

Inorganic Chemistry

including bioinorganic chemistry

Inorg. Chem., 1998, 37(9), 2106-2109, DOI:[10.1021/ic971442n](https://doi.org/10.1021/ic971442n)

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1998 American Chemical Society

Table 1. Crystal data for Mes*Al(H)N(SiMe₃)₂.

Identification code	RW83B
Empirical formula	C ₂₄ H ₄₈ AlNSi ₂
Formula weight	433.79
Crystal size	0.44 x 0.40 x 0.40 mm
Crystal habit	block
Crystal color	colorless
Crystal system	Triclinic
Space group	P $\bar{1}$
Unit cell dimensions	a = 9.615(2) Å α = 70.890(12) ^o b = 11.684(2) Å β = 76.688(13) ^o c = 13.781(2) Å γ = 89.479(13) ^o
Volume	1419.8(4) Å ³
Z	2
Density (calculated)	1.015 Mg•m ⁻³
Absorption coefficient	1.482 mm ⁻¹
F(000)	480
Absorption correction ¹	XABS2
Max. and min. transmission	0.60 and 0.49

- 1) XABS2: an empirical absorption correction program. Parkin, S.; Moezzi, B.; Hope, H. *J. Appl. Cryst.* 1995, 28, 53-56.

Table 2. Data collection for $\text{Mes}^*\text{Al}(\text{H})\text{N}(\text{SiMe}_3)_2$.

Diffractometer	Siemens P4-RA
Temperature	130(2) K
Radiation source	rotating anode
Wavelength	1.54178 Å (CuK α)
Monochromator	nickel foil
θ range for data collection	3.50 to 56.07 $^\circ$
Scan type	2 θ - θ
Index ranges	$-10 \leq h \leq 10$, $-11 \leq k \leq 12$, $-14 \leq l \leq 14$
Reflections collected	7337
Independent reflections	3701 ($R_{\text{int}} = 0.0718$)
Standard reflections	2 (measured every 198 reflections)
Percent decay of standards	10.4

Experimental Details

The para t-butyl group C(11) - C(14A) displayed rotational disorder and was modelled using split occupancies of 0.572(8) for C(12) - C(14) and 0.428(8) for C(12A) - C(14A). In addition, a soft restraint for the C(11) C(12) to C(14A) distances of 1.54(2) Å was applied. H(1) was located in the difference map and refined freely with a fixed, isotropic thermal parameter.

Table 3. Solution and refinement of $\text{Mes*Al(H)N(SiMe}_3)_2$.

System for solution	XS-SHELXTL Ver. 5.03 (Sheldrick, 1994)
Structure solution	direct
System for refinement	XL-SHELXTL Ver. 5.03 (Sheldrick, 1994)
Refinement method	Full-matrix least-squares on F^2
Hydrogen atoms	riding model
Data / restraints / parameters	3701 / 6 / 302
Goodness-of-fit on F^2	1.068
Weighting scheme	$w^{-1} = \sigma^2(\text{Fo}^2) + (0.0521\text{P})^2 + 0.8681\text{P}$, where $\text{P} = (\text{Fo}^2 + 2\text{Fc}^2)/3$
R indices (all data)	$\text{R1} = 0.0518$, $\text{wR2} = 0.1333$
R indices calcd from obsd data	$\text{R1} = 0.0490$, $\text{wR2} = 0.1308$
Observed data ($>2\sigma(\text{I})$)	3429
Largest diff. peak and hole	0.295 and $-0.436 \text{ e}\text{\AA}^{-3}$

$$1) \text{ R1} = \frac{\sum ||\text{Fo}-\text{Fc}||}{\sum |\text{Fo}|}$$

$$\text{wR2} = \left[\frac{\sum [w(\text{Fo}^2 - \text{Fc}^2)^2]}{\sum [w(\text{Fo}^2)^2]} \right]^{1/2}$$

$$2) \text{ Goodness-of-Fit} = \left[\frac{\sum [w(\text{Fo}^2 - \text{Fc}^2)^2]}{(M-N)} \right]^{1/2}$$

where M is the number of reflections

and N is the number of parameters refined.

- 3) Refinement is based on F^2 for ALL reflections except for those with very negative F^2 or flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2 , conventional R-factors R are based on F, with F set to zero for negative F^2 . The observed criterion of $F^2 > 2\sigma(F^2)$ is used only for calculating R indices for observed data and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Table 4. Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement parameters [$\text{\AA}^2 \times 10^3$]

for $\text{Mes*Al(H)N(SiMe}_3)_2$.

$U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Si(1)	3663(1)	4438(1)	3332(1)	31(1)
Si(2)	1027(1)	4786(1)	2423(1)	39(1)
Al(1)	2830(1)	7018(1)	2091(1)	28(1)
N(1)	2530(2)	5374(2)	2656(2)	29(1)
C(1)	4431(2)	8042(2)	2121(2)	24(1)
C(2)	5730(2)	8284(2)	1327(2)	24(1)
C(3)	6860(2)	8995(2)	1366(2)	26(1)
C(4)	6782(2)	9498(2)	2157(2)	25(1)
C(5)	5497(2)	9290(2)	2913(2)	25(1)
C(6)	4333(2)	8576(2)	2918(2)	24(1)
C(7)	6013(3)	7825(2)	374(2)	30(1)
C(8)	4743(3)	7084(3)	349(2)	45(1)
C(9)	6344(3)	8910(2)	-662(2)	35(1)
C(10)	7301(3)	7029(3)	397(2)	45(1)
C(11)	8039(3)	10281(2)	2182(2)	31(1)
C(12)	9397(5)	10394(7)	1325(5)	50(2)
C(13)	8425(7)	9826(6)	3262(5)	43(2)
C(14)	7557(6)	11609(4)	2015(6)	46(2)
C(12A)	9359(8)	9501(11)	2061(10)	68(3)
C(13A)	7852(10)	10457(10)	3227(6)	49(2)
C(14A)	8251(9)	11432(7)	1270(7)	58(3)
C(15)	2989(2)	8448(2)	3824(2)	28(1)
C(16)	1774(3)	7610(3)	3829(2)	39(1)
C(17)	2406(3)	9694(2)	3732(2)	33(1)
C(18)	3372(3)	7913(3)	4908(2)	39(1)
C(19)	5086(3)	5345(3)	3537(2)	39(1)
C(20)	2693(3)	3417(3)	4669(2)	52(1)
C(21)	4649(4)	3487(3)	2580(3)	60(1)
C(22)	1038(4)	5372(3)	983(3)	57(1)
C(23)	891(4)	3090(3)	2809(3)	60(1)
C(24)	-631(3)	5214(4)	3187(3)	68(1)

Table 5. Bond lengths [Å] for Mes*Al(H)N(SiMe₃)₂.

Si (1) -N (1)	1.740 (2)	Si (1) -C (19)	1.864 (3)
Si (1) -C (20)	1.865 (3)	Si (1) -C (21)	1.868 (3)
Si (2) -N (1)	1.744 (2)	Si (2) -C (24)	1.860 (3)
Si (2) -C (23)	1.873 (3)	Si (2) -C (22)	1.872 (3)
Al (1) -H (1)	1.51 (3)	Al (1) -N (1)	1.819 (2)
Al (1) -C (1)	1.974 (2)	C (1) -C (6)	1.416 (3)
C (1) -C (2)	1.419 (3)	C (2) -C (3)	1.395 (3)
C (2) -C (7)	1.542 (3)	C (3) -C (4)	1.386 (3)
C (4) -C (5)	1.384 (3)	C (4) -C (11)	1.535 (3)
C (5) -C (6)	1.399 (3)	C (6) -C (15)	1.546 (3)
C (7) -C (8)	1.516 (4)	C (7) -C (9)	1.537 (4)
C (7) -C (10)	1.542 (4)	C (11) -C (14A)	1.486 (7)
C (11) -C (13A)	1.491 (7)	C (11) -C (12)	1.519 (5)
C (11) -C (13)	1.539 (6)	C (11) -C (12A)	1.565 (8)
C (11) -C (14)	1.574 (5)	C (15) -C (16)	1.528 (3)
C (15) -C (17)	1.533 (4)	C (15) -C (18)	1.547 (3)

Table 6. Bond angles [$^{\circ}$] for $\text{Mes*Al(H)N(SiMe}_3)_2$.

N(1)-Si(1)-C(19)	111.30(11)	N(1)-Si(1)-C(20)	112.47(12)
C(19)-Si(1)-C(20)	106.62(13)	N(1)-Si(1)-C(21)	112.36(13)
C(19)-Si(1)-C(21)	105.11(14)	C(20)-Si(1)-C(21)	108.5(2)
N(1)-Si(2)-C(24)	109.85(13)	N(1)-Si(2)-C(23)	113.99(13)
C(24)-Si(2)-C(23)	107.0(2)	N(1)-Si(2)-C(22)	111.15(13)
C(24)-Si(2)-C(22)	108.9(2)	C(23)-Si(2)-C(22)	105.7(2)
H(1)-Al(1)-N(1)	114.5(10)	H(1)-Al(1)-C(1)	118.0(10)
N(1)-Al(1)-C(1)	127.54(10)	Si(1)-N(1)-Si(2)	121.82(12)
Si(1)-N(1)-Al(1)	123.87(11)	Si(2)-N(1)-Al(1)	114.27(12)
C(6)-C(1)-C(2)	117.2(2)	C(6)-C(1)-Al(1)	122.0(2)
C(2)-C(1)-Al(1)	120.8(2)	C(3)-C(2)-C(1)	120.2(2)
C(3)-C(2)-C(7)	115.2(2)	C(1)-C(2)-C(7)	124.6(2)
C(4)-C(3)-C(2)	122.8(2)	C(3)-C(4)-C(5)	116.8(2)
C(3)-C(4)-C(11)	121.9(2)	C(5)-C(4)-C(11)	121.3(2)
C(4)-C(5)-C(6)	122.8(2)	C(5)-C(6)-C(1)	120.2(2)
C(5)-C(6)-C(15)	115.4(2)	C(1)-C(6)-C(15)	124.5(2)
C(8)-C(7)-C(9)	106.5(2)	C(8)-C(7)-C(2)	113.3(2)
C(9)-C(7)-C(2)	109.8(2)	C(8)-C(7)-C(10)	108.1(2)
C(9)-C(7)-C(10)	109.1(2)	C(2)-C(7)-C(10)	109.8(2)
C(14A)-C(11)-C(13A)	114.1(5)	C(14A)-C(11)-C(4)	108.9(3)
C(13A)-C(11)-C(4)	111.9(3)	C(12)-C(11)-C(4)	115.4(3)
C(12)-C(11)-C(13)	108.2(4)	C(4)-C(11)-C(13)	111.5(3)
C(14A)-C(11)-C(12A)	110.6(6)	C(13A)-C(11)-C(12A)	106.5(6)
C(4)-C(11)-C(12A)	104.4(4)	C(12)-C(11)-C(14)	106.1(4)
C(4)-C(11)-C(14)	108.0(2)	C(13)-C(11)-C(14)	107.2(4)
C(16)-C(15)-C(17)	107.5(2)	C(16)-C(15)-C(18)	106.9(2)
C(17)-C(15)-C(18)	108.8(2)	C(16)-C(15)-C(6)	113.4(2)
C(17)-C(15)-C(6)	110.0(2)	C(18)-C(15)-C(6)	110.1(2)

Table 7. Anisotropic displacement parameters [$\text{\AA}^2 \times 10^3$]for $\text{Mes*Al(H)N(SiMe}_3)_2$.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [(ha^*)^2 U_{11} + \dots + 2hka^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
Si(1)	34(1)	26(1)	29(1)	-5(1)	-5(1)	2(1)
Si(2)	34(1)	41(1)	45(1)	-18(1)	-11(1)	-5(1)
Al(1)	27(1)	28(1)	29(1)	-7(1)	-9(1)	2(1)
N(1)	28(1)	29(1)	30(1)	-8(1)	-6(1)	-2(1)
C(1)	26(1)	20(1)	22(1)	-2(1)	-7(1)	6(1)
C(2)	30(1)	20(1)	22(1)	-4(1)	-7(1)	7(1)
C(3)	24(1)	26(1)	26(1)	-6(1)	-3(1)	5(1)
C(4)	24(1)	24(1)	26(1)	-4(1)	-9(1)	7(1)
C(5)	29(1)	26(1)	24(1)	-9(1)	-11(1)	8(1)
C(6)	24(1)	23(1)	21(1)	-3(1)	-6(1)	7(1)
C(7)	36(1)	26(1)	23(1)	-6(1)	-2(1)	2(1)
C(8)	58(2)	47(2)	27(2)	-16(1)	1(1)	-13(1)
C(9)	45(2)	30(2)	23(1)	-4(1)	-2(1)	3(1)
C(10)	63(2)	40(2)	33(2)	-15(1)	-7(1)	19(1)
C(11)	26(1)	34(2)	33(2)	-9(1)	-10(1)	2(1)
C(12)	28(3)	64(5)	56(4)	-25(4)	2(3)	-15(3)
C(13)	34(3)	43(4)	54(4)	-12(3)	-23(3)	2(3)
C(14)	42(3)	29(3)	74(5)	-16(3)	-29(3)	2(2)
C(12A)	30(4)	93(8)	111(9)	-61(7)	-37(5)	16(4)
C(13A)	50(5)	53(6)	43(5)	-14(4)	-13(4)	-22(4)
C(14A)	61(5)	56(5)	48(5)	-3(4)	-19(5)	-28(4)
C(15)	26(1)	31(2)	22(1)	-6(1)	-3(1)	3(1)
C(16)	34(1)	42(2)	37(2)	-17(1)	4(1)	-2(1)
C(17)	30(1)	35(2)	32(2)	-11(1)	-3(1)	8(1)
C(18)	41(2)	44(2)	23(1)	-3(1)	-4(1)	10(1)
C(19)	38(2)	35(2)	40(2)	-1(1)	-16(1)	7(1)
C(20)	49(2)	50(2)	42(2)	2(2)	-8(2)	-4(2)
C(21)	68(2)	55(2)	61(2)	-27(2)	-16(2)	26(2)
C(22)	74(2)	50(2)	62(2)	-25(2)	-37(2)	4(2)
C(23)	67(2)	49(2)	66(2)	-18(2)	-22(2)	-16(2)
C(24)	33(2)	82(3)	92(3)	-40(2)	-3(2)	-10(2)

Table 8. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $\text{Mes*Al(H)N(SiMe}_3)_2$.

	x	y	z	U(eq)
H(1)	1695 (28)	7641 (25)	1553 (21)	42
H(3A)	7721 (2)	9141 (2)	826 (2)	32
H(5A)	5402 (2)	9647 (2)	3450 (2)	30
H(8A)	4528 (14)	6353 (9)	978 (8)	68
H(8B)	4973 (9)	6850 (16)	-288 (9)	68
H(8C)	3908 (7)	7571 (7)	341 (16)	68
H(9A)	5514 (7)	9403 (9)	-694 (6)	52
H(9B)	6552 (18)	8614 (2)	-1265 (2)	52
H(9C)	7177 (11)	9407 (9)	-689 (6)	52
H(10A)	7091 (9)	6329 (10)	1053 (7)	68
H(10B)	8153 (6)	7507 (6)	365 (16)	68
H(10C)	7479 (13)	6743 (15)	-211 (9)	68
H(12A)	9724 (24)	9584 (7)	1376 (20)	75
H(12B)	10144 (14)	10886 (31)	1419 (20)	75
H(12C)	9201 (12)	10785 (33)	629 (5)	75
H(13A)	8733 (38)	8998 (14)	3393 (14)	64
H(13B)	7584 (13)	9828 (32)	3820 (5)	64
H(13C)	9205 (28)	10362 (21)	3260 (11)	64
H(14A)	6693 (23)	11591 (6)	2562 (18)	69
H(14B)	7351 (38)	11946 (13)	1316 (13)	69
H(14C)	8326 (16)	12118 (9)	2062 (29)	69
H(12D)	9176 (29)	8725 (26)	2645 (28)	102
H(12E)	10213 (16)	9939 (27)	2076 (52)	102
H(12F)	9514 (42)	9349 (49)	1387 (24)	102
H(13D)	7631 (58)	9667 (10)	3793 (6)	73
H(13E)	7064 (42)	10978 (41)	3322 (20)	73
H(13F)	8739 (24)	10841 (48)	3253 (18)	73
H(14D)	8403 (63)	11246 (7)	611 (7)	86
H(14E)	9089 (38)	11915 (25)	1254 (28)	86
H(14F)	7399 (27)	11893 (26)	1344 (24)	86
H(16A)	1477 (14)	7933 (9)	3162 (7)	58
H(16B)	960 (8)	7562 (14)	4422 (9)	58
H(16C)	2108 (7)	6797 (5)	3906 (15)	58
H(17A)	2130 (17)	10030 (7)	3058 (6)	50
H(17B)	3148 (6)	10242 (5)	3762 (13)	50
H(17C)	1567 (11)	9608 (3)	4317 (8)	50
H(18A)	4111 (14)	8451 (9)	4954 (6)	58
H(18B)	3732 (19)	7109 (7)	4980 (6)	58
H(18C)	2515 (5)	7839 (16)	5476 (2)	58
H(19A)	5644 (13)	5881 (13)	2850 (2)	59
H(19B)	5720 (13)	4796 (3)	3904 (14)	59
H(19C)	4641 (3)	5836 (13)	3966 (13)	59
H(20A)	2024 (16)	3879 (5)	5020 (7)	78

H (20B)	3385 (4)	3085 (15)	5093 (6)	78
H (20C)	2161 (18)	2750 (11)	4597 (3)	78
H (21A)	5175 (20)	4013 (3)	1878 (7)	89
H (21B)	3963 (4)	2926 (15)	2503 (16)	89
H (21C)	5324 (18)	3023 (16)	2964 (9)	89
H (22A)	898 (25)	6243 (5)	770 (5)	85
H (22B)	261 (16)	4942 (15)	859 (4)	85
H (22C)	1959 (10)	5237 (19)	567 (3)	85
H (23A)	861 (25)	2728 (3)	3564 (5)	90
H (23B)	1727 (13)	2830 (3)	2395 (13)	90
H (23C)	16 (13)	2824 (3)	2670 (17)	90
H (24A)	-679 (14)	6097 (4)	2920 (14)	103
H (24B)	-616 (14)	4954 (21)	3936 (4)	103
H (24C)	-1470 (3)	4816 (19)	3107 (17)	103
