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Table 1. Crystal data for TR2.

| | |
|---|---|
| Compound | $[Zr(C_{16}H_{14}N_2O_2)(C_4H_8O)Cl_2]$ |
| Formula | $C_{20}$ $H_{22}$ $Cl_2$ $N_2$ $O_3$ Zr |
| Formula mass | 500.52 |
| Space group | Pbca (No. 61) |
| Wavelength, MoK$\alpha$/Å | 0.71073 |
| Crystal system | Orthorhombic |
| Temperature/K | 193(2) |
| a/Å | 10.160(2) |
| b/Å | 14.341(3) |
| c/Å | 28.851(6) |
| $\alpha$/° | 90 |
| $\beta$/° | 90 |
| $\gamma$/° | 90 |
| V/$Å^3$ | 4204(2) |
| Z | 8 |
| $D_c$/g $cm^{-3}$ | 1.582 |
| $\mu/cm^{-1}$ | 8.01 |
| F(000) | 2032 |
| Crystal size/mm | 0.40 x 0.30 x 0.20 |
| $\Theta_{min/max}$/° | 2.56 / 27.50 |
| Diffractometer | Rigaku AFC-7S |
| Reflections collected | 4834 |
| Independent reflections | 4834 [R(int) = 0.0000] |
| Data reduction | TEXSAN (1993) |
| Structure solution | SHELXTL/PC (Sheldrick, 1990) |
| Structure refinement | SHELXL (Sheldrick, 1993) |
| Refinement method on $F^2$ | Full-matrix least-squares |
| Data / restraints / parameters | 4834 (n) / 0 / 253 (p) |
| Goodness-of-fit on $F^2$ (S) | 1.026 |
| Weights | $[\sigma^2(F_o^2)+(0.02P)^2+5.76P]^{-1}$ where $P=[(F_o^2)+2(F_c^2)]/3$ |
| Final R indices [$F>4\sigma(F)$] | R1 = 0.0575, wR2 = 0.1072 |
| R indices (all data) | R1 = 0.1125, wR2 = 0.1278 |
| Extinction correction | Not |
| Absorption correction (psi-scan) | 0.96 < T < 1.00 |
| Residual e-densities/e $Å^{-3}$ | 0.763, -0.407 |

$R1=\Sigma||F_o|-|F_c||/\Sigma|F_o|$ with $F>4\sigma(F)$, function minimized is $wR2=[\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{0.5}$, $S=[\Sigma[w(F_o^2-F_c^2)^2]/(n-p)]^{0.5}$. All non-H atoms were anisotropically refined, H-atoms were given an isotropic displacement factor 1.5x that of the host atom. H-atoms were in calculated positions (riding model).

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for TR2. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Zr(1) | 82(1) | 4450(1) | 1340(1) | 28(1) |
| Cl(1) | 1498(2) | 5335(1) | 797(1) | 67(1) |
| Cl(2) | -1931(2) | 3817(1) | 1718(1) | 49(1) |
| O(1) | 346(3) | 3163(2) | 1067(1) | 37(1) |
| C(2) | 83(5) | 2639(3) | 701(2) | 32(1) |
| C(3) | 618(5) | 1745(4) | 666(2) | 38(1) |
| C(4) | 391(6) | 1195(4) | 282(2) | 43(1) |
| C(5) | -383(6) | 1534(5) | -76(2) | 53(2) |
| C(6) | -946(6) | 2393(4) | -44(2) | 50(2) |
| C(7) | -731(5) | 2961(4) | 343(2) | 36(1) |
| C(8) | -1393(6) | 3843(4) | 369(2) | 43(1) |
| N(9) | -1312(5) | 4455(3) | 690(2) | 41(1) |
| C(10) | -2254(7) | 5240(5) | 666(2) | 67(2) |
| C(11) | -1691(7) | 6070(5) | 875(2) | 63(2) |
| N(12) | -1136(5) | 5831(3) | 1326(2) | 40(1) |
| C(13) | -1416(6) | 6364(4) | 1669(2) | 41(1) |
| C(14) | -922(5) | 6285(4) | 2137(2) | 37(1) |
| C(15) | -1368(6) | 6917(4) | 2468(2) | 47(1) |
| C(16) | -882(6) | 6920(4) | 2913(2) | 49(2) |
| C(17) | 99(6) | 6303(4) | 3025(2) | 48(2) |
| C(18) | 580(5) | 5673(4) | 2709(2) | 41(1) |
| C(19) | 62(5) | 5641(4) | 2262(2) | 34(1) |
| O(20) | 507(4) | 5027(3) | 1954(1) | 38(1) |
| O(21) | 2162(3) | 3902(3) | 1572(1) | 37(1) |
| C(22) | 3043(6) | 3415(5) | 1253(2) | 59(2) |
| C(23) | 4387(7) | 3513(6) | 1450(2) | 72(2) |
| C(24) | 4117(6) | 3529(5) | 1967(2) | 57(2) |
| C(25) | 2878(5) | 4116(4) | 1995(2) | 46(2) |

Table 3. Bond lengths [A] for TR2.

| | |
|---|---|
| Zr(1)-O(20) | 2.002(4) |
| Zr(1)-O(1) | 2.024(3) |
| Zr(1)-N(12) | 2.336(4) |
| Zr(1)-N(9) | 2.352(4) |
| Zr(1)-O(21) | 2.352(3) |
| Zr(1)-Cl(1) | 2.478(2) |
| Zr(1)-Cl(2) | 2.489(2) |
| O(1)-C(2) | 1.323(6) |
| C(2)-C(3) | 1.396(7) |
| C(2)-C(7) | 1.401(7) |
| C(3)-C(4) | 1.379(7) |
| C(3)-H(3A) | 0.93 |
| C(4)-C(5) | 1.388(8) |
| C(4)-H(4A) | 0.93 |
| C(5)-C(6) | 1.361(8) |
| C(5)-H(5A) | 0.93 |
| C(6)-C(7) | 1.400(7) |
| C(6)-H(6A) | 0.93 |
| C(7)-C(8) | 1.435(7) |
| C(8)-N(9) | 1.279(7) |
| C(8)-H(8A) | 0.93 |
| N(9)-C(10) | 1.479(7) |
| C(10)-C(11) | 1.452(9) |
| C(10)-H(10A) | 0.97 |
| C(10)-H(10B) | 0.97 |
| C(11)-N(12) | 1.461(7) |
| C(11)-H(11A) | 0.97 |
| C(11)-H(11B) | 0.97 |
| N(12)-C(13) | 1.282(7) |
| C(13)-C(14) | 1.443(7) |
| C(13)-H(13A) | 0.93 |
| C(14)-C(15) | 1.394(7) |
| C(14)-C(19) | 1.408(7) |
| C(15)-C(16) | 1.376(8) |
| C(15)-H(15A) | 0.93 |
| C(16)-C(17) | 1.371(8) |
| C(16)-H(16A) | 0.93 |
| C(17)-C(18) | 1.373(7) |
| C(17)-H(17A) | 0.93 |
| C(18)-C(19) | 1.395(7) |
| C(18)-H(18A) | 0.93 |
| C(19)-O(20) | 1.330(6) |
| O(21)-C(25) | 1.453(6) |
| O(21)-C(22) | 1.462(6) |
| C(22)-C(23) | 1.485(8) |
| C(22)-H(22A) | 0.97 |
| C(22)-H(22B) | 0.97 |
| C(23)-C(24) | 1.516(9) |
| C(23)-H(23A) | 0.97 |
| C(23)-H(23B) | 0.97 |
| C(24)-C(25) | 1.516(8) |
| C(24)-H(24A) | 0.97 |
| C(24)-H(24B) | 0.97 |
| C(25)-H(25A) | 0.97 |
| C(25)-H(25B) | 0.97 |

Table 4. Bond angles [deg] for TR2.

| | |
|---|---|
| O(20)-Zr(1)-O(1) | 133.86(14) |
| O(20)-Zr(1)-N(12) | 77.2(2) |
| O(1)-Zr(1)-N(12) | 146.8(2) |
| O(20)-Zr(1)-N(9) | 146.6(2) |
| O(1)-Zr(1)-N(9) | 76.8(2) |
| N(12)-Zr(1)-N(9) | 70.4(2) |
| O(20)-Zr(1)-O(21) | 72.08(13) |
| O(1)-Zr(1)-O(21) | 71.81(13) |
| N(12)-Zr(1)-O(21) | 139.8(2) |
| N(9)-Zr(1)-O(21) | 140.32(14) |
| O(20)-Zr(1)-Cl(1) | 102.88(12) |
| O(1)-Zr(1)-Cl(1) | 98.24(12) |
| N(12)-Zr(1)-Cl(1) | 82.08(13) |
| N(9)-Zr(1)-Cl(1) | 80.96(13) |
| O(21)-Zr(1)-Cl(1) | 80.16(10) |
| O(20)-Zr(1)-Cl(2) | 86.57(12) |
| O(1)-Zr(1)-Cl(2) | 86.98(11) |
| N(12)-Zr(1)-Cl(2) | 83.20(13) |
| N(9)-Zr(1)-Cl(2) | 81.72(13) |
| O(21)-Zr(1)-Cl(2) | 119.49(10) |
| Cl(1)-Zr(1)-Cl(2) | 160.24(6) |
| C(2)-O(1)-Zr(1) | 143.4(3) |
| O(1)-C(2)-C(3) | 120.1(5) |
| O(1)-C(2)-C(7) | 121.3(5) |
| C(3)-C(2)-C(7) | 118.7(5) |
| C(4)-C(3)-C(2) | 121.2(5) |
| C(4)-C(3)-H(3A) | 119.4(3) |
| C(2)-C(3)-H(3A) | 119.4(3) |
| C(3)-C(4)-C(5) | 119.5(5) |
| C(3)-C(4)-H(4A) | 120.2(3) |
| C(5)-C(4)-H(4A) | 120.2(3) |
| C(6)-C(5)-C(4) | 120.3(5) |
| C(6)-C(5)-H(5A) | 119.9(4) |
| C(4)-C(5)-H(5A) | 119.8(3) |
| C(5)-C(6)-C(7) | 121.0(6) |
| C(5)-C(6)-H(6A) | 119.5(4) |
| C(7)-C(6)-H(6A) | 119.5(4) |
| C(6)-C(7)-C(2) | 119.3(5) |
| C(6)-C(7)-C(8) | 118.7(5) |
| C(2)-C(7)-C(8) | 122.0(5) |
| N(9)-C(8)-C(7) | 127.7(5) |
| N(9)-C(8)-H(8A) | 116.1(3) |
| C(7)-C(8)-H(8A) | 116.1(3) |
| C(8)-N(9)-C(10) | 116.6(5) |
| C(8)-N(9)-Zr(1) | 127.9(4) |
| C(10)-N(9)-Zr(1) | 115.4(4) |
| C(11)-C(10)-N(9) | 110.5(5) |
| C(11)-C(10)-H(10A) | 109.5(4) |
| N(9)-C(10)-H(10A) | 109.5(3) |
| C(11)-C(10)-H(10B) | 109.5(4) |
| N(9)-C(10)-H(10B) | 109.5(4) |
| H(10A)-C(10)-H(10B) | 108.1 |
| C(10)-C(11)-N(12) | 109.2(5) |
| C(10)-C(11)-H(11A) | 109.8(4) |
| N(12)-C(11)-H(11A) | 109.8(3) |
| C(10)-C(11)-H(11B) | 109.8(4) |
| N(12)-C(11)-H(11B) | 109.8(4) |
| H(11A)-C(11)-H(11B) | 108.3 |
| C(13)-N(12)-C(11) | 117.6(5) |
| C(13)-N(12)-Zr(1) | 127.6(4) |
| C(11)-N(12)-Zr(1) | 114.7(4) |
| N(12)-C(13)-C(14) | 126.6(5) |
| N(12)-C(13)-H(13A) | 116.7(3) |

| | |
|---|---|
| C(14)-C(13)-H(13A) | 116.7(3) |
| C(15)-C(14)-C(19) | 118.8(5) |
| C(15)-C(14)-C(13) | 118.4(5) |
| C(19)-C(14)-C(13) | 122.6(5) |
| C(16)-C(15)-C(14) | 121.7(6) |
| C(16)-C(15)-H(15A) | 119.1(4) |
| C(14)-C(15)-H(15A) | 119.1(3) |
| C(17)-C(16)-C(15) | 118.6(6) |
| C(17)-C(16)-H(16A) | 120.7(4) |
| C(15)-C(16)-H(16A) | 120.7(4) |
| C(16)-C(17)-C(18) | 121.8(6) |
| C(16)-C(17)-H(17A) | 119.1(4) |
| C(18)-C(17)-H(17A) | 119.1(4) |
| C(17)-C(18)-C(19) | 120.1(6) |
| C(17)-C(18)-H(18A) | 120.0(4) |
| C(19)-C(18)-H(18A) | 120.0(3) |
| O(20)-C(19)-C(18) | 120.7(5) |
| O(20)-C(19)-C(14) | 120.3(5) |
| C(18)-C(19)-C(14) | 118.9(5) |
| C(19)-O(20)-Zr(1) | 142.3(3) |
| C(25)-O(21)-C(22) | 108.9(4) |
| C(25)-O(21)-Zr(1) | 128.2(3) |
| C(22)-O(21)-Zr(1) | 122.0(3) |
| O(21)-C(22)-C(23) | 106.1(5) |
| O(21)-C(22)-H(22A) | 110.5(3) |
| C(23)-C(22)-H(22A) | 110.5(4) |
| O(21)-C(22)-H(22B) | 110.5(3) |
| C(23)-C(22)-H(22B) | 110.5(5) |
| H(22A)-C(22)-H(22B) | 108.7 |
| C(22)-C(23)-C(24) | 102.2(5) |
| C(22)-C(23)-H(23A) | 111.3(4) |
| C(24)-C(23)-H(23A) | 111.3(4) |
| C(22)-C(23)-H(23B) | 111.3(4) |
| C(24)-C(23)-H(23B) | 111.3(4) |
| H(23A)-C(23)-H(23B) | 109.2 |
| C(23)-C(24)-C(25) | 102.3(5) |
| C(23)-C(24)-H(24A) | 111.3(4) |
| C(25)-C(24)-H(24A) | 111.3(3) |
| C(23)-C(24)-H(24B) | 111.3(4) |
| C(25)-C(24)-H(24B) | 111.3(4) |
| H(24A)-C(24)-H(24B) | 109.2 |
| O(21)-C(25)-C(24) | 104.6(5) |
| O(21)-C(25)-H(25A) | 110.8(3) |
| C(24)-C(25)-H(25A) | 110.8(4) |
| O(21)-C(25)-H(25B) | 110.8(3) |
| C(24)-C(25)-H(25B) | 110.8(4) |
| H(25A)-C(25)-H(25B) | 108.9 |

Table 5. Anisotropic displacement parameters (A^2 x 10^3) for TR2.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Zr(1) | 28(1) | 32(1) | 25(1) | 2(1) | -1(1) | 2(1) |
| Cl(1) | 47(1) | 83(1) | 71(1) | 39(1) | 8(1) | -10(1) |
| Cl(2) | 42(1) | 53(1) | 51(1) | 11(1) | 12(1) | -5(1) |
| O(1) | 43(2) | 38(2) | 28(2) | -6(2) | -7(2) | 11(2) |
| C(2) | 34(3) | 32(3) | 29(2) | -2(2) | 3(2) | -1(3) |
| C(3) | 41(3) | 43(3) | 30(3) | -3(2) | -1(2) | -1(3) |
| C(4) | 46(3) | 41(3) | 42(3) | -11(3) | 1(3) | 0(3) |
| C(5) | 70(5) | 54(4) | 34(3) | -13(3) | -12(3) | 1(3) |
| C(6) | 70(4) | 44(4) | 35(3) | -1(3) | -12(3) | -3(3) |
| C(7) | 43(3) | 38(3) | 29(3) | -4(2) | -3(2) | 2(3) |
| C(8) | 50(4) | 50(4) | 30(3) | 6(3) | -15(3) | 4(3) |
| N(9) | 42(3) | 44(3) | 38(2) | -2(2) | -12(2) | 13(2) |
| C(10) | 75(5) | 64(5) | 63(4) | -1(4) | -29(4) | 31(4) |
| C(11) | 78(5) | 50(4) | 62(4) | 2(3) | -24(4) | 16(4) |
| N(12) | 52(3) | 33(2) | 36(2) | 1(2) | -9(2) | 5(2) |
| C(13) | 40(3) | 28(3) | 55(4) | -2(3) | -5(3) | 2(2) |
| C(14) | 33(3) | 33(3) | 45(3) | -4(3) | -3(2) | 0(2) |
| C(15) | 42(3) | 37(3) | 62(4) | -11(3) | 3(3) | -2(3) |
| C(16) | 44(4) | 49(4) | 56(4) | -16(3) | 8(3) | -3(3) |
| C(17) | 45(3) | 59(4) | 40(3) | -14(3) | -1(3) | -7(3) |
| C(18) | 31(3) | 47(4) | 45(3) | -9(3) | -2(2) | -2(3) |
| C(19) | 32(3) | 32(3) | 38(3) | -6(2) | 3(2) | -4(3) |
| O(20) | 37(2) | 41(2) | 37(2) | -9(2) | -6(2) | 11(2) |
| O(21) | 29(2) | 49(2) | 33(2) | -3(2) | -2(2) | 6(2) |
| C(22) | 41(3) | 96(5) | 39(3) | -13(4) | 5(3) | 32(4) |
| C(23) | 43(4) | 102(6) | 71(5) | 4(5) | 0(4) | 18(4) |
| C(24) | 39(3) | 58(4) | 74(5) | -10(4) | -24(3) | 10(3) |
| C(25) | 38(3) | 53(4) | 47(3) | -9(3) | -18(3) | 7(3) |

Table 6. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for TR2.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(3A) | 1137 | 1516 | 906 | 57 |
| H(4A) | 753 | 600 | 264 | 64 |
| H(5A) | -518 | 1173 | -340 | 79 |
| H(6A) | -1482 | 2606 | -283 | 75 |
| H(8A) | -1947 | 3987 | 122 | 65 |
| H(10A) | -2474 | 5366 | 345 | 101 |
| H(10B) | -3057 | 5074 | 827 | 101 |
| H(11A) | -2366 | 6543 | 911 | 95 |
| H(11B) | -1007 | 6320 | 675 | 95 |
| H(13A) | -1998 | 6851 | 1611 | 62 |
| H(15A) | -2011 | 7348 | 2386 | 70 |
| H(16A) | -1211 | 7332 | 3134 | 74 |
| H(17A) | 447 | 6311 | 3323 | 72 |
| H(18A) | 1252 | 5267 | 2794 | 61 |
| H(22A) | 3000 | 3691 | 947 | 88 |
| H(22B) | 2801 | 2762 | 1230 | 88 |
| H(23A) | 4802 | 4086 | 1348 | 108 |
| H(23B) | 4941 | 2988 | 1365 | 108 |
| H(24A) | 4837 | 3815 | 2135 | 85 |
| H(24B) | 3969 | 2905 | 2086 | 85 |
| H(25A) | 2364 | 3950 | 2266 | 69 |
| H(25B) | 3094 | 4774 | 2010 | 69 |

Table 7. Least squares planes for TR2.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

7.904 (0.010) x + 5.768 (0.018) y - 13.925 (0.037) z = 0.595 (0.010)

| | | | |
|---|---|---|---|
| * | 0.017 | (0.002) | O1 |
| * | -0.023 | (0.003) | N9 |
| * | 0.023 | (0.003) | N12 |
| * | -0.017 | (0.002) | O20 |
| | 0.169 | (0.002) | Zr1 |
| | 1.175 | (0.006) | O21 |
| | 2.556 | (0.003) | Cl1 |
| | -2.312 | (0.003) | Cl2 |

Rms deviation of fitted atoms = 0.020

7.227 (0.011) x + 9.315 (0.017) y - 7.745 (0.030) z = 3.568 (0.013)

| | | | |
|---|---|---|---|
| * | 0.016 | (0.004) | N12 |
| * | 0.044 | (0.004) | C13 |
| * | -0.034 | (0.005) | C14 |
| * | -0.025 | (0.005) | C15 |
| * | -0.015 | (0.005) | C16 |
| * | 0.031 | (0.005) | C17 |
| * | 0.037 | (0.005) | C18 |
| * | -0.021 | (0.005) | C19 |
| * | -0.033 | (0.003) | O20 |
| | -0.402 | (0.004) | Zr1 |

Rms deviation of fitted atoms = 0.030

8.019 (0.010) x + 5.860 (0.015) y - 13.225 (0.041) z = 0.674 (0.005)

| | | | |
|---|---|---|---|
| * | 0.046 | (0.003) | O1 |
| * | 0.012 | (0.005) | C2 |
| * | -0.037 | (0.004) | C3 |
| * | -0.034 | (0.005) | C4 |
| * | 0.019 | (0.005) | C5 |
| * | 0.028 | (0.005) | C6 |
| * | 0.020 | (0.005) | C7 |
| * | -0.027 | (0.005) | C8 |
| * | -0.027 | (0.004) | N9 |
| | 0.227 | (0.005) | Zr1 |

Rms deviation of fitted atoms = 0.029