## Homogeneous Catalysis

## A Well-Defined Aluminum-Based Lewis Acid as an Effective **Catalyst for Diels-Alder Transformations**

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Abstract: A catalytically active aluminum-based system for Diels-Alder transformations is reported. The system was generated by mixing a β-diketiminate-stabilized aluminum bistriflate compound with Na[BAr<sup>Cl</sup><sub>4</sub>] (Ar<sup>Cl</sup>=3,5-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>). Solid-state analysis of the catalytic system reveals a unique structure incorporating a two-dimensional coordination polymer. According to the experimental results obtained from several Diels-Alder transformations, the aluminum-based system appears to be a more practical and more robust alternative to the recently reported compounds based on carbon and silicon cations.

Lewis acids (LAs) have been known to act as stoichiometric or catalytic reagents in various organic transformations, including allylic substitutions, aldol transformations, C-H activation, cyclization, and cycloaddition reactions.<sup>[1]</sup> As aluminum is not only inherently electron deficient but is also a highly electron-positive element, it is not surprising that reagents based on this metal have been widely used for organic transformations including Diels-Alder reactions. However, on examining a recent book by Yamamoto and Ishihara<sup>[1a]</sup> on Al-based reagents involved in Diels-Alder cycloadditions, it was evident that catalytic transformations, especially those involving less reactive dienes and dienophiles (see below), were not well developed. In fact, the reactions that could be classified as catalytic employed predominantly cyclopentadiene, which is among the more reactive dienes. Furthermore, the vast majority of these Al-based systems were prepared and used in situ without any information on the structural properties of the active species. This could be quite problematic because several recent reports indicated that the observed catalytic activity was due not to the Lewis acid properties of the investigated compound(s) but to their ability to generate hidden Brønsted acids (HBAs).<sup>[2,3,4]</sup>

For example, Fringuelli and co-workers<sup>[5]</sup> identified AlCl<sub>3</sub>·2THF, classified as an aluminum-based Lewis acid system

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cand details regarding single crystal X-ray analysis, is available on the WWW under http://dx.doi.org/10.1002/chem.201502205.

by the work of Yamamoto and Ishihara, as a very reactive system for various Diels-Alder reactions. In fact, the authors of that work refer to the examined transformations as being AICl<sub>3</sub>-catalyzed cycloadditions.<sup>[5]</sup> We believe that this could not be further from the truth, simply because all experimental procedures in this work were performed without the use of glovebox or Schlenk techniques, resulting in AICI<sub>3</sub> being subjected to severe hydrolysis. This assessment is additionally supported by observations made by Cowley and co-workers, who emphasized that molecular AICI<sub>3</sub>·2THF is an extremely air- and moisture-sensitive material.<sup>[6a]</sup> Additionally, mixing AlCl<sub>3</sub> (i.e., Al<sub>2</sub>Cl<sub>6</sub>) with THF, in different ratios and under dry conditions, could form several different products, including [AlCl<sub>2</sub>(THF)<sub>4</sub>][AlCl<sub>4</sub>], adding to the overall uncertainty when attempting to identify the active species.<sup>[6b,c]</sup> Therefore, there is a great probability that, in the work by Fringuelli and co-workers, AlCl<sub>3</sub> was hydrolyzed, producing an HBA (presumably HCl) that was then responsible for the observed catalytic activity. In fact, this study, as was the case for many other reports, did not include any of the control experiments suggested by Hintermann and coworkers to minimize, if not completely eliminate, the possibility of HBA activity.<sup>[2e]</sup>

Considering all the evidence and being inspired by the work on silylium cations<sup>[7]</sup> involved in catalytic Diels-Alder reactions, our goal was to prepare a well-defined and well-characterized triflate-containing aluminum species  $LAI(OTf)_2$  (L = HC(MeC-NAr)<sub>2</sub>; Ar = 2,6-*i*Pr<sub>2</sub>C<sub>6</sub>H<sub>3</sub>) that is capable of acting as a Lewis acid catalyst. We synthesized a  $\beta$ -diketiminate-supported aluminum bistriflate compound that, when mixed with Na[BAr<sup>Cl</sup><sub>4</sub>]  $(Ar^{Cl} = 3,5-Cl_2C_6H_3)$ , catalyzed several Diels-Alder transformations. Additional experiments were also performed to gain evidence against HBA activity.

The overall synthetic pathway for the preparation of the target bistriflate compound LAI(OTf)<sub>2</sub> is summarized in Scheme 1. Even though LAICl<sub>2</sub> had been previously synthesized,<sup>[8]</sup> for this work the precursor has been prepared by in-



Scheme 1. General synthetic procedures. Reaction conditions: a) nBuLi (1.0 equiv.), 2 h, AlCl<sub>3</sub> (1.0 equiv.), overnight, toluene; b) AlOTf (2.1 equiv.), overnight, 1,2-difluorobenzene.

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*situ* lithiation of the corresponding aminoimine (**L**–H) followed by the addition of 1 equivalent of AlCl<sub>3</sub>.<sup>[9]</sup> The <sup>27</sup>Al NMR spectroscopic identification of the dichloro precursor (**L**AlCl<sub>2</sub>) revealed the presence of a signal at  $\delta_{Al} \approx 102$  ppm, whereas the other multinuclear NMR data were consistent with the reported values.

As silver triflate (AgOTf) seemed to be the most appropriate reagent for the triflate-for-chloride exchange,<sup>[10]</sup> it was not surprising to observe the formation of the target bistriflate compound LAI(OTf)<sub>2</sub> by treating the dichloro precursor with 2.1 equivalents of AgOTf (Scheme 1). <sup>27</sup>Al NMR spectroscopy clearly indicated that the substrate exchange had occurred as the original signal at  $\delta_{AI} \approx 102$  ppm was replaced by an upfield signal at  $\delta_{AI} \approx 60$  ppm. The presence of triflate ligand(s) was confirmed by a signal at  $\delta_F = -77.3$  ppm in the <sup>19</sup>F NMR spectrum. Single crystal X-ray analysis (Figure 1) showed that this



**Figure 1.** Molecular structure for LAI(OTf)<sub>2</sub>. Thermal ellipsoids have been drawn at 50% probability level. All hydrogen atoms have been omitted for clarity. Selected bond lengths (Å) and angles (°): Al1–N1 1.853(3), Al1–N2 1.841(3), Al1–O1 1.765(3), Al1–O4 1.769(3), S1–O1 1.509(3), S1–O2 1.417(3), S1–O3 1.420(3), S2–O4 1.508(2), S2–O5 1.413(3), S2–O6 1.408(3); N1-Al1-N2 101.3(1), O1-Al1-O4 106.1(1).

bistriflate species, reminiscent of the precursor, was four-coordinate.<sup>[11]</sup> This might be somewhat unusual, considering the multidentate nature of triflate substituents<sup>[12]</sup> and the ability of similar complexes to expand the coordination number to six.<sup>[10a]</sup> The Al-N bond lengths (1.841(3) and 1.853(3) Å) and the N1-Al1-N2 bond angle (101.27(12)°) for [LAI(OTf)<sub>2</sub>] shortened (by ca. 0.04 Å, Figure 1) and widened (by ca. 2°), respectively, in comparison to [LAICI<sub>2</sub>],<sup>[8]</sup> potentially hinting at electron density depletion at the aluminum center after the ligand exchange. The Al-O bond lengths (1.765(3) and 1.769(3) Å) certainly support this conclusion, as these bond lengths are the shortest among crystallographically elucidated triflate-containing aluminum compounds.<sup>[10a,c, 13]</sup> For example, the reported Al-OTf bond lengths range from 1.807(10)-1.877(3) for 4coordinate<sup>[13a,b,c]</sup> to 1.857(12)-2.074(4) for 5-cooordinate<sup>[10c, 13d]</sup> and 1.922(2)-1.982(2) for 6-coordinate<sup>[10a]</sup> aluminum compounds that contain one or two triflate ligands.

After thorough characterization of LAI(OTf)<sub>2</sub>, our next task was to investigate whether it would be capable of catalyzing Diels–Alder transformations. For this purpose, we investigated

the cycloaddition of 2,3-dimethylbutadiene (1), which is 250 times less reactive than cyclopentadiene, and methyl vinyl ketone (2).<sup>[2b]</sup> Unfortunately, LAI(OTf)<sub>2</sub>, using 5.0 mol%, was not active for the targeted transformations, although <sup>1</sup>H NMR spectroscopic evidence suggested dienophile coordination/activation at the central aluminum. Subsequently, we decided to further activate the bistriflate compound by the addition of 1 equivalent of Na[BAr<sup>CI</sup><sub>4</sub>] (Ar<sup>CI</sup>=3,5-CI<sub>2</sub>C<sub>6</sub>H<sub>3</sub>). Indeed, the addition of Na[BAr<sup>CI</sup><sub>4</sub>] resulted in a quite catalytically active system that was capable of catalyzing the target Diels–Alder reaction (see below; Table 1, entry 1). It is worth noting that the cyclo-addition did not occur in the presence of only Na[BAr<sup>CI</sup><sub>4</sub>].



**Figure 2.** Graphical representation (ltop) and the molecular structure (bottom) for {[LAI(OTf)<sub>2</sub>Na]<sub>2</sub>[BAr<sup>CI</sup><sub>4</sub>]<sub>2</sub>]<sub>n</sub>. Thermal ellipsoids (apart from CF<sub>3</sub> and non-bonding Dip groups) have been drawn at 50% probability level. All hydrogen atoms and [BAr<sup>CI</sup><sub>4</sub>]<sup>-</sup> anions (apart from coordinating Cl atoms) have been omitted for clarity. Selected bond lengths (Å) and angles (°): Al1–N1 1.814(3), Al1–N2 1.849(3), Al1–O1 1.772(3), Al1–O4 1.770(3), S1–O1 1.497(2), S1–O2 1.417(3), S1–O3 1.419(3), S2–O4 1.485(3), S2–O5 1.425(3), S2–O6 1.427(3), Na1–O2 2.368(3), Na1–O6 2.334(3), Na1–O3<sup>#</sup> 2.251(3), Na1–Cl2 2.867(3), Na1–Cl7 3.033(2); N1-Al1-N2 105.2(2), O1-Al1-O4 106.3(1).



in light of this initially exciting observation, it was of paramount importance to gather more spectroscopic information about the active species before we further investigated its catalytic activities. Multinuclear NMR spectroscopy clearly suggested that the reaction between the bistriflates and Na[BAr<sup>Cl</sup><sub>4</sub>] has occurred. For example, the <sup>27</sup>Al NMR signal associated with the initial bistriflate compound disappeared, presumably due to peak broadening, whereas the <sup>19</sup>F NMR signal for the triflate ligand shifted downfield to  $\delta = -76.5$  ppm, suggesting a higher "degree of coordination" for the triflate ligand(s).<sup>[14]</sup> <sup>1</sup>H NMR spectroscopy indicated the formation of new species but without any evidence of decomposition/hydrolysis that is, formation of signal(s) linked to, for example, the free/protonated  $\beta$ -diketiminate ligand.<sup>[15]</sup> However, the lack of any precipitate formation suggested the absence of the targeted NaOTf elimination and formation of a charged aluminum species.

Fortunately, after numerous crystallization attempts we succeeded in obtaining a few crystals for the LAI(OTf)<sub>2</sub>/Na[BAr<sup>CI</sup><sub>4</sub>] system that were suitable for X-ray analysis, which revealed an unexpected structure (Figure 2). Although both OTf ligands were still coordinated to the aluminum center, they were additionally coordinated, through different O atoms, to an Na ion, forming a [LAI(OTf)<sub>2</sub>Na]<sup>+</sup> unit. One of the OTf ligands and one of the aryl substituents were further coordinated to a second Na ion belonging to a different cationic unit, and vice versa, effectively creating a dimeric [LAI(OTf)<sub>2</sub>Na]<sub>2</sub><sup>2+</sup> fragment. Each sodium cation was further weakly coordinated by two CI atoms found on two distinct [BAr<sup>CI</sup><sub>4</sub>]<sup>-</sup> anions, resulting in the formation of a 2-dimensional coordination polymer. To our knowledge, this is the first example of a coordination polymer that involves a tetraaryl borate anion. Nevertheless, we do not expect that this particular solid-state structure is retained in solution, as the Na ion is weakly coordinated by the anion and the aryl substituent.

With this information in hand, we attempted a series of Diels-Alder reactions between the diene 1 and several different dienophiles in the presence of 5 mol% of the LAI(OTf)<sub>2</sub>/ Na[BAr<sup>Cl</sup><sub>4</sub>] system (Table 1). In most cases the LAI(OTf)<sub>2</sub>/Na[-BAr<sup>Cl</sup><sub>4</sub>] system proved to be an excellent catalyst for the cycloaddition of the unsaturated acyclic ketones 2-5 and cyclohexen-2-one 7 (Table 1, entries 1, 4, 5, 8 and 10), affording the corresponding trans adducts (where applicable) as the only observable isomers. A catalyst loading of 10% was required for cycloaddition reactions involving cyclopenten-2-one 6 and ethyl crotonate 9 (Table 1, entries 9 and 12), which was not surprising as these substrates are considered less reactive dienophiles. Additional amounts of the diene were also necessary for the latter reaction to obtain the product 17 in good yields. The catalytic system was also compatible with the Diels-Alder transformation of crotonaldehyde 8, which resulted in the formation of the desired product in good yield and acceptable isomeric ratio (Table 1, entry 11). The use of less reactive dienes 18 and 19 (Table 1, entries 2, 3, 6, and 7) did not have any significant impact on the reaction outcomes involving dienophile 2. However, the less reactive dienophile 4 required higher catalyst loadings in order to obtain quantitative yields.

**Table 1.** LAI(OTf)<sub>2</sub>/Na[BAr<sup>CI</sup><sub>4</sub>]- and tBuCI/AgOTf (HBA)-catalyzed Diels–Alderreactions of dienophiles 2–9 with 2,3-dimethyl-1,3-butadiene (1), 2-methyl-1,3-butadiene (18), or cyclohexadiene (19). The results are listed as: time(h), yield (%), trans/cis or endo/exo, and para/meta ratios for each run.



Chem. Eur. J. 2015, 21, 11344 - 11348

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Additional experiments were carried out to show that the presence and subsequent catalytic activity of an HBA for the examined Diels-Alder transformations was highly unlikely. First of all, we generated the HBA HOTf, as described by Hintermann and co-workers, by treating tBuCl with 1 equivalent of AgOTf.<sup>[2e]</sup> By using the suggested 1 mol% of this HBA system with respect to the dienophile, we attempted the target cycloadditions. Even though the target transformations involving 2 and 3 were as fast and produced comparable yields, the cycloadditions using the other dienophiles (4-9) were dramatically different (longer reaction times, lower yields and/or isomeric ratios) with respect to our aluminum-based system (Table 1). For example, only the swift polymerization of the diene was observed when less reactive dienophiles 6, 7, and 9 were used. As anticipated, it was possible to completely quench the catalytic activity of this HBA system by introducing equimolar amounts of 2,6-di-tert-butylpyridine, 18 (Table 2, entries 1 and 2). In contrast, for the reactions between 1 and 2 or 5 in the presence of [LAI(OTf)<sub>2</sub>]/Na[BAr<sup>CI</sup><sub>4</sub>], the presence of **18** showed only subtle effects on the yields, presumably due to the possibility of a dienophile-to-enolate side reaction<sup>[16]</sup> and/or the presence of an extra reagent (18). It is also noteworthy that introducing 1 equivalent of 18 to a DCM solution containing our system did not produce any significant amounts of the corresponding pyridinium ion.<sup>[17]</sup> Therefore, all of the aforementioned evidence indicated that the target transformations were most likely catalyzed by our LA system rather than an HBA.

When considering the overall reaction conditions (catalyst loading, reaction times and temperatures), dienophile scope, and product yields and isomeric ratios, our catalytic system appears to be more robust and/or practical than the most established Diels–Alder catalysts.<sup>[7,18]</sup> For example, the silicon-based



cation  $[FcSi(Me)(tBu)]^+$  (**19**; Fc=ferrocenyl), as reported by Oestreich and co-workers,<sup>[7c]</sup> is certainly a more active catalyst than our system. However, this catalyst must be operated below -30 °C in DCM to avoid deactivation and is reported to act as a proton source in the presence of 18. Base-induced decomposition was also reported for the trityl cation.<sup>[18]</sup> Furthermore, apart from 19, most Diels-Alder transformations for "difficult" dienophiles 4-7 are not demonstrated, including transformations using [SiEt<sub>3</sub>(toluene)]<sup>+, [7a,c, 18]</sup> Interestingly, although quantitative conversions were reported for cycloadditions involving 19 and dienophiles 1-7, the product yields were significantly lower than those reported herein.<sup>[7c]</sup> For instance, we isolated product 14 in excellent yield, whereas complete product decomposition was observed when 19 was used as the catalyst. One has to wonder whether the exceptional activity of 19 is also responsible for these results.

In summary, we have synthesized a  $\beta$ -diketiminate supported aluminum bistriflate compound that, when combined with Na[BAr<sup>Cl</sup><sub>4</sub>], formed a quite active LA system capable of performing Diels–Alder transformations. Several additional experiments have been conducted to demonstrate that the target cycloadditions were indeed catalyzed by the LA system and not by an HBA. This system also showed (i) indefinite stability, great activity and selectivity in DCM at room temperature, (ii) remarkable versatility with respect to the dienophile scope, (iii) high resistance to deactivation by base **18** and (iv) no tendency for product decomposition.



We thank the A\*STAR (Grant ID: 1220703062) for funding.

**Keywords:** aluminum · homogeneous catalysis · Diels–Alder reaction · Lewis acids · triflate

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Received: June 5, 2015 Published online on June 29, 2015