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DR0695D 1

$[Me_2AlN(Li)Ph(THF)_2]_2$

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## STRUCTURE DETERMINATION SUMMARY

### Crystal Data

| | |
|---|---|
| Empirical Formula | $C_{16}H_{27}AlLiNO_2$ |
| Color; Habit | YELLOW DIAMOND |
| Crystal size (mm) | 1.0 x 0.6 x 0.4 |
| Crystal System | Triclinic |
| Space Group | $P\bar{1}$ |
| Unit Cell Dimensions | $a = 9.957(5)$ Å |
| | $b = 10.074(5)$ Å |
| | $c = 10.579(5)$ Å |
| | $\alpha = 115.66(4)^\circ$ |
| | $\beta = 97.31(4)^\circ$ |
| | $\gamma = 103.50(4)^\circ$ |
| Volume | $897.9(8)$ Å$^3$ |
| Z | 2 |
| Formula weight | 299.3 |
| Density(calc.) | 1.107 Mg/m$^3$ |
| Absorption Coefficient | 0.115 mm$^{-1}$ |
| F(000) | 324 |

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## Data Collection

| | |
|---|---|
| Diffractometer Used | Siemens ~~R3m/V~~ P-4 |
| Radiation | MoKα (λ = 0.71073 Å) |
| Temperature (K) | 298 |
| Monochromator | Highly oriented graphite crystal |
| 2θ Range | 3.5 to 45.0° |
| Scan Type | 2θ-θ |
| Scan Speed | Variable; 12.00 to 60.00°/min. in ω |
| Scan Range (ω) | 0.74° plus Kα-separation |
| Background Measurement | Stationary crystal and stationary counter at beginning and end of scan, each for 0.5% of total scan time |
| Standard Reflections | 3 measured every 100 reflections |
| Index Ranges | $-4 \le h \le 10$, $-9 \le k \le 9$ $-11 \le l \le 11$ |
| Reflections Collected | 2397 |
| Independent Reflections | 2237 ($R_{int}$ = 2.45%) |
| Observed Reflections | 1483 ($F > 6.0\sigma(F)$) |
| Absorption Correction | N/A |

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Solution and Refinement

| | |
|---|---|
| System Used | Siemens SHELXTL PLUS (PC Version) |
| Solution | Direct Methods |
| Refinement Method | Full-Matrix Least-Squares |
| Quantity Minimized | $\sum w(F_o - F_c)^2$ |
| Absolute Structure | N/A |
| Extinction Correction | N/A |
| Hydrogen Atoms | Riding model, fixed isotropic U |
| Weighting Scheme | $w^{-1} = \sigma^2(F) + 99.0000F^2$ |
| Number of Parameters Refined | 187 |
| Final R Indices (obs. data) | R = 6.96 %, wR = 6.93 % |
| R Indices (all data) | R = 10.22 %, wR = 10.19 % |
| Goodness-of-Fit | 0.91 |
| Largest and Mean $\Delta/\sigma$ | 0.000, 0.000 |
| Data-to-Parameter Ratio | 7.9:1 |
| Largest Difference Peak | 0.36 $e\text{Å}^{-3}$ |
| Largest Difference Hole | -0.27 $e\text{Å}^{-3}$ |

Table 1. Atomic coordinates ($x10^5$) and equivalent isotropic displacement coefficients ($Å^2 x10^4$)

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Al(1) | 10589(16) | 49770(18) | 59309(18) | 430(8) |
| O(1) | -13853(40) | 4816(45) | 22368(46) | 647(23) |
| O(2) | 17982(40) | 11331(44) | 28750(47) | 645(24) |
| N(1) | 6409(42) | 44701(48) | 39533(45) | 437(22) |
| Li(1) | 3735(101) | 21835(115) | 32193(123) | 655(58) |
| C(1) | 14638(55) | 47227(58) | 30503(60) | 462(28) |
| C(2) | 8367(62) | 43587(69) | 16401(64) | 584(32) |
| C(3) | 16198(76) | 45728(74) | 7127(69) | 696(38) |
| C(4) | 30983(77) | 51542(74) | 11862(81) | 728(41) |
| C(5) | 37427(68) | 55240(75) | 25494(80) | 699(39) |
| C(6) | 29716(57) | 53143(66) | 34921(66) | 573(33) |
| C(7) | 8343(68) | 30446(69) | 60987(66) | 672(36) |
| C(8) | 29027(60) | 65250(71) | 72958(69) | 702(35) |
| C(9) | -20103(82) | -3027(92) | 7278(86) | 970(49) |
| C(10) | -35485(58) | -11677(69) | 5437(86) | 1220(58) |
| C(11) | -34696 | -14716 | 17564 | 1139(61) |
| C(12) | -23055(73) | -1317(89) | 29268(88) | 892(49) |
| C(13) | 14671(72) | -4576(74) | 25522(88) | 833(44) |
| C(14) | 27936(88) | -7137(104) | 28914(124) | 1309(77) |
| C(15) | 38453(87) | 7772(103) | 37777(122) | 1311(70) |
| C(16) | 33076(65) | 19025(79) | 35763(86) | 837(45) |

* Equivalent isotropic U defined as one third of the trace of the orthogonalized $U_{ij}$ tensor

Table 2. Bond lengths (Å)

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| | | | | | |
|---|---|---|---|---|---|
| Al(1)-N(1) | 1.888 | (5) | Al(1)-Li(1) | 2.868 | (9) |
| Al(1)-C(7) | 1.994 | (8) | Al(1)-C(8) | 1.981 | (5) |
| Al(1)-Al(1A) | 2.720 | (4) | Al(1)-N(1A) | 1.904 | (5) |
| O(1)-Li(1) | 1.918 | (9) | O(1)-C(9) | 1.409 | (9) |
| O(1)-C(12) | 1.432 | (11) | O(2)-Li(1) | 1.940 | (12) |
| O(2)-C(13) | 1.430 | (9) | O(2)-C(16) | 1.444 | (7) |
| N(1)-Li(1) | 2.023 | (12) | N(1)-C(1) | 1.400 | (8) |
| N(1)-Al(1A) | 1.904 | (5) | Li(1)-C(1) | 2.627 | (14) |
| Li(1)-C(7) | 2.722 | (15) | C(1)-C(2) | 1.390 | (9) |
| C(1)-C(6) | 1.411 | (7) | C(2)-H(2A) | 0.960 | |
| C(2)-C(3) | 1.384 | (11) | C(3)-H(3A) | 0.960 | |
| C(3)-C(4) | 1.385 | (10) | C(4)-H(4A) | 0.960 | |
| C(4)-C(5) | 1.348 | (12) | C(5)-H(5A) | 0.960 | |
| C(5)-C(6) | 1.389 | (11) | C(6)-H(6A) | 0.960 | |
| C(7)-H(7A) | 0.960 | | C(7)-H(7B) | 0.960 | |
| C(7)-H(7C) | 0.960 | | C(8)-H(8A) | 0.960 | |
| C(8)-H(8B) | 0.960 | | C(8)-H(8C) | 0.960 | |
| C(9)-H(9A) | 0.960 | | C(9)-H(9B) | 0.960 | |
| C(9)-C(10) | 1.516 | (10) | C(10)-H(10A) | 0.960 | |
| C(10)-H(10B) | 0.960 | | C(10)-C(11) | 1.439 | |
| C(11)-H(11A) | 0.960 | | C(11)-H(11B) | 0.960 | |
| C(11)-C(12) | 1.468 | | C(12)-H(12A) | 0.960 | |
| C(12)-H(12B) | 0.960 | | C(13)-H(13A) | 0.960 | |
| C(13)-H(13B) | 0.960 | | C(13)-C(14) | 1.435 | (13) |
| C(14)-H(14A) | 0.960 | | C(14)-H(14B) | 0.960 | |
| C(14)-C(15) | 1.424 | (10) | C(15)-H(15A) | 0.960 | |
| C(15)-H(15B) | 0.960 | | C(15)-C(16) | 1.442 | (15) |
| C(16)-H(16A) | 0.960 | | C(16)-H(16B) | 0.960 | |

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Table 3. Bond angles (°)

| | | | |
|---|---|---|---|
| N(1)-Al(1)-Li(1) | 44.7(3) | N(1)-Al(1)-C(7) | 109.8(2) |
| Li(1)-Al(1)-C(7) | 65.2(3) | N(1)-Al(1)-C(8) | 120.8(3) |
| Li(1)-Al(1)-C(8) | 132.5(3) | C(7)-Al(1)-C(8) | 107.3(3) |
| N(1)-Al(1)-Al(1A) | 44.4(2) | Li(1)-Al(1)-Al(1A) | 72.9(3) |
| C(7)-Al(1)-Al(1A) | 118.6(2) | C(8)-Al(1)-Al(1A) | 134.0(3) |
| N(1)-Al(1)-N(1A) | 88.3(2) | Li(1)-Al(1)-N(1A) | 106.5(2) |
| C(7)-Al(1)-N(1A) | 110.4(3) | C(8)-Al(1)-N(1A) | 119.1(2) |
| Al(1A)-Al(1)-N(1A) | 43.9(2) | Li(1)-O(1)-C(9) | 125.1(6) |
| Li(1)-O(1)-C(12) | 125.4(5) | C(9)-O(1)-C(12) | 109.4(5) |
| Li(1)-O(2)-C(13) | 121.3(5) | Li(1)-O(2)-C(16) | 123.7(4) |
| C(13)-O(2)-C(16) | 108.2(6) | Al(1)-N(1)-Li(1) | 94.3(5) |
| Al(1)-N(1)-C(1) | 134.0(3) | Li(1)-N(1)-C(1) | 98.6(5) |
| Al(1)-N(1)-Al(1A) | 91.7(2) | Li(1)-N(1)-Al(1A) | 115.5(3) |
| C(1)-N(1)-Al(1A) | 120.9(4) | Al(1)-Li(1)-O(1) | 128.9(5) |
| Al(1)-Li(1)-O(2) | 117.2(4) | O(1)-Li(1)-O(2) | 102.4(5) |
| Al(1)-Li(1)-N(1) | 41.0(2) | O(1)-Li(1)-N(1) | 127.3(6) |
| O(2)-Li(1)-N(1) | 128.6(5) | Al(1)-Li(1)-C(1) | 66.8(3) |
| O(1)-Li(1)-C(1) | 134.0(7) | O(2)-Li(1)-C(1) | 104.5(5) |
| N(1)-Li(1)-C(1) | 31.8(3) | Al(1)-Li(1)-C(7) | 41.7(2) |
| O(1)-Li(1)-C(7) | 107.0(6) | O(2)-Li(1)-C(7) | 95.9(5) |
| N(1)-Li(1)-C(7) | 82.6(4) | C(1)-Li(1)-C(7) | 106.6(3) |
| N(1)-C(1)-Li(1) | 49.6(4) | N(1)-C(1)-C(2) | 121.6(5) |
| Li(1)-C(1)-C(2) | 104.8(4) | N(1)-C(1)-C(6) | 122.5(6) |
| Li(1)-C(1)-C(6) | 114.2(5) | C(2)-C(1)-C(6) | 115.9(6) |
| C(1)-C(2)-H(2A) | 117.7(4) | C(1)-C(2)-C(3) | 123.1(5) |
| H(2A)-C(2)-C(3) | 119.2(4) | C(2)-C(3)-H(3A) | 120.0(4) |
| C(2)-C(3)-C(4) | 119.3(7) | H(3A)-C(3)-C(4) | 120.7(5) |
| C(3)-C(4)-H(4A) | 120.2(5) | C(3)-C(4)-C(5) | 119.2(8) |
| H(4A)-C(4)-C(5) | 120.6(5) | C(4)-C(5)-H(5A) | 118.9(5) |
| C(4)-C(5)-C(6) | 122.1(6) | H(5A)-C(5)-C(6) | 119.0(4) |
| C(1)-C(6)-C(5) | 120.4(6) | C(1)-C(6)-H(6A) | 119.2(4) |
| C(5)-C(6)-H(6A) | 120.4(4) | Al(1)-C(7)-Li(1) | 73.1(4) |
| Al(1)-C(7)-H(7A) | 109.7(2) | Li(1)-C(7)-H(7A) | 60.9(2) |
| Al(1)-C(7)-H(7B) | 109.4(2) | Li(1)-C(7)-H(7B) | 78.2(3) |
| H(7A)-C(7)-H(7B) | 109.5(1) | Al(1)-C(7)-H(7C) | 109.3(2) |
| Li(1)-C(7)-H(7C) | 169.9(2) | H(7A)-C(7)-H(7C) | 109.5(1) |
| H(7B)-C(7)-H(7C) | 109.5(1) | Al(1)-C(8)-H(8A) | 109.2(3) |
| Al(1)-C(8)-H(8B) | 108.8(2) | H(8A)-C(8)-H(8B) | 109.5(1) |
| Al(1)-C(8)-H(8C) | 110.5(2) | H(8A)-C(8)-H(8C) | 109.5(1) |
| H(8B)-C(8)-H(8C) | 109.5(1) | O(1)-C(9)-H(9A) | 109.8(4) |
| O(1)-C(9)-H(9B) | 110.8(5) | H(9A)-C(9)-H(9B) | 109.1(1) |
| O(1)-C(9)-C(10) | 104.9(7) | H(9A)-C(9)-C(10) | 110.4(4) |
| H(9B)-C(9)-C(10) | 111.9(5) | C(9)-C(10)-H(10A) | 111.5(4) |
| C(9)-C(10)-H(10B) | 110.2(5) | H(10A)-C(10)-H(10B) | 109.0(1) |
| C(9)-C(10)-C(11) | 103.6(5) | H(10A)-C(10)-C(11) | 111.7(1) |
| H(10B)-C(10)-C(11) | 110.7(1) | C(10)-C(11)-H(11A) | 110.7(1) |
| C(10)-C(11)-H(11B) | 110.1(1) | H(11A)-C(11)-H(11B) | 108.8 |
| C(10)-C(11)-C(12) | 104.7(6) | H(11A)-C(11)-C(12) | 112.1(4) |
| H(11B)-C(11)-C(12) | 110.4(5) | O(1)-C(12)-C(11) | 106.4(7) |
| O(1)-C(12)-H(12A) | 110.3(4) | C(11)-C(12)-H(12A) | 110.2(4) |
| O(1)-C(12)-H(12B) | 110.0(4) | C(11)-C(12)-H(12B) | 110.8(5) |
| H(12A)-C(12)-H(12B) | 109.1(1) | O(2)-C(13)-H(13A) | 109.7(4) |
| O(2)-C(13)-H(13B) | 110.3(4) | H(13A)-C(13)-H(13B) | 108.8(1) |
| O(2)-C(13)-C(14) | 107.4(6) | H(13A)-C(13)-C(14) | 111.3(5) |
| H(13B)-C(13)-C(14) | 109.4(7) | C(13)-C(14)-H(14A) | 109.3(6) |
| C(13)-C(14)-H(14B) | 111.4(7) | H(14A)-C(14)-H(14B) | 108.5(1) |

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| | | | |
|---|---|---|---|
| C(13)-C(14)-C(15) | 107.0(8) | H(14A)-C(14)-C(15) | 108.3(7) |
| H(14B)-C(14)-C(15) | 112.2(8) | C(14)-C(15)-H(15A) | 108.3(8) |
| C(14)-C(15)-H(15B) | 111.8(6) | H(15A)-C(15)-H(15B) | 108.7(1) |
| C(14)-C(15)-C(16) | 107.7(8) | H(15A)-C(15)-C(16) | 109.8(6) |
| H(15B)-C(15)-C(16) | 110.6(5) | O(2)-C(16)-C(15) | 106.4(6) |
| O(2)-C(16)-H(16A) | 109.6(3) | C(15)-C(16)-H(16A) | 111.5(5) |
| O(2)-C(16)-H(16B) | 110.2(5) | C(15)-C(16)-H(16B) | 110.4(6) |
| H(16A)-C(16)-H(16B) | 108.8(1) | | |

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Table 4. Anisotropic displacement coefficients ($Å^2 x 10^4$)

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| Al(1) | 370(9) | 427(10) | 543(10) | 106(7) | 58(7) | 302(8) |
| O(1) | 510(24) | 587(26) | 752(31) | 6(20) | 86(22) | 348(24) |
| O(2) | 533(26) | 547(27) | 992(33) | 168(21) | 210(23) | 486(25) |
| N(1) | 364(24) | 513(28) | 508(27) | 130(21) | 78(21) | 322(23) |
| Li(1) | 465(58) | 554(63) | 983(82) | 179(49) | 150(54) | 404(61) |
| C(1) | 455(33) | 380(32) | 606(38) | 105(26) | 122(28) | 305(29) |
| C(2) | 481(35) | 707(43) | 569(37) | 139(31) | 152(30) | 332(34) |
| C(3) | 860(52) | 752(47) | 581(40) | 276(40) | 223(38) | 392(37) |
| C(4) | 810(52) | 677(46) | 853(53) | 224(39) | 530(44) | 425(41) |
| C(5) | 534(39) | 746(47) | 899(53) | 162(34) | 300(39) | 456(42) |
| C(6) | 433(34) | 665(41) | 714(40) | 103(29) | 136(31) | 452(35) |
| C(7) | 824(46) | 729(44) | 682(42) | 308(37) | 195(36) | 499(37) |
| C(8) | 494(36) | 674(43) | 773(45) | 124(32) | -51(32) | 295(37) |
| C(9) | 976(61) | 916(58) | 933(61) | 236(48) | 197(49) | 419(50) |
| C(10) | 786(58) | 830(60) | 1464(86) | 39(46) | -468(55) | 368(58) |
| C(11) | 725(57) | 935(64) | 1588(90) | 122(49) | 345(59) | 522(67) |
| C(12) | 675(47) | 922(56) | 1254(67) | 223(43) | 237(47) | 693(53) |
| C(13) | 769(49) | 589(46) | 1283(64) | 205(38) | 260(44) | 581(45) |
| C(14) | 919(65) | 1026(74) | 2448(123 | 595(60) | 548(73) | 1063(82) |
| C(15) | 683(56) | 1059(72) | 2146(110 | 319(54) | 0(61) | 809(75) |
| C(16) | 549(44) | 715(49) | 1281(65) | 120(38) | 181(42) | 566(47) |

The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

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Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

| | x | y | z | U |
|---|---|---|---|---|
| H(2A) | -186 | 3912 | 1296 | 80 |
| H(3A) | 1137 | 4323 | -246 | 80 |
| H(4A) | 3655 | 5300 | 552 | 80 |
| H(5A) | 4767 | 5943 | 2878 | 80 |
| H(6A) | 3458 | 5578 | 4455 | 80 |
| H(7A) | -44 | 2259 | 5438 | 80 |
| H(7B) | 1625 | 2678 | 5868 | 80 |
| H(7C) | 816 | 3270 | 7075 | 80 |
| H(8A) | 3040 | 7470 | 7237 | 80 |
| H(8B) | 2872 | 6728 | 8264 | 80 |
| H(8C) | 3680 | 6137 | 7058 | 80 |
| H(9A) | -1530 | -1023 | 241 | 80 |
| H(9B) | -1948 | 421 | 358 | 80 |
| H(10A) | -3925 | -2112 | -367 | 80 |
| H(10B) | -4135 | -522 | 595 | 80 |
| H(11A) | -4364 | -1575 | 2015 | 80 |
| H(11B) | -3246 | -2415 | 1515 | 80 |
| H(12A) | -2684 | 640 | 3530 | 80 |
| H(12B) | -1782 | -445 | 3514 | 80 |
| H(13A) | 968 | -1125 | 1546 | 80 |
| H(13B) | 871 | -663 | 3135 | 80 |
| H(14A) | 3066 | -1229 | 2011 | 80 |
| H(14B) | 2711 | -1351 | 3358 | 80 |
| H(15A) | 3977 | 999 | 4772 | 80 |
| H(15B) | 4746 | 811 | 3538 | 80 |
| H(16A) | 3442 | 2802 | 4487 | 80 |
| H(16B) | 3783 | 2229 | 2975 | 80 |

 



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