Aluminium Bond Configuration in $AlCl_3 \cdot NH_3$: an Electron Diffraction Study

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Summary The aluminium bond configuration in the co-ordination molecule $AlCl_3 \cdot NH_3$ has been determined by gas-phase electron diffraction.

THE gas-phase electron diffraction experiments and structure analysis in this work were carried out following the procedure normally used in the Budapest laboratory. Nozzle temperatures of *ca.* 250 °C were used and intensity data in the interval $2{\cdot}50 \leqslant s \leqslant 30{\cdot}50$ Å^{-1} were utilized.

It was assumed that the $AlCl_3 \cdot NH_3$ molecule has C_{3v} symmetry, with a staggered conformation. The information provided by the experimental radial distribution (see Figure) was straightforward as far as the aluminium bond configuration is concerned, with the exception of the Al–N

bond distance. The most important individual interatomic distances and their relative weights are shown in the Figure.

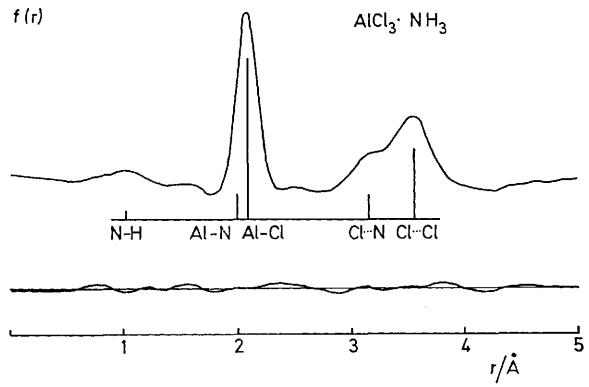

FIGURE. Experimental radial distribution and difference from the theoretical curve for the best model. The most important individual interatomic distances and their relative weights are shown.

Least-squares refinement based on molecular intensities yielded the values of r(Al–N) = 1·996 ± 0·019, r(Al–Cl) = 2·100 ± 0·005 Å, and ∠ Cl–Al–Cl = $116{\cdot}3_5$ ± 0·4°. Less reliable information was obtained for the nitrogen bond configuration.

The Al–N bond length indicates that a strong linkage exists. This is further supported by mass spectrometric evidence.[1] The value obtained is similar to the Al–N bond length in crystalline $AlCl_3 \cdot NMe_3$, 1·96 ± 0·01 Å.[2] As expected with ligand electronegativity differences, a significantly longer Al–N bond was found in $Me_3Al \cdot NMe_3$, 2·099 ± 0·010 Å, by gas-phase electron diffraction.[3]

The configuration of the $AlCl_3$ part of the compound is intermediate between those of monomeric $AlCl_3$ and the $AlCl_4^-$ ion. Gas-phase electron diffraction data[4] gave a value of r(Al–Cl) = 2·06 ± 0·01 Å for $AlCl_3$ and indicated a nearly planar configuration (∠ Cl–Al–Cl 118 ± 1·5°). From crystalline molecular structure data for $AlCl_4^-$, *e.g.* in $AlSeCl_7$, the average Al-Cl distance is shown to be 2·13 Å with a nearly regular tetrahedral arrangement.[5]

Added in proof: In August 1973, Dr. Arne Haaland of Oslo, Norway, informed us of the results of a gas-phase electron diffraction study of $AlCl_3 \cdot NMe_3$. The geometric parameters found for the molecules in the gas phase are in agreement with those of the crystal.[2]

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