

Dalton Transactions

Synthesis and temperature-dependent NMR studies of monomeric and dimeric tris(dialkylamino)alanes

Erik Schumann, Uwe Böhme, Erica Brendler and Florian Mertens

Electronic Supplementary Information

DOI: 0.0000/xxxxxxxxxx

Crystal structures

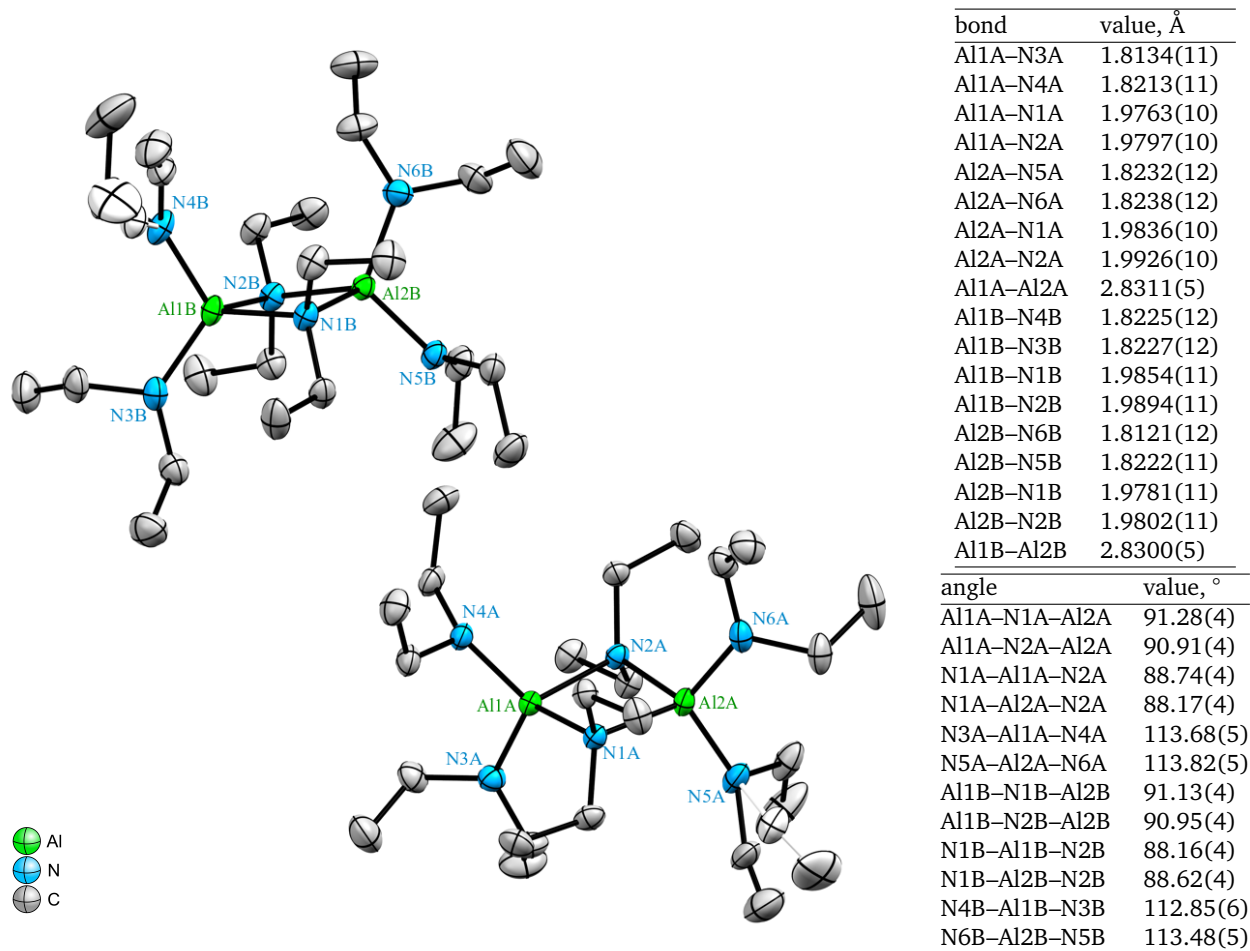


Fig. ESI.1 Asymmetric unit of tris(diethylamino)alane (left); selected bond lengths and angles (right).

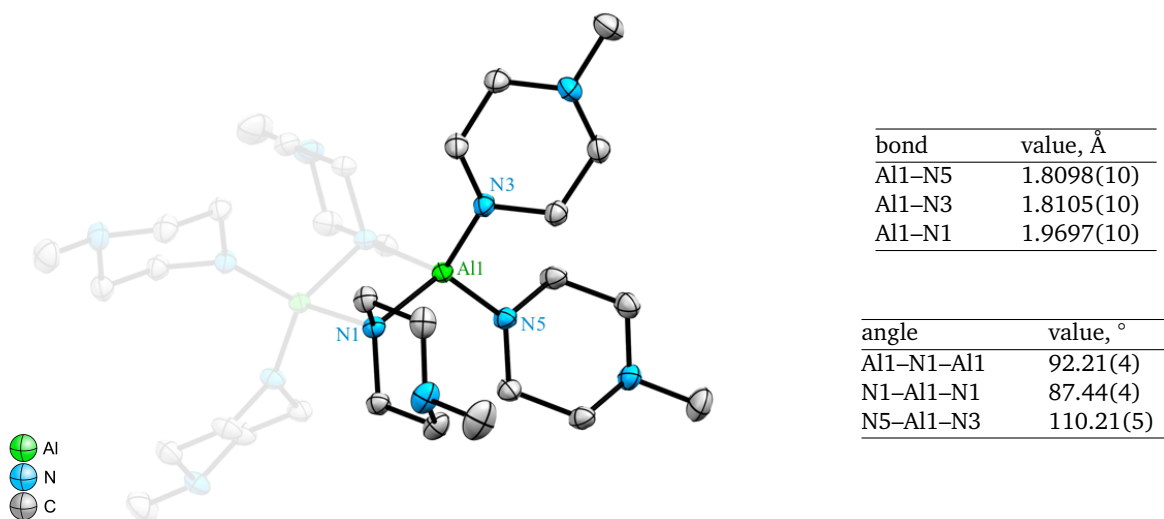


Fig. ESI.2 Asymmetric unit of tris(*N*-methylpiperazino)alane (left); selected bond lengths and angles (right).

Synthetic procedures

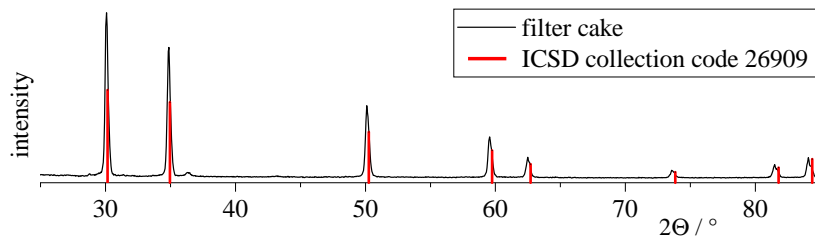


Fig. ESI.3 Example of a powder diffractogram of the separated lithium chloride.

Aluminum titration

$\text{AlH}_3 \cdot \frac{1}{5}\text{Et}_2\text{O}_{(s)}$ is carefully deactivated by diffusion of water vapor. The substance is then transferred to a 100 mL beaker containing 20 mL of distilled water and 10 mL of hydrochloric acid (2 molL^{-1}). The suspension is heated to boiling and the resulting clear solution is transferred to a 100 mL volumetric flask. To 25 mL of the solution, 30 mL of an EDTA solution (0.025 molL^{-1}) and 1 mL of hydrochloric acid (2 molL^{-1}) are added. The solution is boiled for 10 min and then cooled to room temperature. A pH value of 7 is adjusted with CH_3COONa . By adding a spatula tip of a xylenol orange/ KNO_3 trituration (1:100), the solution takes on an intense yellow color. Using a ZnSO_4 solution (0.2 molL^{-1}), the yellow solution is titrated until an orange color is apparent.

Temperature-dependent NMR spectra

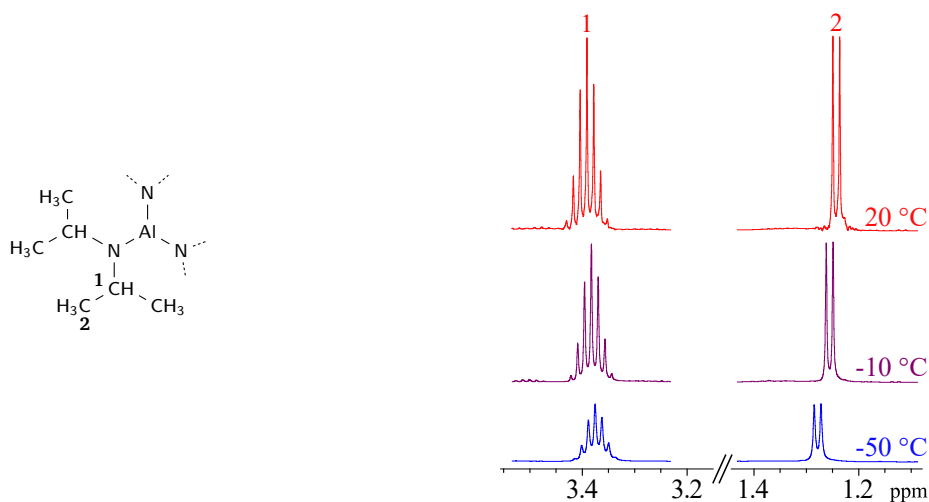


Fig. ESI.4 Temperature-dependent ^1H NMR spectra of tris(diisopropylamino)alane.

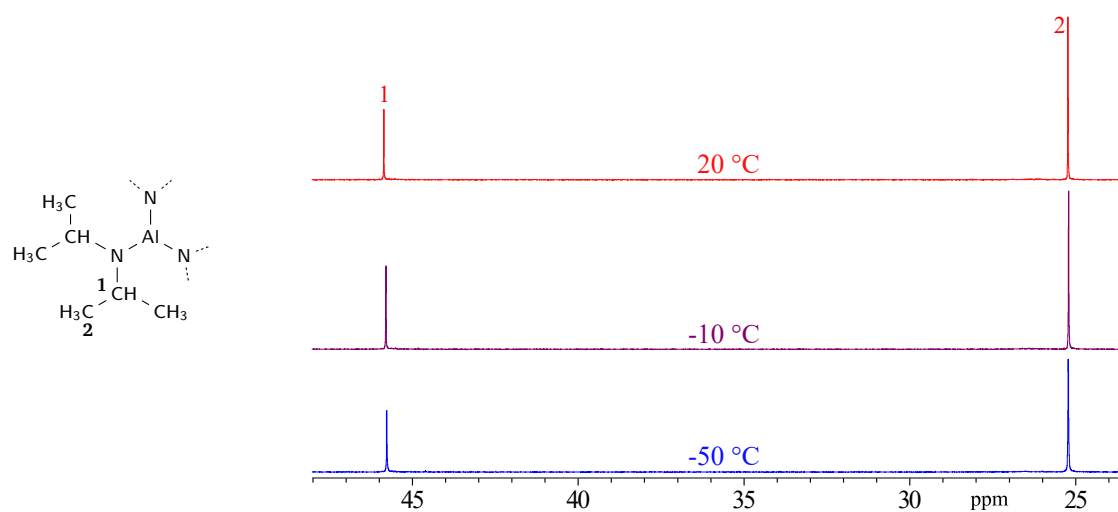


Fig. ESI.5 Temperature-dependent ^{13}C NMR spectra of tris(diisopropylamino)alane.

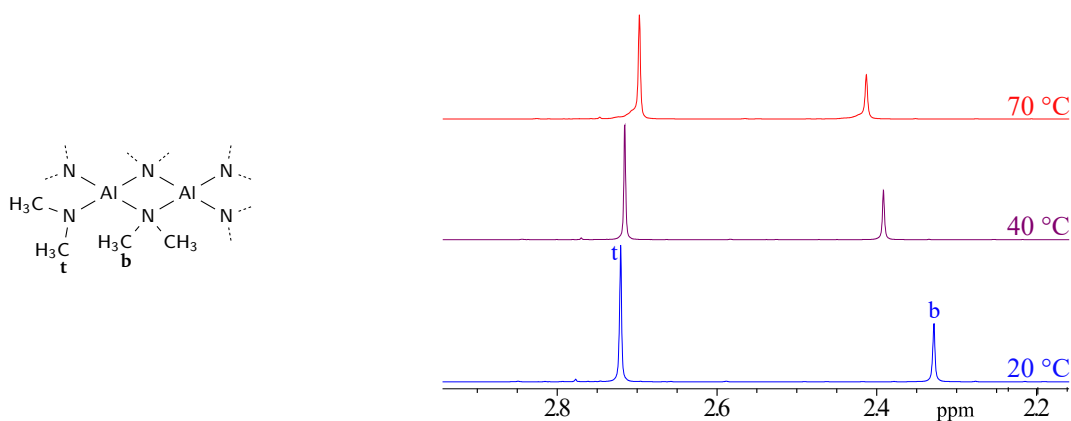


Fig. ESI.6 Temperature-dependent ^1H NMR spectra of tris(dimethylamino)alane.

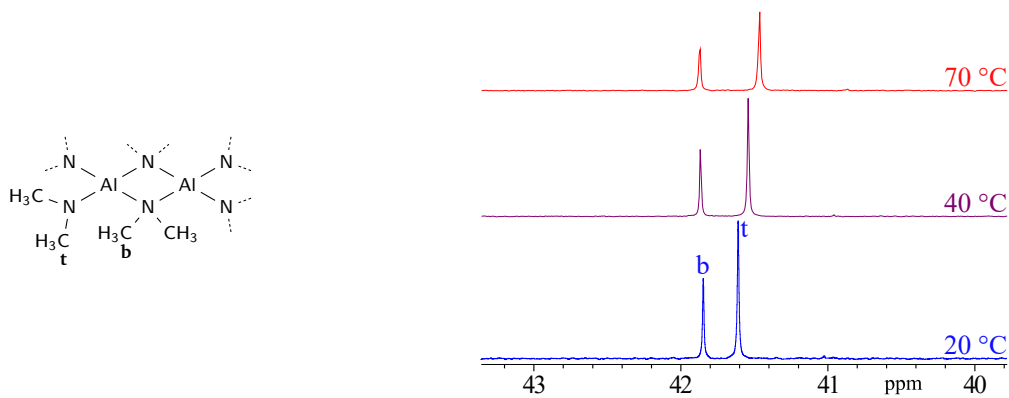


Fig. ESI.7 Temperature-dependent ^{13}C NMR spectra of tris(dimethylamino)alane.

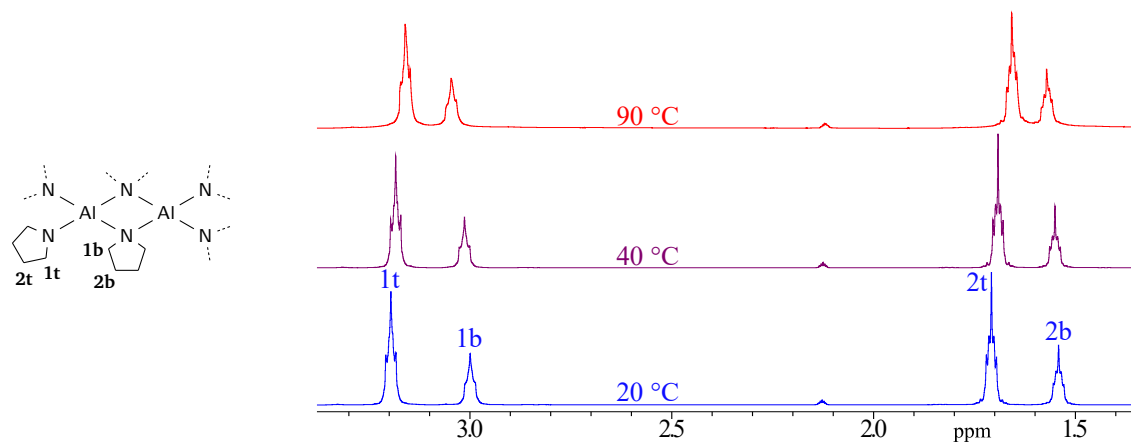


Fig. ESI.8 Temperature-dependent ^1H NMR spectra of tris(pyrrolidino)alane.

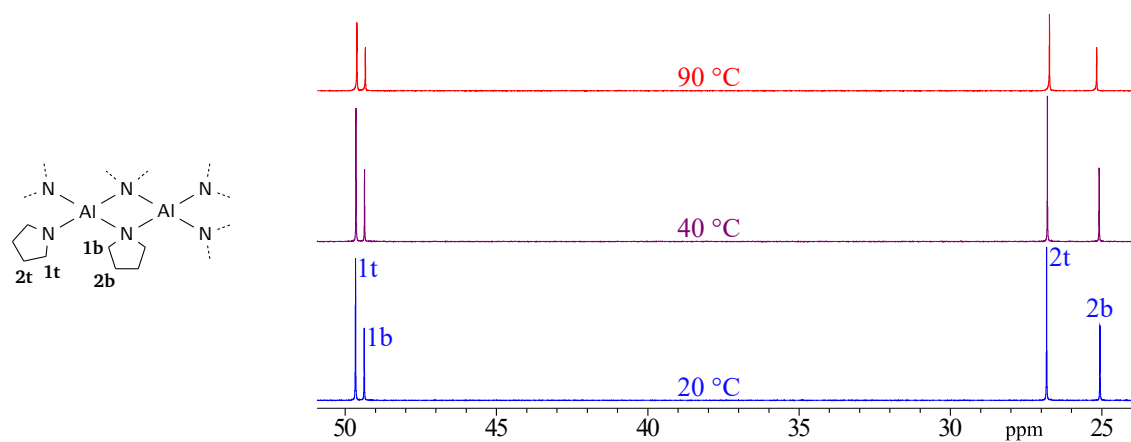


Fig. ESI.9 Temperature-dependent ^{13}C NMR spectra of tris(pyrrolidino)alane.

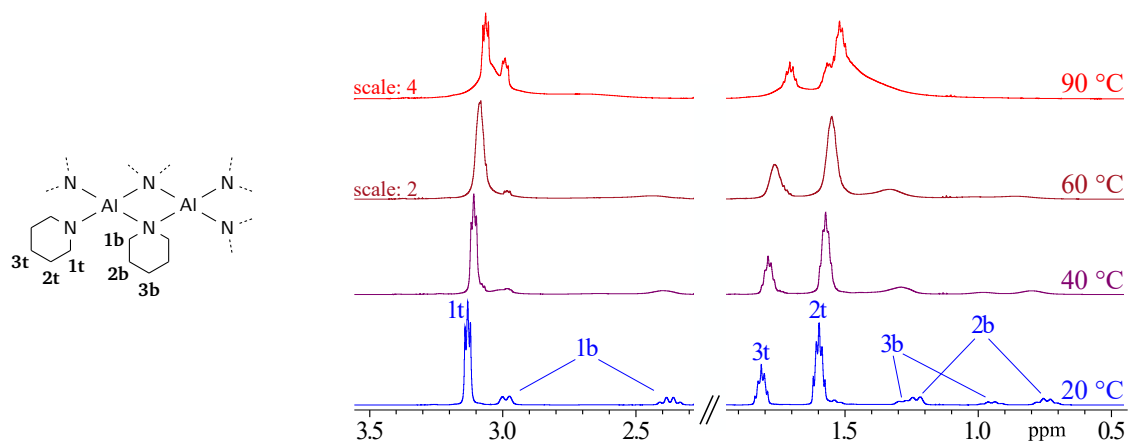


Fig. ESI.10 Temperature-dependent ^1H NMR spectra of tris(piperidino)alane.

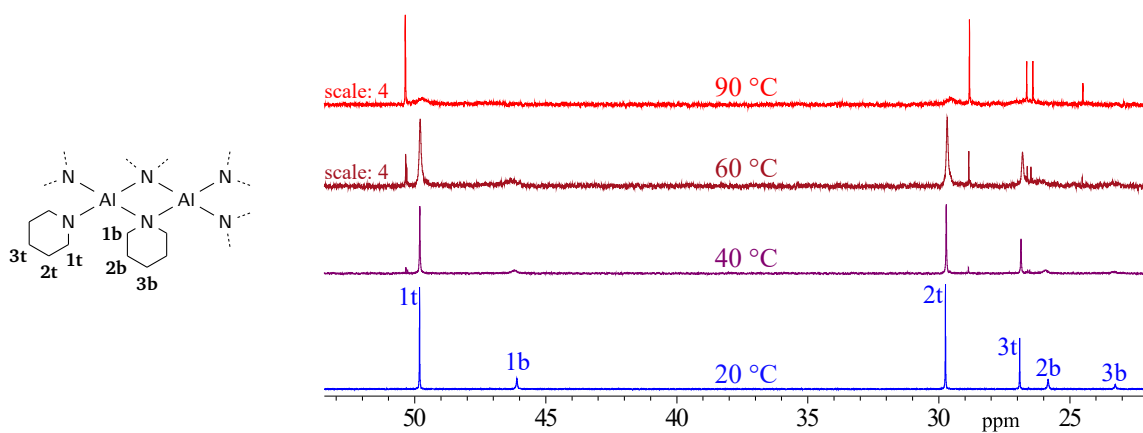


Fig. ESI.11 Temperature-dependent ¹³C NMR spectra of tris(piperidino)alane.