

### **Referee 1 (R1)**

Comments: The paper describes the synthesis and characterization of Sb(III) derivatives supported by a dihydroacridine NNN, uni-negative pincer ligand, which, according to the author, has not been used previously in main group chemistry. In addition to describing the dichloride complex 2, the authors also report a monocationic derivative (3) obtained by chloride anion abstraction. The Lewis acidity of 3 is confirmed by the isolation of the DMAP adduct. The paper is concluded by a study involving 3 as a catalyst for the cyanosilylation of ketones.

I found the work to be well done and of sufficient maturity for publication in Dalton Transactions. I do have a few comments that the authors should consider, and I also suggest that additional experiments or calculations be considered.

1. The colors of the compounds are interesting. Are the absorbance bands in the UV spectrum due to ligand-based transitions? If unsure, please consider TD-DFT calculations to assign these features.
2. Please provide information about the stability of these compounds. Is compound 2 water-stable?
3. How does compound 3 react with TMSCN? Does it remain as an Sb-Cl compound or does it convert into an Sb-CN derivative? See *Organometallics* 2003, 22, 6, 1275–1280 for the type of exchange reaction that this referee anticipates. By the way, this *Organometallics* paper also deals with the cyanosilylation of carbonyls.
4. The mechanistic analyses are sensible. Maybe cite other works in which the silicon center is responsible for carbonyl activation. The seminal work of Piers on the hydrosilylation of carbonyls comes to mind (*J. Am. Chem. Soc.* 1996, 118, 39, 9440–9441).
5. Are steric effects responsible for the poor Gutmann-Becket Lewis acidity measured?
6. There are no EAs but the spectra are quite clean. I think this is OK but the editor may differ.

### **Referee 2 (R2)**

Comments: This work reports the synthesis of one antimony(III) pincer cationic compound and its utilization in catalysis. In fact, the target compound has many literature analogues and also the cyanosilylation on a limited set of compounds seems to be quite routine among main group element compounds nowadays. Therefore, I am doubtful if this paper meets standards necessary for communications in Dalton Trans. I suggest to perform the revision based on point mentioned below and re-submit this work as a full paper.

1. The statement in the abstract - Our findings demonstrate the potential of bis(imino)dihydroacridanide ligands in stabilizing unusual p-block species. – is my opinion a bit exaggerated. 1) only one cationic compound is reported 2) it has quite usual structure among other pincer compounds. Please omit this. In this regard, I strongly suggest to extend this study at least to the bismuth analogues, as bismuth is regarded as non-toxic element and therefore more relevant to catalysis. I guess it is a next step for the authors.

2. Figure 5 should be assigned as a Scheme.

3. Regarding the catalysis, I miss the result of a blank experiment without the catalyst and importantly comparison with literature results on similar main group element cations.

4. I am confused by the Table 1. The yields are reported, but in some cases also the residual of starting 6 are present, but where is the rest? E.g. entries 2, 7, 8, 10: what happened to ca 25 percent of the material (any side-product, lost during work-up due to the evaporation of the starting material, other possibility)? In other cases, the sum of both values is far from 100% as well. I don't understand this approach and the whole situation should carefully clarified.

Looking to the work-up (ESI), the reaction mixtures were evaporated using vacuum. It means some volatile by-products may be lost. Then the mixtures were analysed in CDCl<sub>3</sub> using an external standard, but the content of the evaporated sample certainly do not correspond to the reaction mixture after 9h as according to the results some material is lost.

I think that either real isolated yields of the product should be presented or the whole reaction should be performed in such a way that the real reaction mixture is analysed. I.e. do the catalysis in deuterated solvent (toluene-D<sub>8</sub>, but I am not sure if all material will be dissolved) than you can easily compare integrals of starting material and product and probably identify by-products, thereby easily get the yields and conversion. The alternative is to evaporated the toluene after catalysis, but justify that none of reactants or products is lost during this process.

5. The authors ascribe different yields in catalysis to electronic nature of the substrate. It seems to be a logic statement, but looking to the table it is not so obvious. E.g. in entry 10 the yield is lower than in 9, but authors mention that electron withdrawing group lowers the yield!

The more careful discussion is needed. I also recommend to include a really electron-rich 1-[4-(Dimethylamino)phenyl]ethanone as a counterpart to nitro-compound in entry 3. Then compare the yields it should be very high in the case of the former. Both substrates should be also included to DFT study and have a look to the differences to support the whole conclusions about attack of carbonyl toward silyl group.

6. Several proton NMR spectra in ESI are hardly visible especially in aromatic region due to many overlapping integrals, the same is valid for aromatic carbon NMR spectra. Maybe a zooming these regions will be valuable. Please also make better phase correction for figure S20.

7. I do not understand the reference 21 and its meaning for the article.