Inorganic Chemistry
including bioinorganic chemistry
Inorg. Chem., 1997, 36(1), 59-63, DOI:10.1021/ic960671r

**Table S1.** Crystallographic Data for $Et_4N[V_2O_3(L\text{-}alsal)_2].MeCN$

| | |
|---|---|
| chemical formula | $C_{30}H_{41}N_4O_9V_2$ |
| fw | 703.5 |
| crystal size, mm | 0.30 x 0.40 x 0.30 |
| crystal system | orthorhombic |
| space group(No.) | $P2_12_12_1(19)$ |
| a, Å | 9.797(5) |
| b, Å | 12.854(6) |
| c, Å | 27.550(11) |
| V, Å$^3$ | 3469(3) |
| Z | 4 |
| T, $^oC$ | 22 |
| λ, Å | 0.71073 |
| $\rho_{obsd}$, g cm$^{-3}$ | 1.350 |
| $\rho_{calcd}$, g cm$^{-3}$ | 1.347 |
| μ(MoKα), cm$^{-1}$ | 5.93 |
| F(000) | 1468 |
| total no. of reflections | 3586 |
| no. of unique reflections | 3481 |
| no. of observed reflections (I>3σ(I)) | 1831 |
| no. of refined parameters | 306 |
| transm. coeff. | 0.6086-0.6293 |
| R,[a] % | 5.25 |
| Rw,[b] % | 5.56 |
| GOF[c] | 1.26 |
| maximum and mean Δ/σ | 0.098, 0.006 |
| data-to-parameter ratio | 6.0:1 |
| maximum, minimum difference peaks, eÅ$^{-3}$ | 0.41, -0.25 |

[a] $R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$. [b] $Rw = [\Sigma w(|F_o|-|F_c|)^2/\Sigma w|F_o|^2]^{1/2}$; $w^{-1} = \sigma^2(|F_o|)+g|F_o|^2$; g = 0.0001 . [c] The goodness of fit is defined as $[\Sigma w(|F_o|-|F_c|)^2/(n_o-n_v)]^{1/2}$ where $n_o$ and $n_v$ denote the number of data and variables, respectively.

**Table S2.** Atomic Coordinates ( x$10^4$) and Equivalent Isotropic*
Displacement Coefficients ($Å^2$x$10^3$) for $Et_4N[V_2O_3(L\text{-}alsal)_2]$ . MeCN

| | x | y | z | U(eq) |
|---|---|---|---|---|
| V(1) | 9216(2) | 2096(1) | 2109(1) | 49(1) |
| V(2) | 9597(2) | 1678(1) | 1021(1) | 55(1) |
| O(1) | 8520(8) | 1526(5) | 2558(3) | 73(3) |
| O(2) | 11139(6) | 2005(5) | 2313(2) | 53(2) |
| O(3) | 7740(8) | 2717(5) | 1775(3) | 92(3) |
| O(4) | 12916(8) | 2734(6) | 2658(3) | 82(3) |
| O(5) | 9452(8) | 1127(5) | 1661(2) | 67(3) |
| O(6) | 10684(9) | 1017(7) | 728(3) | 90(3) |
| O(7) | 7800(8) | 1095(5) | 861(2) | 66(3) |
| O(8) | 10501(8) | 2844(5) | 1325(2) | 69(3) |
| O(9) | 5999(8) | 1101(7) | 377(3) | 86(3) |
| N(1) | 9660(8) | 3615(5) | 2347(3) | 46(2) |
| N(2) | 8863(8) | 2754(7) | 524(3) | 52(3) |
| C(1) | 11733(12) | 2742(8) | 2537(4) | 53(3) |
| C(2) | 10828(11) | 3660(7) | 2678(3) | 50(2) |
| C(3) | 10406(13) | 3554(8) | 3199(4) | 71(3) |
| C(4) | 9175(10) | 4481(7) | 2178(3) | 48(2) |
| C(5) | 8086(10) | 4556(7) | 1839(3) | 49(2) |
| C(6) | 7686(12) | 5543(9) | 1675(4) | 72(3) |
| C(7) | 6677(14) | 5644(12) | 1329(5) | 90(4) |
| C(8) | 5998(14) | 4819(9) | 1156(4) | 84(4) |
| C(9) | 6367(13) | 3808(10) | 1312(4) | 79(4) |
| C(10) | 7440(11) | 3684(8) | 1648(4) | 57(3) |
| C(11) | 7105(13) | 1433(8) | 491(4) | 64(3) |
| C(12) | 7816(12) | 2283(8) | 212(3) | 63(3) |
| C(13) | 8481(13) | 1815(10) | -242(4) | 85(4) |
| C(14) | 9148(12) | 3702(9) | 488(4) | 65(3) |
| C(15) | 10131(11) | 4245(8) | 784(4) | 55(3) |
| C(16) | 10410(14) | 5285(9) | 656(4) | 84(4) |
| C(17) | 11348(15) | 5833(13) | 933(5) | 104(4) |
| C(18) | 12050(14) | 5387(11) | 1312(5) | 84(4) |
| C(19) | 11753(12) | 4359(9) | 1428(4) | 72(3) |
| C(20) | 10783(12) | 3792(8) | 1171(3) | 56(3) |
| N(3) | 4241(9) | -258(6) | 1655(3) | 52(3) |
| C(21) | 4621(17) | -511(10) | 1142(5) | 96(6) |
| C(22) | 3519(24) | -687(17) | 792(6) | 161(10) |
| C(23) | 3454(12) | -1158(10) | 1877(5) | 75(5) |
| C(24) | 4100(15) | -2210(10) | 1840(6) | 104(6) |
| C(25) | 5577(11) | -79(9) | 1932(5) | 77(5) |
| C(26) | 5410(16) | 186(11) | 2466(5) | 109(7) |
| C(27) | 3336(13) | 652(10) | 1680(5) | 83(5) |
| C(28) | 3926(18) | 1650(10) | 1490(6) | 119(7) |
| C(29) | 7875(26) | 8072(22) | 460(7) | 194(13) |
| C(30) | 9036(51) | 8529(37) | 167(19) | 317(30) |
| N(4) | 9927(26) | 8404(20) | -113(10) | 223(13) |

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

**Table S3.** Bond Lengths ( Å) for $Et_4N[V_2O_3(L\text{-}alsal)_2]$ . MeCN

| | | | |
|---|---|---|---|
| V(1)-V(2) | 3.067 (3) | V(1)-O(1) | 1.591 (7) |
| V(1)-O(2) | 1.969 (6) | V(1)-O(3) | 1.890 (9) |
| V(1)-O(5) | 1.768 (6) | V(1)-N(1) | 2.106 (7) |
| V(2)-O(5) | 1.905 (7) | V(2)-O(6) | 1.583 (9) |
| V(2)-O(7) | 1.964 (8) | V(2)-O(8) | 1.932 (7) |
| V(2)-N(2) | 2.075 (8) | O(2)-C(1) | 1.271 (12) |
| O(3)-C(10) | 1.325 (13) | O(4)-C(1) | 1.206 (14) |
| O(7)-C(11) | 1.300 (14) | O(8)-C(20) | 1.319 (12) |
| O(9)-C(11) | 1.206 (15) | N(1)-C(2) | 1.464 (12) |
| N(1)-C(4) | 1.297 (11) | N(2)-C(12) | 1.469 (13) |
| N(2)-C(14) | 1.253 (14) | C(1)-C(2) | 1.526 (14) |
| C(2)-C(3) | 1.500 (13) | C(4)-C(5) | 1.422 (14) |
| C(5)-C(6) | 1.402 (15) | C(5)-C(10) | 1.389 (14) |
| C(6)-C(7) | 1.379 (18) | C(7)-C(8) | 1.340 (19) |
| C(8)-C(9) | 1.415 (18) | C(9)-C(10) | 1.411 (16) |
| C(11)-C(12) | 1.506 (16) | C(12)-C(13) | 1.534 (16) |
| C(14)-C(15) | 1.441 (15) | C(15)-C(16) | 1.410 (15) |
| C(15)-C(20) | 1.374 (14) | C(16)-C(17) | 1.386 (20) |
| C(17)-C(18) | 1.377 (20) | C(18)-C(19) | 1.389 (18) |
| C(19)-C(20) | 1.390 (16) | N(3)-C(21) | 1.497 (16) |
| N(3)-C(23) | 1.520 (15) | N(3)-C(25) | 1.532 (14) |
| N(3)-C(27) | 1.470 (15) | C(21)-C(22) | 1.466 (25) |
| C(23)-C(24) | 1.496 (18) | C(25)-C(26) | 1.519 (19) |
| C(27)-C(28) | 1.500 (19) | C(29)-C(30) | 1.514 (56) |
| C(30)-N(4) | 1.174 (57) | | |

**Table S4.** Bond Angles (°) for $Et_4N[V_2O_3(L\text{-}alsal)_2]$ . MeCN

| | | | |
|---|---|---|---|
| V(2)-V(1)-O(1) | 137.0(3) | V(2)-V(1)-O(2) | 98.7(2) |
| O(1)-V(1)-O(2) | 99.3(3) | V(2)-V(1)-O(3) | 72.1(3) |
| O(1)-V(1)-O(3) | 104.2(4) | O(2)-V(1)-O(3) | 153.6(3) |
| V(2)-V(1)-O(5) | 34.8(2) | O(1)-V(1)-O(5) | 105.9(3) |
| O(2)-V(1)-O(5) | 91.9(3) | O(3)-V(1)-O(5) | 93.4(4) |
| V(2)-V(1)-N(1) | 116.2(2) | O(1)-V(1)-N(1) | 105.9(3) |
| O(2)-V(1)-N(1) | 76.6(3) | O(3)-V(1)-N(1) | 85.3(3) |
| O(5)-V(1)-N(1) | 147.5(3) | V(1)-V(2)-O(5) | 32.0(2) |
| V(1)-V(2)-O(6) | 132.4(3) | O(5)-V(2)-O(6) | 108.8(4) |
| V(1)-V(2)-O(7) | 100.2(2) | O(5)-V(2)-O(7) | 90.0(3) |
| O(6)-V(2)-O(7) | 106.5(4) | V(1)-V(2)-O(8) | 59.8(2) |
| O(5)-V(2)-O(8) | 85.5(3) | O(6)-V(2)-O(8) | 109.2(4) |
| O(7)-V(2)-O(8) | 143.6(3) | V(1)-V(2)-N(2) | 119.1(2) |
| O(5)-V(2)-N(2) | 146.4(3) | O(6)-V(2)-N(2) | 104.7(4) |
| O(7)-V(2)-N(2) | 78.2(3) | O(8)-V(2)-N(2) | 85.9(3) |
| V(1)-O(2)-C(1) | 122.2(6) | V(1)-O(3)-C(10) | 134.0(7) |
| V(1)-O(5)-V(2) | 113.2(3) | V(2)-O(7)-C(11) | 121.2(7) |
| V(2)-O(8)-C(20) | 132.4(6) | V(1)-N(1)-C(2) | 113.1(5) |
| V(1)-N(1)-C(4) | 127.5(6) | C(2)-N(1)-C(4) | 118.4(7) |
| V(2)-N(2)-C(12) | 110.7(6) | V(2)-N(2)-C(14) | 128.5(7) |
| C(12)-N(2)-C(14) | 120.7(9) | O(2)-C(1)-O(4) | 124.6(10) |
| O(2)-C(1)-C(2) | 115.7(9) | O(4)-C(1)-C(2) | 119.6(9) |
| N(1)-C(2)-C(1) | 105.4(7) | N(1)-C(2)-C(3) | 112.1(8) |
| C(1)-C(2)-C(3) | 109.5(8) | N(1)-C(4)-C(5) | 124.7(8) |
| C(4)-C(5)-C(6) | 118.9(9) | C(4)-C(5)-C(10) | 122.4(9) |
| C(6)-C(5)-C(10) | 118.7(9) | C(5)-C(6)-C(7) | 120.6(11) |
| C(6)-C(7)-C(8) | 121.8(13) | C(7)-C(8)-C(9) | 119.5(12) |
| C(8)-C(9)-C(10) | 119.5(11) | O(3)-C(10)-C(5) | 123.8(9) |
| O(3)-C(10)-C(9) | 116.4(10) | C(5)-C(10)-C(9) | 119.8(10) |
| O(7)-C(11)-O(9) | 123.7(10) | O(7)-C(11)-C(12) | 113.6(10) |
| O(9)-C(11)-C(12) | 122.7(10) | N(2)-C(12)-C(11) | 108.9(8) |
| N(2)-C(12)-C(13) | 110.0(9) | C(11)-C(12)-C(13) | 109.2(9) |
| N(2)-C(14)-C(15) | 125.2(10) | C(14)-C(15)-C(16) | 116.7(10) |
| C(14)-C(15)-C(20) | 123.0(9) | C(16)-C(15)-C(20) | 120.4(10) |
| C(15)-C(16)-C(17) | 118.2(12) | C(16)-C(17)-C(18) | 122.5(14) |
| C(17)-C(18)-C(19) | 117.7(12) | C(18)-C(19)-C(20) | 121.7(11) |
| O(8)-C(20)-C(15) | 122.9(9) | O(8)-C(20)-C(19) | 117.7(9) |
| C(15)-C(20)-C(19) | 119.4(10) | C(21)-N(3)-C(23) | 110.0(8) |
| C(21)-N(3)-C(25) | 106.9(9) | C(23)-N(3)-C(25) | 110.3(8) |
| C(21)-N(3)-C(27) | 111.6(9) | C(23)-N(3)-C(27) | 106.3(9) |
| C(25)-N(3)-C(27) | 111.8(8) | N(3)-C(21)-C(22) | 118.1(14) |
| N(3)-C(23)-C(24) | 116.4(10) | N(3)-C(25)-C(26) | 115.2(10) |
| N(3)-C(27)-C(28) | 115.5(11) | C(29)-C(30)-N(4) | 148.8(43) |

**Table S5.** Anisotropic Displacement Coefficients ($Å^2 \times 10^3$) for $Et_4N[V_2O_3(L\text{-}alsal)_2]$ . MeCN

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| V(1) | 53(1) | 37(1) | 57(1) | -4(1) | -8(1) | 5(1) |
| V(2) | 57(1) | 47(1) | 61(1) | 3(1) | -10(1) | -9(1) |
| O(1) | 78(5) | 61(5) | 78(5) | -9(4) | -2(4) | 13(4) |
| O(2) | 46(4) | 48(4) | 64(4) | 6(3) | -6(3) | -6(3) |
| O(3) | 78(6) | 38(4) | 160(8) | -11(4) | -59(5) | 15(5) |
| O(4) | 52(5) | 74(5) | 119(6) | -3(4) | -21(4) | -6(5) |
| O(5) | 92(6) | 38(3) | 70(5) | -8(4) | -30(5) | 1(3) |
| O(6) | 81(6) | 101(6) | 89(5) | 13(6) | -13(5) | -31(5) |
| O(7) | 77(5) | 60(4) | 60(4) | -22(4) | -17(4) | 3(4) |
| O(8) | 83(5) | 51(4) | 71(4) | -18(5) | -21(4) | 7(3) |
| O(9) | 63(5) | 118(7) | 77(5) | -32(5) | -19(4) | 11(5) |
| N(1) | 47(5) | 35(4) | 55(4) | 3(4) | -5(4) | -2(3) |
| N(2) | 53(5) | 60(5) | 43(4) | -10(4) | 1(4) | -2(4) |
| N(3) | 47(5) | 50(5) | 59(6) | 11(5) | 9(5) | 5(4) |
| C(21) | 114(12) | 85(8) | 88(10) | 22(9) | 33(10) | 17(8) |
| C(22) | 236(24) | 164(17) | 84(10) | 65(17) | -14(15) | -17(11) |
| C(23) | 53(7) | 88(9) | 84(8) | -26(7) | 4(6) | 3(7) |
| C(24) | 85(9) | 69(8) | 158(12) | -14(9) | -16(9) | 28(8) |
| C(25) | 40(7) | 62(6) | 129(11) | -3(6) | 8(7) | 10(7) |
| C(26) | 108(13) | 101(10) | 118(12) | -2(10) | -39(11) | -29(9) |
| C(27) | 69(9) | 98(10) | 82(9) | 21(8) | 7(7) | -12(8) |
| C(28) | 169(16) | 55(7) | 132(12) | 27(11) | 42(11) | 22(8) |
| C(29) | 217(25) | 244(26) | 121(15) | 55(26) | -5(17) | -63(17) |
| C(30) | 285(49) | 311(49) | 354(59) | -186(44) | -86(41) | 183(46) |
| N(4) | 225(24) | 184(17) | 260(24) | -12(18) | 136(20) | 81(16) |

The anisotropic displacement factor exponent takes the form:

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

**Table S6.** H-atom Coordinates ( $x10^4$) and Isotropic Displacement Coefficients ($Å^2x10^3$) for $Et_4N[V_2O_3(L\text{-}alsal)_2]$ . MeCN

| | x | y | z | U |
|---|---|---|---|---|
| H(2A) | 11307 | 4305 | 2634 | 80 |
| H(3A) | 9851 | 4129 | 3300 | 80 |
| H(3B) | 9896 | 2920 | 3229 | 80 |
| H(3C) | 11206 | 3517 | 3399 | 80 |
| H(4A) | 9568 | 5116 | 2297 | 80 |
| H(6A) | 8096 | 6148 | 1817 | 80 |
| H(7A) | 6438 | 6317 | 1204 | 80 |
| H(8A) | 5312 | 4881 | 909 | 80 |
| H(9A) | 5876 | 3204 | 1205 | 80 |
| H(12A) | 7173 | 2814 | 125 | 80 |
| H(13A) | 8934 | 2355 | -421 | 80 |
| H(13B) | 9132 | 1292 | -151 | 80 |
| H(13C) | 7784 | 1508 | -441 | 80 |
| H(14A) | 8659 | 4098 | 250 | 80 |
| H(16A) | 9952 | 5606 | 386 | 80 |
| H(17A) | 11514 | 6549 | 853 | 80 |
| H(18A) | 12713 | 5780 | 1492 | 80 |
| H(19A) | 12249 | 4016 | 1682 | 80 |
| H(21A) | 5202 | -1113 | 1141 | 80 |
| H(21B) | 5134 | 61 | 1011 | 80 |
| H(22A) | 3824 | -840 | 469 | 80 |
| H(22B) | 3013 | -1268 | 917 | 80 |
| H(22C) | 2944 | -82 | 786 | 80 |
| H(23A) | 2561 | -1162 | 1734 | 80 |
| H(23B) | 3350 | -1016 | 2217 | 80 |
| H(24A) | 3533 | -2726 | 1991 | 80 |
| H(24B) | 4188 | -2362 | 1500 | 80 |
| H(24C) | 4986 | -2215 | 1989 | 80 |
| H(25A) | 6047 | 497 | 1786 | 80 |
| H(25B) | 6151 | -680 | 1902 | 80 |
| H(26A) | 6277 | 299 | 2619 | 80 |
| H(26B) | 4855 | 797 | 2500 | 80 |
| H(26C) | 4960 | -394 | 2617 | 80 |
| H(27A) | 3046 | 745 | 2010 | 80 |
| H(27B) | 2543 | 492 | 1490 | 80 |
| H(28A) | 3260 | 2195 | 1514 | 80 |
| H(28B) | 4707 | 1824 | 1684 | 80 |
| H(28C) | 4198 | 1569 | 1158 | 80 |
| H(29A) | 7428 | 8564 | 669 | 80 |
| H(29B) | 7228 | 7798 | 232 | 80 |
| H(29C) | 8241 | 7514 | 652 | 80 |