THE MOLECULAR STRUCTURE OF [$AlH_2Cl(Quinuclidine)_2$]

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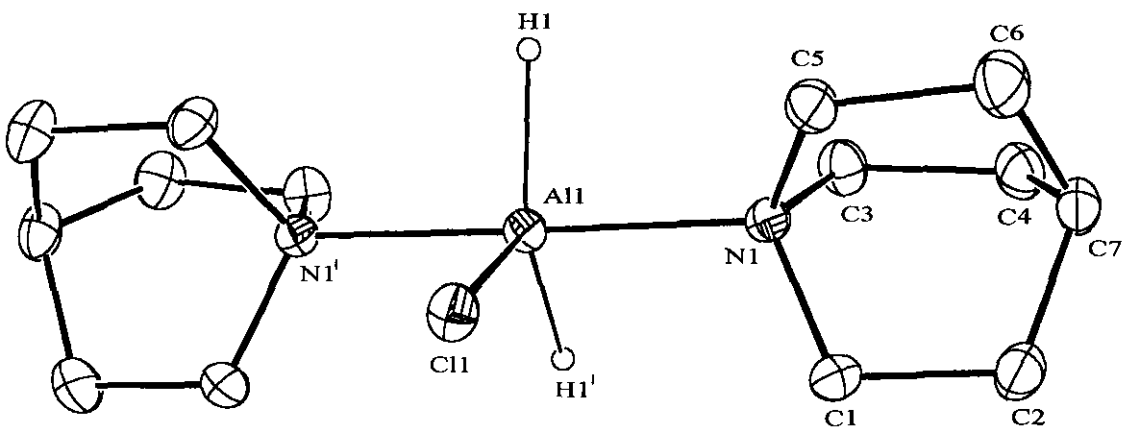

Figure 1. Molecular structure of [$AlH_2Cl(Quinuclidine)_2$] . Selected bond lengths (Å) and angles(°): Cl(1)-Al(1) 2.2145(13), Al(1)-N(1) 2.1354(17), Al(1)-H(1) 1.63(2), N(1)-Al(1)-N(1)' 175.90(10), N(1)-Al(1)-Cl(1) 92.05(5), N(1)-Al(1)-H(1) 88.9(8), N(1)'-Al(1)-H(1) 89.3(8), H(1)-Al(1)-Cl(1) 116.2(7), H(1)-Al(1)-H(1)' 127.7(9).

Comment

The hydride ligand H(1) was located from difference maps and refined isotropically. The Al(1)-H(1) distance is in the normal region for such interactions [1]. The compound is isomorphous to its gallane counterpart, [$GaH_2Cl(Quinuclidine)_2$] [2].

Experimental

Preparation of [$AlH_2Cl(Quinuclidine)_2$] :

Solid quinuclidine hydrochloride (1.55g, 10.5 mmol) was added to a stirred slurry of $LiAlH_4$ (0.2g, 5.3 mmol) in diethyl ether (40 cm^3) sustained at a temperature of -50°C. The resultant colourless slurry was then warmed to room temperature over 5hrs, by which time gas evolution had ceased. Volatiles were removed *in vacuo* to yield an oily white solid that was subsequently recrystallised from diethyl ether. Placement at -30°C overnight gave the title compound as colourless prisms. Yield 1.19g, 79%; mp 182°C (dec). ^1H NMR (400 MHz, C_6D_6, 300K): δ 1.12 (12H, m, CH_2), 1.24 (2H, m, CH), 2.64 (12H, t, ^3JHH 9.2 Hz, NCH_2), 4.22 (2H, br s, Al-H). ^{13}C NMR (100.6 MHz, C_6D_6, 300K): δ 19.8 (s, CH), 27.6 (s, CH_2), 56.3 (s, NCH_2). MS EI: m/z (%) 176 [{M-Quin}$^+$, 100], 251 [{M-Cl}$^+$, 64], 111 [Quin$^+$, 42]. IR (Nujol)ν/cm-1: 1691 cm^{-1} (s, br, Al-H).

Crystallography:

Table 1. Crystal data for $[AlH_2Cl(Quinuclidine)_2]$

Formula	$C_{14}H_{28}AlClN_2$	Formula weight	286.81
Crystal system	orthorhombic	Crystal size, mm	0.15x0.15x0.15
Space Group	*Pbcn*	*a*, Å	10.6225(10)
b, Å	12.1485(11)	*c*, Å	12.4366(11)
V, Å^3	1604.9(3)	*Z*	4
Diffractometer	Bruker AXS 1000 CCD	Temperature, K	223(2)
μ(Mo-K_α), mm^{-1}	0.28	D_{calcd}, g cm^{-3}	1.187
F(000)	624	θ_{max},^{o}	23.28
Reflns meas.	6794	Reflns unique	1156
Reflns with $I > 2\sigma(I)$	903	$R(F^2)$, $R_w(F^2)$ (all data)	0.050, 0.121
ρ, e Å^{-3}	0.28	G.O.F	1.062
No. obs/No. para	1156/88		
Programs used	SHELX-97 [3], Ortep-3 [4]		
Deposition number	CCDC 159330		

Acknowledgements
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References

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