THE MOLECULAR STRUCTURE OF [AlCl($C_5H_3MeNSiMe_3$)$_2$], ($C_5H_3MeNHSiMe_3$ = 6-METHYL-2-(TRIMETHYLSILYLAMINO)PYRIDINE)

Cameron Jones,[1] Peter C. Junk*[2] Stuart G. Leary[2] and Neil A. Smithies[1,2]

[1] Department of Chemistry, University of Wales, Cardiff, P.O. Box 912, Park Place, Cardiff CF10 3TB, UK
[2] Department of Chemistry, James Cook University, Townsville, Qld, 4811, Australia <peter.junk@jcu.edu.au>

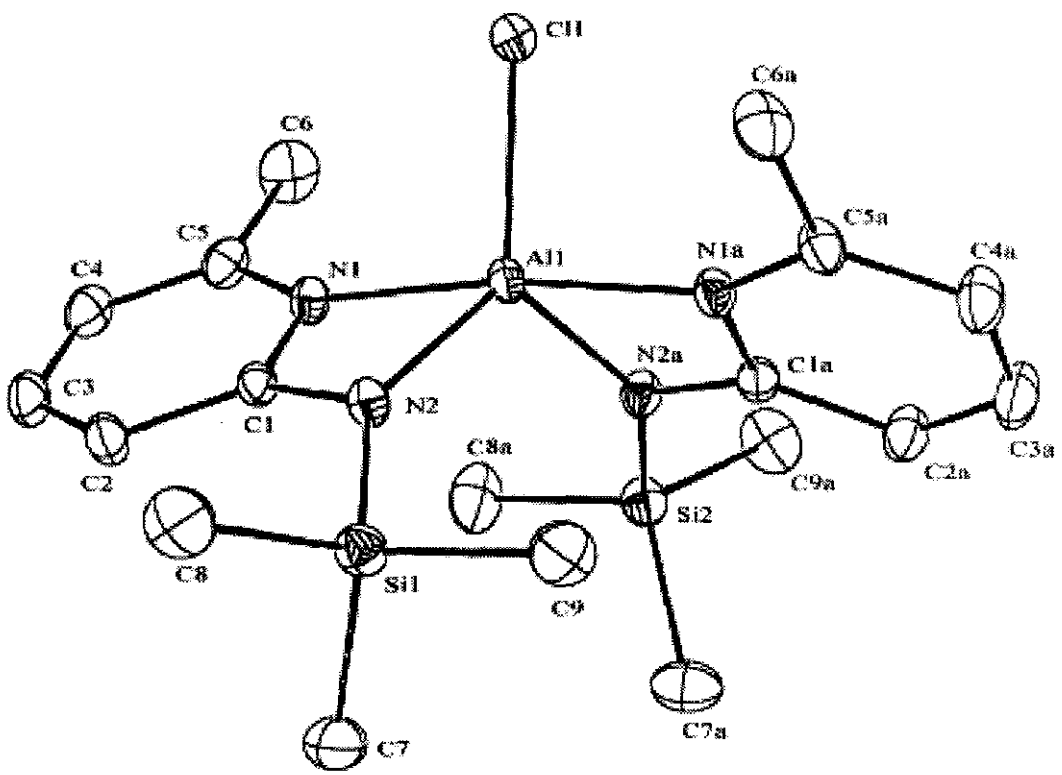

Figure 1. Molecular structure of [AlCl($C_5H_3MeNSiMe_3$)$_2$]. Selected bond lengths (Å) and angles(°): Al1-Cl1 2.1865(10), Al1-N1 2.008(2), Al1-N2 1.899(2), Al1-N1a 1.988(2), Al1-N2a 1.906(2); N1-Al1-Cl1 93.26(7), N1a-Al1-Cl1 94.47(7), N2-Al1-Cl1 124.13(8), N2a-Al1-Cl1 120.70(7), N1-Al1-N1a 171.31(9), N1-Al1-N2 69.18(9), N1-Al1-N2a 109.86(10), N1a-Al1-N2a 69.32(10), N2-Al1-N1a 103.06(9), N2-Al1-N2a 115.13(9).

Comment

The five-coordinate aluminium centre is distorted trigonal bipyramidal with the two aromatic nitrogens in axial positions and the two amido nitrogens and chloride atoms in the equatorial positions. The overall structure is similar to the related [AlCl(8-amidoquinoline)$_2$],[1] except that in the present compound, the chelating ligand has a much smaller bite angle, forming a four-membered chelate ring rather than a five-membered ring.

Experimental

Preparation of [AlCl($C_5H_3MeNSiMe_3$)$_2$] :

nBuLi (4.76 cm^3, 2.5 M, 11.9 mmol) was added in a dropwise fashion to a solution of 6-methyl-2-(trimethylsilylamino)pyridine (2.15 g, 11.9 mmol) in diethylether (30 cm^3) at 0°C. After stirring for 15 minutes anhydrous $AlCl_3$ (0.80 g, 6.0 mmol) was added forming a white precipitate. Filtration afforded a yellow solution that was reduced in volume in vacuo to the point of crystallisation and placed at -35° C overnight, yielding colourless crystals. Yield 1.51 g, 60%, mp 89-90°C. ^{1}H-NMR (300MHz, C_6D_6) δ 0.086

(s, 18H, SiCH_3), 2.35 (s, 6H, CH_3), 5.90 - 6.90 (m, 6H, H_{arom}) ppm. ^{13}C-NMR (75.46 MHz, C_6D_6) δ 1.95 (SiCH_3), 22.78 (CH_3), 108.59, 112.43, 142.66, 154.79, 167.49 (aromatic *C*) ppm. IR(nujol, NaCl plates, ν): 1599, 1565, 1248, 1166, 1013, 963, 843, 781, 740, 688, 635, 580 cm^{-1}.

Crystallography:

Table 1. Crystal data for of [AlCl($C_5H_3MeNSiMe_3$)$_2$]

Formula	$C_{18}H_{30}AlClN_4Si_2$	Formula weight	421.07
Crystal system	monoclinic	Crystal size, mm	0.48 x 0.38 x 0.36
Space Group	$P2_1/n$	*a*, Å	10.341(2)
b, Å	16.197(2)	*c*, Å	14.231(2)
β, °	92.570(10)	*V*, Å^3	2381.2(6)
Z	4	Diffractometer	Enraf-Nonius CAD4
Temperature, K	296(2)	μ(Mo-K$_\alpha$), mm^{-1}	0.308
D_{calcd}, g cm^{-3}	1.175	*F*(000)	896
θ_{max},°	24.98	Reflns meas.	4422
Reflns unique	4178	Reflns with $I > 2\sigma(I)$	2420
$R(F^2)$ [$I > 2\sigma(I)$]	0.035	$R_w(F^2)$ (all data)	0.110
ρ, e Å^{-3}	0.21	G.O.F	1.00
No. parameters	243		
Programs used	SHELX-97 [2], XSEED [3]		
Deposition number	CCDC 159567		
No absorption correction was applied			

Acknowledgements
We gratefully acknowledge financial support from the Australian Research Council.

References

[1] L. M. Engelhardt, M. G. Gardiner, C. Jones, P. C. Junk, C. L. Raston and A. H. White, *J. Chem. Soc., Dalton Trans*, (1996), 3053.
[2] G.M. Sheldrick, *SHELX-97*, University of Göttingen, Germany (1997).
[3] L. J. Barbour, 'XSeed, A Program of the Manipulation and Display of Crystallographic Models', University of Missouri - Columbia, (1999).

Received: March 16, 2001 - Accepted: March 21, 2001 – Accepted in publishable format: May 17, 2001