Introduction

Group 13 nitrides of Al, Ga, and In are materials with promising applications in new microelectronic and optoelectronic devices.[1,2] Thin films of these materials can be deposited using chemical vapor deposition techniques starting from organometallic precursors (MOCVD). Modern MOCVD of group 13 nitrides are based on an early investigation of Wiberg.[3] He found that the thermolysis of ammonia trimethylalane, $Me_3Al\text{-}NH_3$ (**1**), led to a loss of methane according to eq. 1.

$$\underset{\mathbf{1}}{Me_3Al\text{-}NH_3} \xrightarrow{-\,CH_4} \underset{\mathbf{2}}{(Me_2AlNH_2)_x} \xrightarrow{-\,CH_4} \underset{\mathbf{3}}{(MeAlNH)_y} \xrightarrow{-\,CH_4} \underset{\mathbf{4}}{AlN} \qquad (1)$$

Until recently, only the structure of the end product AlN (**4**) was known. Fifty years after Wiberg investigated the reaction sequence eq. 1 we were able to solve the molecular structure of the starting compound $Me_3Al\text{-}NH_3$ (**1**) by the X-ray powder diffraction method.[4] In the case of $Me_3Al\text{-}NH_3$ (**1**), it was impossible to grow single crystals for a X-ray structural determination, only the powder method led to a success.

Results and Discussion

Crystalline $Me_3Al\text{-}NH_3$ (**1**) was melted around 60°C in inert atmosphere using Schlenk-flask technique. Methane evolved from the clear liquid and close to the end of this reaction the melt suddenly crystallized to give a micro-crystalline powder of compound **2**. We aimed at a structural analysis of the aminoalane **2**, which is the first intermediate of Wiberg's reaction sequence.

Using the technique of simulated annealing and consecutive Rietveld refinement it was possible to solve the structure of the aminoalane **2** from high-resolution synchrotron powder diffraction data (figure 1). Compound **2** crystallizes in the monoclinic space group C2/c (a = 15.0047, b = 8.7500, c = 24.4702, β = 107.2900) with 8 molecular units in the cell. The molecular unit is a trimer of the formula $(Me_2AlNH_2)_3$. This trimer exhibits a boat conformation, in contrast to the known the known trimer $(Me_2AlNH_2)_3$, which exhibits a twist conformation. The latter result was deduced from a single-crystal X-ray determination.[5] Figure 2 compares the two trimeric aminodimethylalanes, the one from the known single-crystal analysis[5] and the one of the current investigation. These results will be published in full detail shortly.

References

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(4) Müller, J.; Ruschewitz, U.; Indris, O.; Hartwig, H.; Stahl, W. *J. Am. Chem. Soc.* **1999**, *121*, 4647–4652.
(5) Interrante, L.V.; Sigel, G.A.; Garbauskas, M; Hejna, C.; Slack, G.A. *Inorg. Chem.* **1989**, *28*, 252-257.

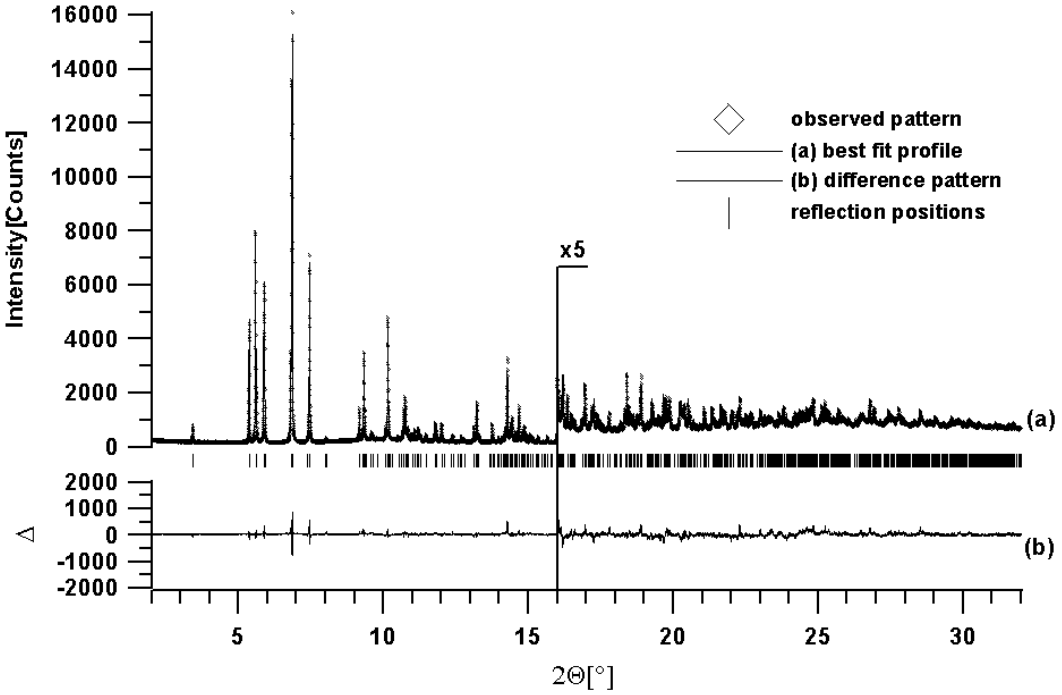
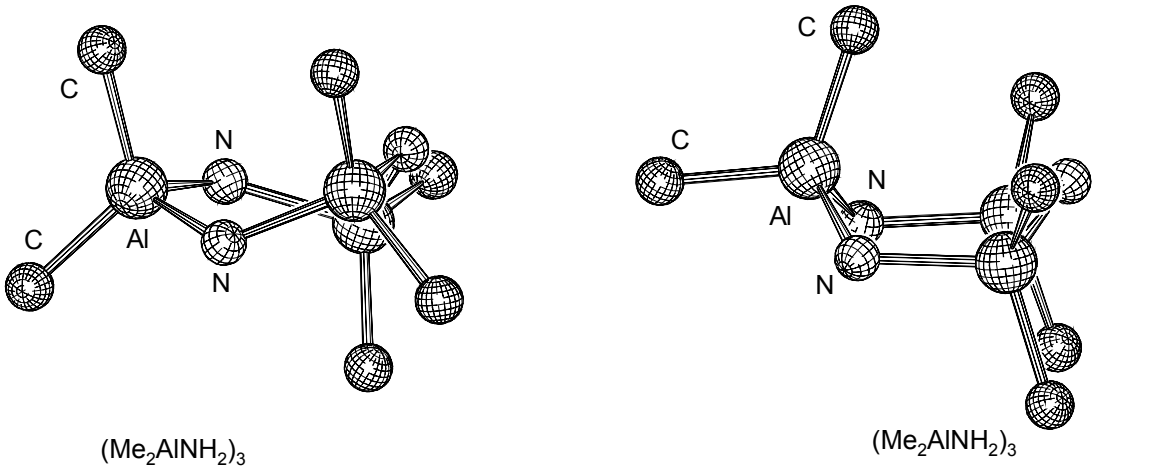


Figure 1. High-resolution X-ray synchrotron powder diffraction of compound **2**



$(Me_2AlNH_2)_3$
twist conformation (see reference 5)

$(Me_2AlNH_2)_3$
boat conformation (current investigation)

Figure 2. Comparison of the known trimer (left) and the so far unknown trimer (right)