Crystal and Molecular Structure of Hepta-μ_3-methylimido-heptakis-(methylaluminium). A New Aluminium–Nitrogen Cage

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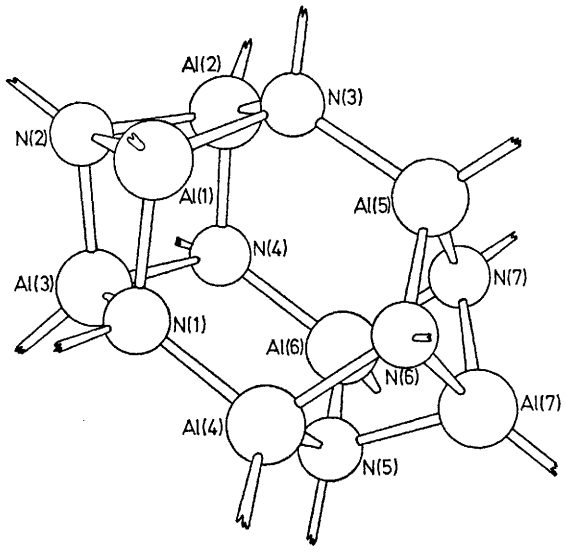
Summary The molecule $(MeAlNMe)_7$ has a cage structure; each aluminium and nitrogen atom is four-co-ordinate, and is bound to one methyl group and three atoms within the cage.

THERMAL decomposition of the adduct Me_3Al,NH_2Me in toluene at 215 °C yields a solution from which white crystals $(MeAlNMe)_x$ may be isolated.[1] Cryoscopic measurements in benzene appeared to indicate that the species in solution were octameric; an *X*-ray structural analysis, however, now shows that the molecules are heptameric.

Crystals of the compound $(MeAlNMe)_7$ were sealed in Pyrex capillaries and found to be monoclinic with $a = 14{\cdot}059(7)$, $b = 14{\cdot}407(8)$, $c = 14{\cdot}435(8)$ Å, $\beta = 93{\cdot}10(5)°$, space group $P2_1/c$, $Z = 4$[$(MeAlNMe)_7$ units] $D_c = 1{\cdot}13$ g cm^{-3}. Intensity data for 928 independent reflections having $F_o \geqslant 6{\cdot}0\ \sigma(F_o)$ and $1° \leqslant 2\theta \leqslant 42°$ were obtained on a Hilger and Watts Y290 four-circle diffractometer with Mo-K_α radiation. The structure was solved using a symbolic addition procedure, and refinement of the positional parameters of all nonhydrogen atoms with isotropic Debye factors has converged to $R = 0{\cdot}123$.

The structure consists of well separated molecules $(MeAlNMe)_7$ with approximately C_{3v} symmetry (Figure). The C_3 axis is along a line through Al(7) and N(2). All methyl groups point outwards from the cage. The aluminium–nitrogen framework comprises three six-membered $(AlN)_3$ rings, Al(1)N(3)Al(5)N(6)Al(4)N(1), Al(2)N(4)Al(6)-N(7)Al(5)N(3), and Al(3)N(1)Al(4)N(5)Al(6)N(4) with a boat conformation and six four-membered $(AlN)_2$ rings Al(4)N(6)Al(7)N(5), Al(5)N(7)Al(7)N(6), Al(6)N(5)Al(7)N(7), Al(1)N(2)Al(2)N(3), Al(2)N(2)Al(3)N(4), and Al(3)N(2)-Al(1)N(1). The six-membered rings with mean Al–N bond length 1·96 Å and angles Al–N–Al, 120·0° and N–Al–N, 110·7° are similar to those in the molecules $(Me_2AlNHMe)_3$[2] and the four-membered rings, with Al–N 1·90 Å, and angles Al–N–Al, 88·8°; N–Al–N, 89·5° are like those in the molecules $(PhAlNPh)_4$[3] and $(Me_2AlNMe_2)_2$.[2] The n.m.r. spectrum[1] of



FIGURE

$(MeAlNMe)_7$ is now readily explained; it is clear that both this compound and the ethyl analogue $(EtAlNMe)_7$[4] are heptameric in solution and that the previously reported molecular weights were inaccurate.

Examples of organometallic compounds $(R^1XYR^2)_x$ are known for values of x from 1 to 5[5] and 8[6] but the molecule $(MeAlNMe)_7$ is the first heptameric species to be characterised by X-ray methods. It is possible that the compounds $EtMgOPr^n$, Pr^iMgOMe, and Pr^iMgOEt, which are apparently heptameric or octameric in solution,[7] form similar isoelectronic cages.

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