-C(18)	80.0(13)
Ta(1)-C(12)-C(17)-C(18)	169.4(8)

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Observed and calculated structure factors for (C<sub>5</sub>Me<sub>4</sub>Et)Ta(hpp)Cl<sub>2</sub>.

h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
2	0	0	115	104	2	5	4	0	721	733	6	0	9	0	614	627	14	2	-8	1	0	56	1	-7	-3	1	402	413	5
3	0	0	690	617	5	6	4	0	151	144	7	1	9	0	500	517	17	3	-8	1	378	388	15	-6	-3	1	68	78	42
4	0	0	1418	1209	9	7	4	0	425	427	10	2	9	0	217	227	21	4	-8	1	276	274	16	-5	-3	1	697	710	7
5	0	0	224	212	7	8	4	0	454	443	7	3	9	0	141	152	103	5	-8	1	137	13	102	-4	-3	1	1073	1093	9
6	0	0	310	296	6	9	4	0	227	227	21	4	9	0	464	469	13	6	-8	1	225	277	21	-3	-3	1	632	662	7
7	0	0	603	549	7	10	4	0	207	233	15	5	9	0	160	181	43	-10	-7	1	0	16	1	-2	-3	1	623	618	5
8	0	0	244	228	17	11	4	0	259	289	20	6	9	0	113	89	26	-9	-7	1	364	358	10	-1	-3	1	1291	1297	13
9	0	0	245	234	21	-8	5	0	312	286	20	7	9	0	261	278	10	-8	-7	1	484	491	8	0	-3	1	1358	1369	11
10	0	0	435	415	11	-7	5	0	416	406	15	8	9	0	219	249	15	-7	-7	1	66	93	47	1	-3	1	446	450	4
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h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s	h	k	l	10Fo	10Fc	10s
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3																													



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