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Titanacarborane mediated C–N bond forming/breaking reactions

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ABSTRACT

Constrained-geometry titanacarboranes [σ:η^1:η^5-(OCH$_2$)(R$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NR$_2$) (R = Me, Et) are synthesized via an unexpected reaction of [Me$_3$NH][μ-7,8-CH$_2$OCH$_2$-7,8-C$_2$B$_9$H$_{10}$] with Ti(NR$_2$)$_4$ (R = Me, Et), involving a C–O bond cleavage and C–N bond formation. These complexes can be readily converted to new amide species or alkoxide by reacting with amines or esters, respectively. Insertion of a series of unsaturated molecules into the Ti–N bond of the aforementioned complexes results in the formation of various half-sandwich titanacarboranes. [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NMe$_2$) is also able to efficiently catalyze the hydroamination of carbodiimides and the transamination of guanidines. These results are summarized in this brief account.

1. Introduction

Ligands impose a dominant control over both chemical and physical properties of the resulting metal complexes [1–12]. Transition metal complexes bearing linked cyclopentadienyl-amido ligands, often referred to as constrained-geometry complexes (CGCs), have found wide interest both in academia and industry since their first description in 1990 [13,14]. Particularly, catalytic systems based on group 4 metal compounds give access to a large array of polymers with unique material properties and considerable commercial values [4–12,15–27]. In this connection, replacement of a negative cyclopentadienyl (Cp) in the classical constrained-geometry ligand by an isolobal, dinegative dicarbollide ion can provide new metal/charge combinations, which would have an impact on the properties of resultant metal complexes. The chemistry of this kind of group 4 metal CGCs has been studied by us and other groups [9–12,28–35], which is discussed in a recent review [11]. This brief account summarizes our very recent work on the synthesis, reactivity and catalysis of [σ:η^1:η^5-(OCH$_2$)(R$_2$NCH$_2$)C$_2$B$_9$H$_9$]-Ti(NR$_2$) (R = Me, Et) [36–38].

2. Synthesis

μ-1,2-CH$_2$OCH$_2$-1,2-C$_2$B$_{10}$H$_{10}$ was converted to [Me$_3$NH][μ-7,8-CH$_2$OCH$_2$-7,8-C$_2$B$_9$H$_{10}$] in 95% isolated yield in KOH/EtOH solution, followed by treatment with Me$_3$NHCl. This dicarbollide salt reacted readily with 1 equiv. of M(NMe$_2$)$_4$ (M = Zr, Hf) in toluene to give the expected amine elimination products [η^5-(CH$_2$OCH$_2$)C$_2$B$_9$H$_9$]M-(NMe$_2$)$_2$(NHMe$_2$) (M = Zr, Hf) in almost quantitative yields [36]. However, unprecedented ring-opening products, [σ:η^1:η^5-(OCH$_2$)(R$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NR$_2$) (R = Me, Et), were isolated in high yields from the reactions of Ti(NR$_2$)$_4$ (R = Me, Et) with [Me$_3$NH][μ-7,8-CH$_2$OCH$_2$-7,8-C$_2$B$_9$H$_{10}$] in refluxing toluene (Scheme 1) [36]. This route can serve as a convenient and practical method for the preparation of constrained-geometry half-sandwich titanacarboranes with two different functional sidearms in a single step. One sidearm in the complexes is strongly bonded to the Ti center by taking the advantage of its high oxophilicity whereas the other is hemilabile in nature. The solid–state structures of [σ:η^1:η^5-(OCH$_2$)(R$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NR$_2$) (R = Me, Et) are confirmed by single-crystal X-ray diffraction studies to adopt a three-legged piano stool structure containing an η^5-dicarbollyl ligand, one amido unit, a tethered amine and an alkoxy group in the basal positions.

The NMR experiments on the reaction of Ti(NR$_2$)$_4$ (R = Me, Et) with [Me$_3$NH][μ-7,8-CH$_2$OCH$_2$-7,8-C$_2$B$_9$H$_{10}$] offered some insight into the reaction pathway. A major species was observed upon heating the NMR solution at 80 °C, which was slowly converted to the above isolated product. Many attempts to isolate the intermediate formed at 80 °C failed due to its instability. Therefore, an unsaturated molecule was employed to trap this intermediate. Treatment of [Me$_3$NH][μ-7,8-CH$_2$OCH$_2$-7,8-C$_2$B$_9$H$_{10}$] with 1 equiv. of Ti(NEt$_2$)$_4$ at 80 °C for 6 h (monitored by ^{11}B NMR), followed by addition of excess CS$_2$, gave an insertion product [σ-(OCH$_2$)(Et$_2$NHCH$_2$)C$_2$B$_9$H$_{10}$]-Ti(η^2-S$_2$CNEt$_2$)$_3$ in 36% yield (Scheme 1).

The isolation and full characterization of this insertion product helps us to understand the possible reaction pathway, as shown in

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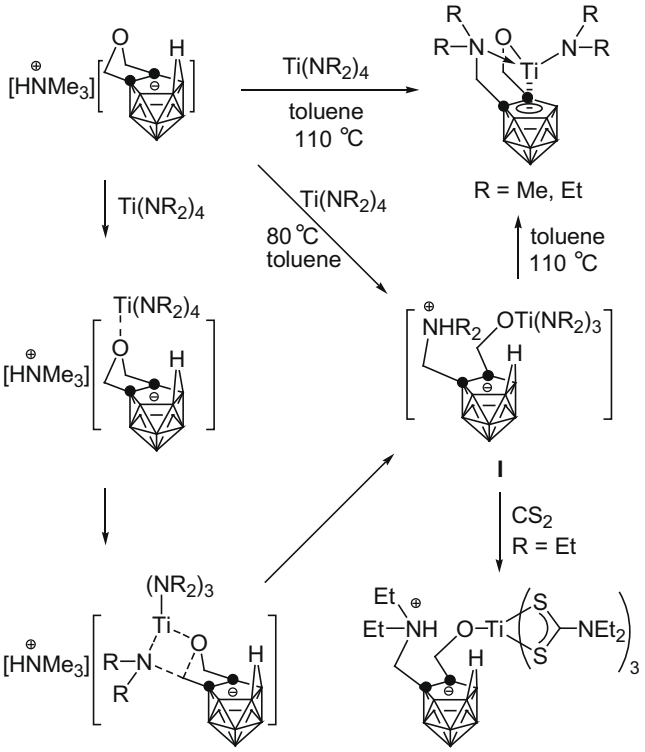
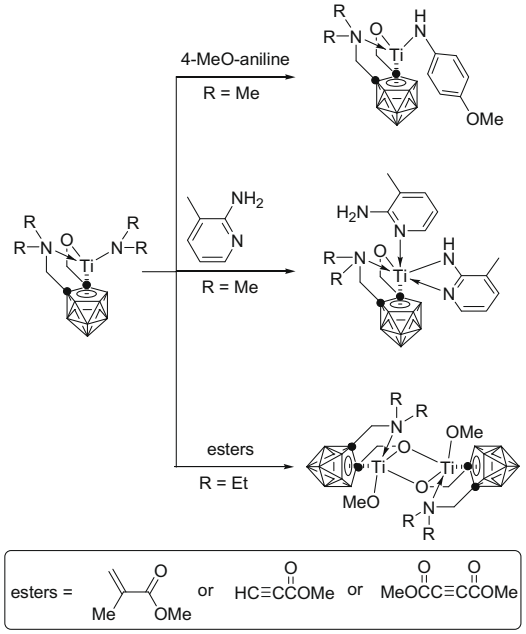
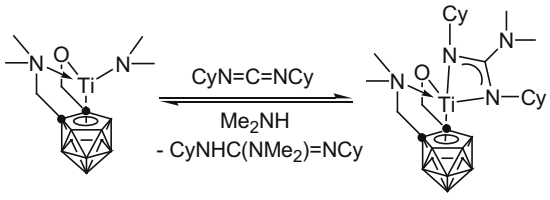


Scheme 1.

Scheme 1. Coordination of the oxygen atom to the Ti atom, followed by a σ-bond metathesis via the C–O bond cleavage and the formation of a new N–C bond, leads to the production of the intermediate **I**. Insertion of CS_2 into the Ti–N bonds in **I** generates $[\sigma\text{-}(OCH_2)(R_2NHCH_2)C_2B_9H_{10}]Ti(\eta^2\text{-}S_2CNR_2)_3$. On the other hand,



Scheme 2.



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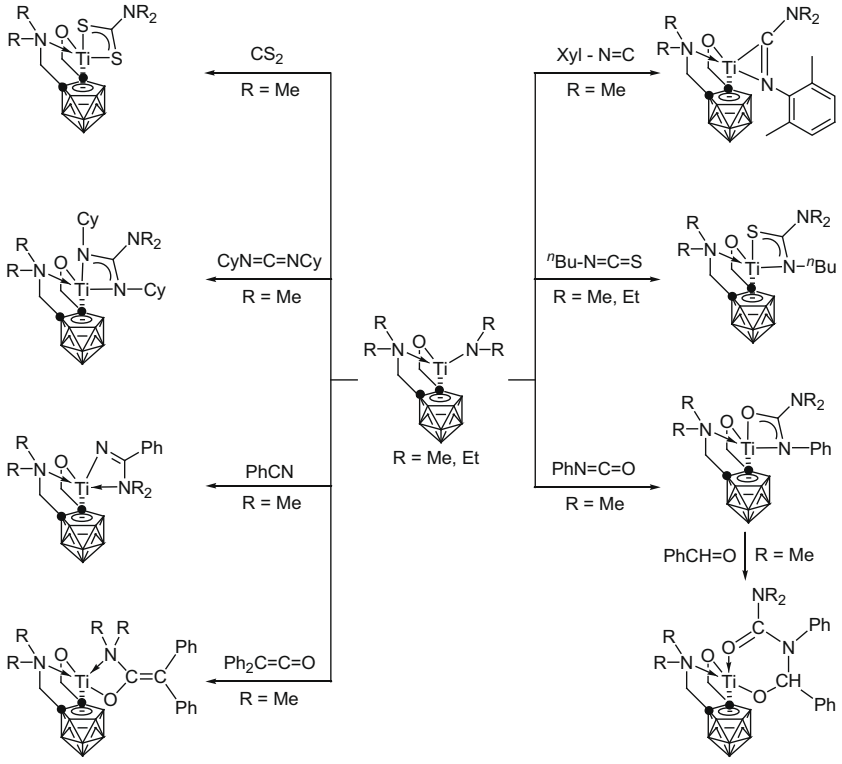
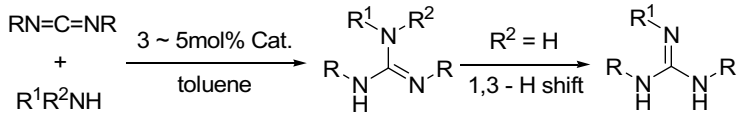


Scheme 3.

Table 1
Catalytic addition of amines to carbodiimides.



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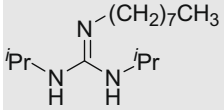
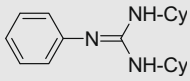
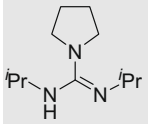
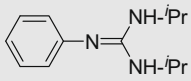
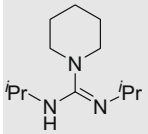
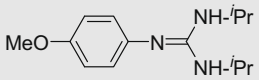
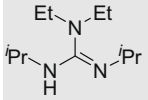
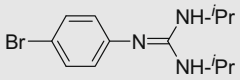
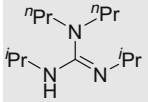
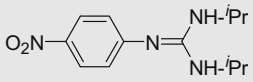
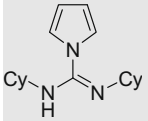
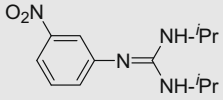
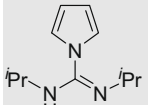
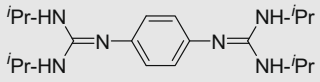
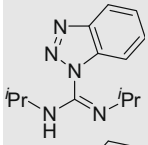
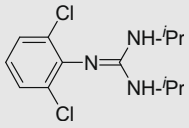
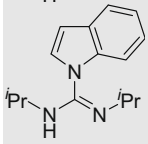
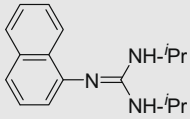
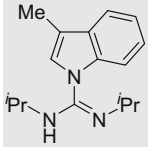
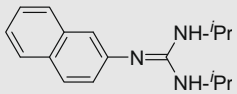
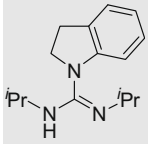
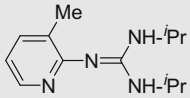
	92		96
	97		97
	95		97
	93		95
	90		92
	71		87
	70		92
	71		94
	82		90
	70		92
	88		91

intramolecular amine elimination of **I** affords the final product, which is promoted by heat. The higher oxophilicity of the Ti atom over the Zr and Hf, resulted from the size effect [39,40], facilitates the breaking of the C–O bond and results in different reaction pathways in the reaction of $M(NMe_2)_4$ with $[Me_3NH][\mu\text{-}7,8\text{-}CH_2OCH_2\text{-}7,8\text{-}C_2B_9H_{10}]$.

3. Reactivity

3.1. Exchange reaction

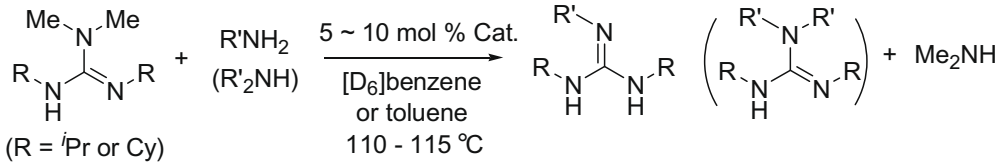
Amine exchange reaction is a useful method for the preparation of metal amides. Complex $[\sigma{:}\eta^1{:}\eta^5\text{-}(OCH_2)(Me_2NCH_2)C_2B_9H_9]$-

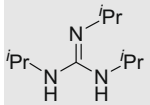
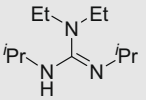
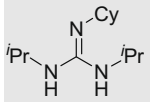
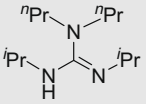
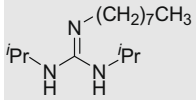
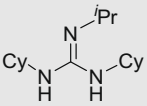
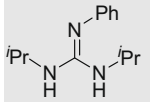
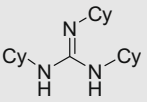
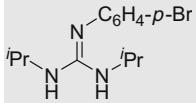
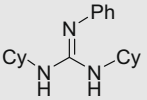
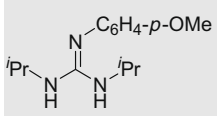
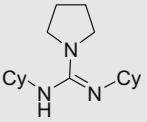
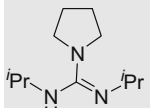
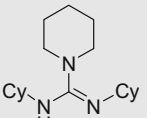
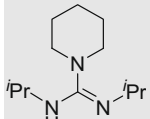
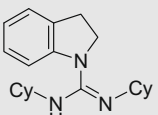
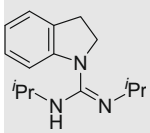
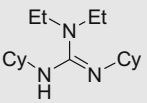
$Ti(NMe_2)$ reacted readily with 1 equiv. of 4-methoxy-aniline or 2 equiv. of 2-amino-3-picoline in toluene at room temperature to give $[\sigma:\eta^1:\eta^5\text{-}(OCH_2)(Me_2NCH_2)C_2B_9H_9]Ti[NH(C_6H_4\text{-}4\text{-}OMe)]$ or $[\sigma:\eta^1:\eta^5\text{-}(OCH_2)(Me_2NCH_2)C_2B_9H_9]Ti[\sigma:\eta^1\text{-}(2\text{-}NH\text{-}3\text{-}CH_3\text{-}C_5H_3N)][\eta^1\text{-}C_5H_3N\text{-}2\text{-}NH_2\text{-}3\text{-}CH_3]$ in 61% and 46% yields, respectively (Scheme 2). The ^1H NMR spectra indicated the absence of the Me_2N amido groups and the presence of the corresponding aromatic proton resonances in both products.

An X-ray analysis reveals that $[\sigma:\eta^1:\eta^5\text{-}(OCH_2)(Me_2NCH_2)C_2B_9H_9]Ti[\sigma:\eta^1\text{-}(2\text{-}NH\text{-}3\text{-}CH_3\text{-}C_5H_3N)][\eta^1\text{-}C_5H_3N\text{-}2\text{-}NH_2\text{-}3\text{-}CH_3]$ adopts a distorted-octahedral geometry by an η^5-dicarbollyl ligand, one oxygen and one nitrogen from the sidearms, and three nitrogen atoms from the two picoline units. This structure clearly implies that the coordination number of the Ti atom can be increased by 2 units, facilitating the subsequent insertion of unsaturated molecules.

The amido group in the Ti amide complex can be substituted by an alkoxy unit. Stoichiometric reactions of $[\sigma:\eta^1:\eta^5\text{-}(OCH_2)(Et_2NCH_2)C_2B_9H_9]Ti(NEt_2)$ with methyl esters such as methyl metacrylate (MMA), methyl propiolate or dimethyl acetylenedicarbox-

Table 2
Catalytic transamination of guanidines with amines.



Product	Yield (%)[a]	Product	Yield (%)[a]
	>95(94)		75
	>95(92)		65
	>95		>95(97)
	>95		>95(98)
	>95		>95
	>95		>95(97)
	>95		>95(96)
	>95		>95(83)
	>95		78

[a] Yields determined by integration of ^1H NMR relative to internal standard of ferrocene; isolated yield in parentheses from scale-up reactions.

ylate afforded the same dimeric complex [{σ:η^1:η^5-(μ-OCH$_2$)(Et$_2$NCH$_2$)C$_2$B$_9$H$_9$}Ti(OMe)]$_2$ in *ca.* 80% isolated yields (Scheme 2).

3.2. Insertion reaction

As shown in Scheme 3, S=C=S, Cy–N=C=N–Cy, Ph–C≡N, Ph$_2$C=C=O, Xyl–N=C, ^{n}Bu–N=C=S, and Ph–N=C=O are able to insert into the Ti–N σ-bond of [σ:η^1:η^5-(OCH$_2$)(R$_2$NCH$_2$)C$_2$B$_9$H$_9$]-Ti(NR$_2$) (R = Me, Et) to form an array of insertion products. Among these, the reactions of Ph$_2$C=C=O and PhN=C=O are of particular interesting.

Insertion of the C=C or C=O bond of a ketene into the Ti–O or M–N bond has been documented [41–44]. In the present case, only C=O insertion product [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]-Ti[σ:η^1-OC(NMe$_2$)=CPh$_2$] was isolated.

It has been reported that PhNCO can insert into either a Ti–N or Ti–O bond [45–47]. Reaction of [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NMe$_2$) with PhNCO afforded exclusively [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti[η^2-OC(NMe$_2$)NPh]. Benzaldehyde can further insert into the newly formed Ti–N bond to give a six-membered metallacycle with the formation of a new C–N bond. The Ti–O bond remains intact. The two amido fragments on the six-membered metallacycle are in *trans* positions due to the steric reasons [36].

4. Catalysis

4.1. Hydroamination of carbodiimide

On the basis of the amine exchange and aforementioned insertion reactions, reactivity of the insertion products toward amines was investigated in the hope to realize a catalytic cycle. Only [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti[η^2-CyNC(NMe$_2$)NCy] can react with Me$_2$NH to regenerate [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NMe$_2$) with the formation of CyN=C(NMe$_2$)NHCy as indicated by NMR (Scheme 4). The catalytic hydroamination reaction of carbodiimides using [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]-Ti(NMe$_2$) as the catalyst was then explored, and the results are compiled in Table 1.

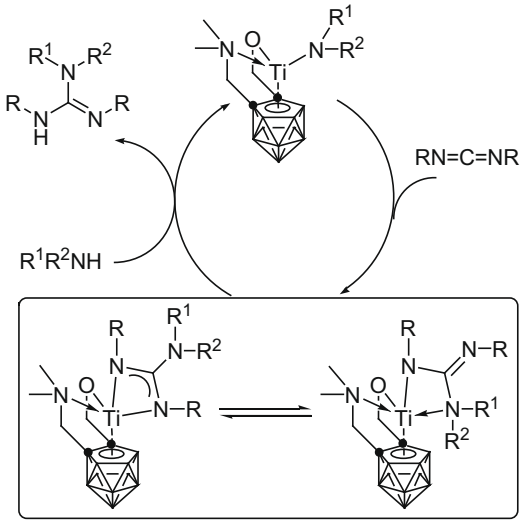
These results indicate that complex [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NMe$_2$) is a very robust, effective, and elegant catalyst for the catalytic addition of primary and secondary aliphatic and aromatic amines to carbodiimides with good functional group tolerance. Less nucleophilic pyrrole, indole, and benzotriazole also offer good yields.

A possible catalytic cycle is proposed in Scheme 5. Interaction of the RN=C=NR with amide yields guanidinates [48–53]. Acid–base reaction between guanidinate complexes and R$_1$R$_2$NH releases the products, meanwhile, regenerates the amide species to complete this catalytic cycle. Significantly different from Richeson's work [54], no titanium imido species are involved in this system, which largely broadens the reaction scope. The hemilabile nature of amine sidearm might play a role in the catalytic cycle since it could reversibly coordinate to the Ti center, thus stabilizing a highly reactive intermediate.

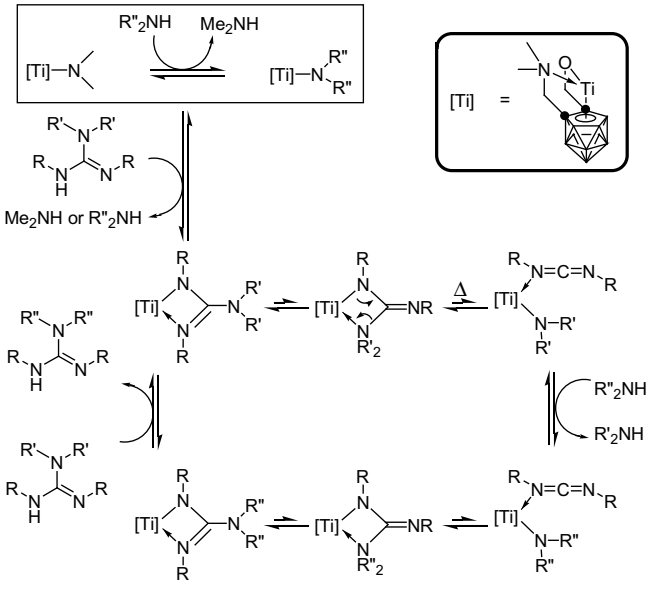
4.2. Transamination of guanidine

Stimulated by our studies on [σ:η^5-(C$_9$H$_6$)C$_2$B$_9$H$_{10}$]Zr(NMe$_2$)(DME) mediated C–N bond cleavage of the coordinated guanidinate ligand [55], we found that [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]Ti(NMe$_2$) can also catalyze the transamination of guanidines. The results are compiled in Table 2. Except for Et$_2$NH and ^{n}Pr$_2$NH, the yields are almost quantitative spanning a broad substrate scope of primary, secondary, heterocyclic, aliphatic and aromatic amines. It is noted that the trisubstituted guanidines are much more stable than the tetrasubstituted ones in this reaction system. No further transamination reaction of the products was observed. The catalyst can also tolerate functional groups such as methoxyl and bromo.

A catalytic cycle for the transamination of guanidines is proposed in Scheme 6. Complex [σ:η^1:η^5-(OCH$_2$)(Me$_2$NCH$_2$)C$_2$B$_9$H$_9$]-Ti(NMe$_2$) undergoes amine exchange with R"$_2$NH to give new amide species [Ti]—NR$''_2$. Acid–base reaction of [Ti]–NMe$_2$ or [Ti]—NR$''_2$ with guanidine R—N=C(NR$'_2$)NH—R results in the formation of [Ti]—{η^2-C(NR$'_2$)(NR)$_2$} to enter the catalytic cycle. Isomerization followed by the C–N bond cleavage, which is promoted by heat, forms the amide [Ti]—NR$'_2$ complex and carbodiimide. Amine exchange reaction with R$''_2$NH releases R$'_2$NH and generates [Ti]—NR$''_2$ species. Insertion of carbodiimide into the Ti–N bond and isomerization produces [Ti]—{η^2-C(NR$''_2$)(NR)$_2$}. Guanidine exchange affords product R—N=C(NR$''_2$)NH—R and regenerates complex [Ti]—{η^2-C(NR$'_2$)(NR)$_2$} to complete the catalytic cycle. The mechanism of the reaction is obviously different from the



Scheme 5.



Scheme 6.

titanium-imide catalyzed system in which the regeneration of Ti=N moiety is essential [54].

5. Conclusion and perspective

Half-sandwich constrained-geometry titanacarboranes $[\sigma:\eta^1:\eta^5\text{-}(OCH_2)(R_2NCH_2)C_2B_9H_9]Ti(NR_2)$ (R = Me, Et) have two different functional sidearms: one strongly bonds to the Ti atom preserving the integrity of the constrained-geometry unit, and the other (amine) coordinates reversibly to the Ti atom stabilizing the reactive intermediate. Such a unique structural feature makes these complexes as robust and efficient catalysts for hydroamination of carbodiimides and transamination of guanidines. It is anticipated that this type of complexes might catalyze other reactions such as hydroboration and hydrophosphination. On the other hands, $[\sigma:\eta^1:\eta^5\text{-}(OCH_2)(R_2NCH_2)C_2B_9H_9]Ti(NR_2)$ (R = Me, Et) are metal-centered chiral complexes. If a pure enantiomer of the planar chiral dicarbollyl ligands could be prepared, optically pure metallacarboranes could be achieved, which would make asymmetrical catalysis feasible. This work is awaiting exploration.

Acknowledgements

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