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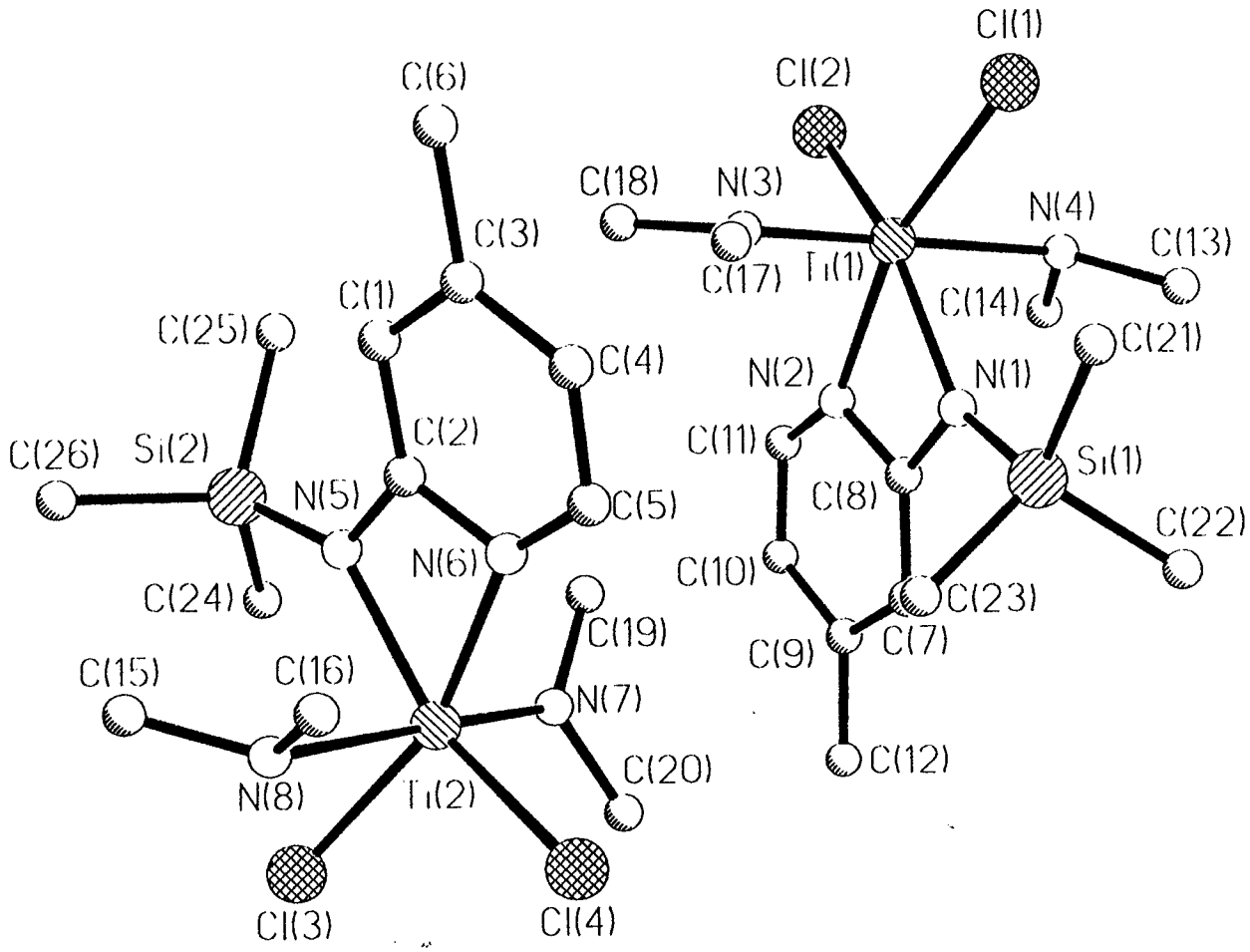


(4-Me-TMS- APy)($NMe_2$)($HNMe_2$)$TiCl_2$ (**1**)

Table 1. Crystal data and structure refinement for 1.

| | |
|---|---|
| Identification code | 520 |
| Empirical formula | C13 H28 Cl2 N4 Si1 Ti1 |
| Formula weight | 372 |
| Crystal system | Monoclinic |
| Space group | P21/c |
| Unit cell dimensions | a= 16.754(2) A alpha= 90 deg.<br>b= 14.395(2) A beta= 110.38(1) deg.<br>c= 17.890(3) A gamma= 90 deg. |
| Temperature for cell | 293(2) |
| Number of reflecions for cell | 2000 |
| Theta for cell determination | 4 to 24 deg |
| Volume | 4044.5(9) A^3 |
| Z | 8 |
| Density (calculated) | 1.272 Mg/m^3 |
| Absorption coefficient | 0.746 mm^-1 |
| F(000) | 1632 |
| Crystal colour | brown |
| Crystal description | prism |
| Crystal size | 0.7 x 0.5 x 0.2 mm |
| Temperature | 293(2) K |
| Wavelength | 0.71069 A |
| Radiation type | MoK\a |
| Radiation source | fine-focus sealed tube |
| Monochromator | graphite |
| Measurment device | STOE-IPDS |
| Scan method | laser scanned imaging plate |
| Standards (intensity + orient.) | 50-200 |
| Intensity after | 360 sec |
| Theta range for data collection | 1.86 to 24.33 deg. |
| Index ranges | -19<=h<=17, -16<=k<=16, 0<=l<=20 |
| Reflections collected | 25874 |
| Independent reflections | 6501 [R(int) = 0.0453] |

| | |
|---|---|
| Reflections unique | 6501 |
| Reflections observed | 3876 |
| Criterion | >2sigma(I) |
| Structure solution primary | direct |
| Structure solution secondary | difmap |
| Hydrogen positions | geom |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 6501 / 0 / 380 |
| Final R indices [I>2sigma(I)] | R1 = 0.0414, wR2 = 0.0991 |
| R indices (all data) | R1 = 0.0848, wR2 = 0.1131 |
| Goodness-of-fit on F^2(all) | 0.885 |
| Goodness-of-fit on F^2(obs) | 1.022 |
| Shift/esd(max) | -0.001 |
| Shift/esd(mean) | 0.000 |
| Weighting scheme | |
| calc w=1/[\s^2^(Fo^2^)+(0.0622P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3 | |
| Largest diff. peak and hole | 0.320 and -0.297 e.A^-3 |
| Diff. density rms | 0.056 |
| Data collection | STOE-EXPOSE |
| Cell refinement | STOE-CELL |
| Data reduction | STOE-INTEGRATE |
| Structure solution | SHELXS-86 (Sheldrick, 1990) |
| Structure refinement | SHELXL-93 (Sheldrick, 1993) |
| Molecular graphics | XP-SIEMENS |
| Publication material | SHELXL-93 |

Table 2. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | 7565(2) | -2309(2) | 6281(2) | 42(1) |
| C(2) | 7927(2) | -1439(2) | 6250(2) | 35(1) |
| C(3) | 7902(2) | -2866(2) | 6941(2) | 45(1) |
| C(4) | 8615(2) | -2551(2) | 7563(2) | 53(1) |
| C(5) | 8959(2) | -1709(2) | 7502(2) | 56(1) |
| C(6) | 7526(3) | -3801(2) | 6994(2) | 72(1) |
| C(7) | 7455(2) | 2218(2) | 8532(2) | 42(1) |
| C(8) | 6995(2) | 1432(2) | 8617(2) | 38(1) |
| C(9) | 7056(2) | 2888(2) | 7980(2) | 47(1) |
| C(10) | 6203(2) | 2782(2) | 7523(2) | 56(1) |
| C(11) | 5773(2) | 2022(2) | 7634(2) | 60(1) |
| C(12) | 7537(3) | 3729(2) | 7866(3) | 70(1) |
| C(13) | 6464(3) | 1177(3) | 10468(3) | 105(2) |
| C(14) | 5425(3) | 2043(3) | 9422(3) | 107(2) |
| C(15) | 9058(3) | -1518(3) | 4879(3) | 87(2) |
| C(16) | 9954(3) | -1945(3) | 6196(3) | 89(2) |
| C(17) | 6811(3) | -1255(3) | 8029(3) | 102(2) |
| C(18) | 5575(3) | -615(4) | 7080(3) | 121(2) |
| C(19) | 7481(3) | 841(3) | 6733(3) | 91(2) |
| C(20) | 8716(4) | 1772(3) | 6907(3) | 109(2) |
| C(21) | 8356(3) | -710(3) | 10180(3) | 91(2) |
| C(22) | 8773(3) | 1300(3) | 10445(3) | 94(2) |
| C(23) | 8935(3) | 353(3) | 9011(3) | 86(1) |
| C(24) | 6650(3) | 597(3) | 4591(3) | 94(2) |
| C(25) | 5864(3) | -1075(4) | 5032(3) | 105(2) |
| C(26) | 6867(3) | -1294(4) | 3968(3) | 127(2) |
| N(1) | 7239(2) | 678(2) | 9110(2) | 40(1) |
| N(2) | 6159(2) | 1359(2) | 8166(2) | 46(1) |
| N(3) | 6127(2) | -644(2) | 7927(2) | 58(1) |
| N(4) | 5777(2) | 1109(2) | 9681(2) | 64(1) |
| N(5) | 7730(2) | -768(2) | 5669(1) | 37(1) |
| N(6) | 8625(2) | -1166(2) | 6865(2) | 42(1) |
| N(7) | 8298(2) | 889(2) | 6613(2) | 60(1) |
| N(8) | 9518(2) | -1164(2) | 5678(2) | 61(1) |
| Si(1) | 8295(1) | 418(1) | 9674(1) | 51(1) |
| Si(2) | 6805(1) | -649(1) | 4837(1) | 47(1) |
| Cl(1) | 6102(1) | -1029(1) | 9667(1) | 69(1) |
| Cl(2) | 4547(1) | 254(1) | 8144(1) | 86(1) |
| Cl(3) | 8909(1) | 849(1) | 5125(1) | 75(1) |
| Cl(4) | 10182(1) | 159(1) | 7124(1) | 95(1) |
| Ti(1) | 6023(1) | 119(1) | 8735(1) | 43(1) |
| Ti(2) | 8814(1) | -25(1) | 6186(1) | 45(1) |

Table 3. Bond lengths [A] and angles [deg] for 1.

| | |
|---|---|
| C(1)-C(3) | 1.374(4) |
| C(1)-C(2) | 1.402(4) |
| C(2)-N(6) | 1.355(4) |
| C(2)-N(5) | 1.373(4) |
| C(2)-Ti(2) | 2.546(3) |
| C(3)-C(4) | 1.394(4) |
| C(3)-C(6) | 1.504(4) |
| C(4)-C(5) | 1.363(5) |
| C(5)-N(6) | 1.334(4) |
| C(7)-C(9) | 1.375(4) |
| C(7)-C(8) | 1.407(4) |
| C(8)-N(2) | 1.355(4) |
| C(8)-N(1) | 1.368(4) |
| C(8)-Ti(1) | 2.552(3) |
| C(9)-C(10) | 1.386(5) |
| C(9)-C(12) | 1.508(4) |
| C(10)-C(11) | 1.363(5) |
| C(11)-N(2) | 1.344(4) |
| C(13)-N(4) | 1.479(5) |
| C(14)-N(4) | 1.477(5) |
| C(15)-N(8) | 1.460(5) |
| C(16)-N(8) | 1.478(4) |
| C(17)-N(3) | 1.407(5) |
| C(18)-N(3) | 1.476(5) |
| C(19)-N(7) | 1.459(5) |
| C(20)-N(7) | 1.458(5) |
| C(21)-Si(1) | 1.845(4) |
| C(22)-Si(1) | 1.841(4) |
| C(23)-Si(1) | 1.858(4) |
| C(24)-Si(2) | 1.844(4) |
| C(25)-Si(2) | 1.835(4) |
| C(26)-Si(2) | 1.845(4) |
| N(1)-Si(1) | 1.745(3) |
| N(1)-Ti(1) | 2.073(3) |
| N(2)-Ti(1) | 2.107(3) |
| N(3)-Ti(1) | 1.872(3) |
| N(4)-Ti(1) | 2.355(3) |
| N(5)-Si(2) | 1.743(2) |
| N(5)-Ti(2) | 2.032(2) |
| N(6)-Ti(2) | 2.132(3) |
| N(7)-Ti(2) | 1.877(3) |
| N(8)-Ti(2) | 2.378(3) |
| Cl(1)-Ti(1) | 2.3172(11) |
| Cl(2)-Ti(1) | 2.3347(12) |
| Cl(3)-Ti(2) | 2.3286(12) |
| Cl(4)-Ti(2) | 2.3369(12) |

| | |
|---|---|
| C(3)-C(1)-C(2) | 120.0(3) |
| N(6)-C(2)-N(5) | 108.9(3) |
| N(6)-C(2)-C(1) | 119.3(3) |
| N(5)-C(2)-C(1) | 131.7(3) |
| N(6)-C(2)-Ti(2) | 56.8(2) |
| N(5)-C(2)-Ti(2) | 52.67(14) |
| C(1)-C(2)-Ti(2) | 169.8(2) |
| C(1)-C(3)-C(4) | 118.6(3) |
| C(1)-C(3)-C(6) | 121.3(3) |
| C(4)-C(3)-C(6) | 120.1(3) |
| C(5)-C(4)-C(3) | 119.6(3) |
| N(6)-C(5)-C(4) | 121.7(3) |
| C(9)-C(7)-C(8) | 119.6(3) |
| N(2)-C(8)-N(1) | 109.7(3) |
| N(2)-C(8)-C(7) | 119.3(3) |

| | |
|---|---|
| N(2)-C(8)-Ti(1) | 55.6(2) |
| N(1)-C(8)-Ti(1) | 54.2(2) |
| C(7)-C(8)-Ti(1) | 173.9(2) |
| C(7)-C(9)-C(10) | 119.4(3) |
| C(7)-C(9)-C(12) | 120.7(3) |
| C(10)-C(9)-C(12) | 119.9(3) |
| C(11)-C(10)-C(9) | 119.4(3) |
| N(2)-C(11)-C(10) | 121.6(3) |
| C(8)-N(1)-Si(1) | 123.9(2) |
| C(8)-N(1)-Ti(1) | 93.5(2) |
| Si(1)-N(1)-Ti(1) | 141.99(14) |
| C(11)-N(2)-C(8) | 120.6(3) |
| C(11)-N(2)-Ti(1) | 146.9(2) |
| C(8)-N(2)-Ti(1) | 92.4(2) |
| C(17)-N(3)-C(18) | 110.0(4) |
| C(17)-N(3)-Ti(1) | 123.9(3) |
| C(18)-N(3)-Ti(1) | 125.7(3) |
| C(14)-N(4)-C(13) | 109.4(3) |
| C(14)-N(4)-Ti(1) | 118.5(3) |
| C(13)-N(4)-Ti(1) | 116.8(2) |
| C(2)-N(5)-Si(2) | 128.6(2) |
| C(2)-N(5)-Ti(2) | 94.8(2) |
| Si(2)-N(5)-Ti(2) | 136.47(14) |
| C(5)-N(6)-C(2) | 120.7(3) |
| C(5)-N(6)-Ti(2) | 146.9(2) |
| C(2)-N(6)-Ti(2) | 91.0(2) |
| C(19)-N(7)-C(20) | 110.2(4) |
| C(19)-N(7)-Ti(2) | 128.0(3) |
| C(20)-N(7)-Ti(2) | 121.8(3) |
| C(15)-N(8)-C(16) | 109.0(3) |
| C(15)-N(8)-Ti(2) | 116.9(2) |
| C(16)-N(8)-Ti(2) | 119.2(3) |
| N(1)-Si(1)-C(22) | 112.0(2) |
| N(1)-Si(1)-C(21) | 110.2(2) |
| C(22)-Si(1)-C(21) | 107.7(2) |
| N(1)-Si(1)-C(23) | 109.7(2) |
| C(22)-Si(1)-C(23) | 108.4(2) |
| C(21)-Si(1)-C(23) | 108.8(2) |
| N(5)-Si(2)-C(25) | 111.9(2) |
| N(5)-Si(2)-C(24) | 108.0(2) |
| C(25)-Si(2)-C(24) | 108.2(2) |
| N(5)-Si(2)-C(26) | 112.0(2) |
| C(25)-Si(2)-C(26) | 107.2(3) |
| C(24)-Si(2)-C(26) | 109.4(3) |
| N(3)-Ti(1)-N(1) | 97.60(12) |
| N(3)-Ti(1)-N(2) | 93.96(12) |
| N(1)-Ti(1)-N(2) | 64.36(10) |
| N(3)-Ti(1)-Cl(1) | 98.02(10) |
| N(1)-Ti(1)-Cl(1) | 103.94(8) |
| N(2)-Ti(1)-Cl(1) | 164.39(8) |
| N(3)-Ti(1)-Cl(2) | 93.90(11) |
| N(1)-Ti(1)-Cl(2) | 151.73(8) |
| N(2)-Ti(1)-Cl(2) | 89.21(8) |
| Cl(1)-Ti(1)-Cl(2) | 99.88(5) |
| N(3)-Ti(1)-N(4) | 175.25(13) |
| N(1)-Ti(1)-N(4) | 86.03(11) |
| N(2)-Ti(1)-N(4) | 84.82(11) |
| Cl(1)-Ti(1)-N(4) | 84.02(8) |
| Cl(2)-Ti(1)-N(4) | 81.50(9) |
| N(3)-Ti(1)-C(8) | 97.87(12) |
| N(1)-Ti(1)-C(8) | 32.34(9) |
| N(2)-Ti(1)-C(8) | 32.05(9) |
| Cl(1)-Ti(1)-C(8) | 135.20(8) |
| Cl(2)-Ti(1)-C(8) | 120.44(7) |
| N(4)-Ti(1)-C(8) | 83.54(10) |

| | |
|---|---|
| N(7)-Ti(2)-N(6) | 97.34(12) |
| N(5)-Ti(2)-N(6) | 64.36(10) |
| N(7)-Ti(2)-Cl(3) | 97.19(10) |
| N(5)-Ti(2)-Cl(3) | 102.56(8) |
| N(6)-Ti(2)-Cl(3) | 161.26(8) |
| N(7)-Ti(2)-Cl(4) | 95.96(11) |
| N(5)-Ti(2)-Cl(4) | 152.17(8) |
| N(6)-Ti(2)-Cl(4) | 88.92(8) |
| Cl(3)-Ti(2)-Cl(4) | 101.27(5) |
| N(7)-Ti(2)-N(8) | 177.84(13) |
| N(5)-Ti(2)-N(8) | 86.97(10) |
| N(6)-Ti(2)-N(8) | 82.99(11) |
| Cl(3)-Ti(2)-N(8) | 82.94(8) |
| Cl(4)-Ti(2)-N(8) | 81.91(8) |
| N(7)-Ti(2)-C(2) | 100.60(12) |
| N(5)-Ti(2)-C(2) | 32.50(9) |
| N(6)-Ti(2)-C(2) | 32.14(9) |
| Cl(3)-Ti(2)-C(2) | 132.49(8) |
| Cl(4)-Ti(2)-C(2) | 119.99(8) |
| N(8)-Ti(2)-C(2) | 80.83(10) |

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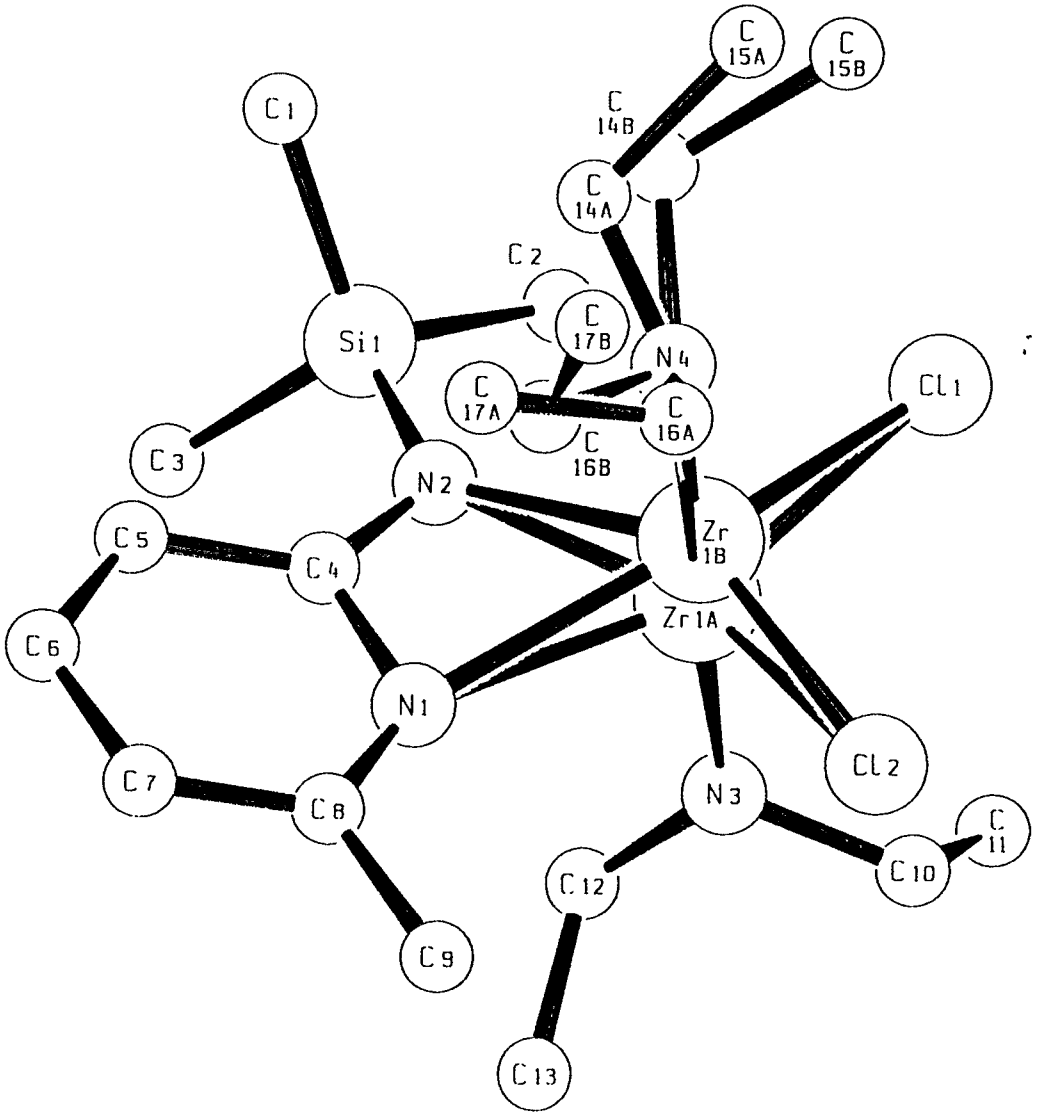
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 1. 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 |
|---|---|---|---|---|---|---|
| C(1) | 46(2) | 41(2) | 37(2) | -2(2) | 12(2) | -6(2) |
| C(2) | 36(2) | 36(2) | 35(2) | -6(2) | 14(2) | -3(1) |
| C(3) | 52(2) | 38(2) | 51(2) | 9(2) | 25(2) | 2(2) |
| C(4) | 65(3) | 53(2) | 41(2) | 9(2) | 17(2) | 6(2) |
| C(5) | 55(2) | 58(2) | 46(2) | 1(2) | 4(2) | 0(2) |
| C(6) | 84(3) | 51(2) | 83(3) | 19(2) | 30(3) | -11(2) |
| C(7) | 43(2) | 38(2) | 43(2) | 1(2) | 13(2) | -3(2) |
| C(8) | 40(2) | 34(2) | 41(2) | 6(2) | 15(2) | 5(2) |
| C(9) | 62(2) | 38(2) | 46(2) | -1(2) | 26(2) | 0(2) |
| C(10) | 64(3) | 45(2) | 52(2) | 19(2) | 13(2) | 8(2) |
| C(11) | 49(2) | 57(2) | 60(2) | 21(2) | 2(2) | 6(2) |
| C(12) | 81(3) | 48(2) | 87(3) | 14(2) | 38(3) | -10(2) |
| C(13) | 134(5) | 111(4) | 82(3) | -37(3) | 54(4) | -4(4) |
| C(14) | 136(5) | 50(3) | 168(5) | -7(3) | 96(4) | 20(3) |
| C(15) | 77(3) | 92(3) | 95(4) | -33(3) | 33(3) | -1(3) |
| C(16) | 78(3) | 70(3) | 133(4) | 26(3) | 55(3) | 35(2) |
| C(17) | 139(5) | 81(3) | 90(4) | -17(3) | 47(3) | 30(3) |
| C(18) | 123(5) | 166(6) | 66(3) | -29(3) | 24(3) | -31(4) |
| C(19) | 110(4) | 92(3) | 84(3) | -29(3) | 49(3) | 13(3) |
| C(20) | 181(6) | 52(3) | 107(4) | -20(3) | 67(4) | -29(3) |
| C(21) | 64(3) | 79(3) | 115(4) | 40(3) | 13(3) | 10(2) |
| C(22) | 85(3) | 93(4) | 73(3) | -21(3) | -12(3) | -3(3) |
| C(23) | 56(3) | 106(4) | 96(3) | 15(3) | 28(2) | 21(2) |
| C(24) | 77(3) | 63(3) | 108(4) | 34(3) | -12(3) | -2(2) |
| C(25) | 53(3) | 129(4) | 120(4) | 51(4) | 14(3) | -6(3) |
| C(26) | 106(4) | 186(6) | 58(3) | -39(3) | -11(3) | 49(4) |
| N(1) | 41(2) | 34(1) | 44(2) | 5(1) | 15(1) | 2(1) |
| N(2) | 36(2) | 41(2) | 54(2) | 9(1) | 8(1) | 2(1) |
| N(3) | 72(2) | 49(2) | 56(2) | -9(2) | 27(2) | -13(2) |
| N(4) | 75(2) | 48(2) | 90(3) | -2(2) | 55(2) | 2(2) |
| N(5) | 42(2) | 37(1) | 33(1) | 6(1) | 12(1) | -1(1) |
| N(6) | 49(2) | 35(2) | 37(2) | 3(1) | 8(1) | -2(1) |
| N(7) | 86(2) | 40(2) | 56(2) | -5(2) | 26(2) | -5(2) |
| N(8) | 44(2) | 60(2) | 84(2) | 5(2) | 30(2) | -1(2) |
| Si(1) | 41(1) | 51(1) | 53(1) | 8(1) | 7(1) | 5(1) |
| Si(2) | 44(1) | 47(1) | 44(1) | 6(1) | 8(1) | -1(1) |
| Cl(1) | 87(1) | 51(1) | 80(1) | 20(1) | 41(1) | 3(1) |
| Cl(2) | 41(1) | 75(1) | 127(1) | 6(1) | 10(1) | -6(1) |
| Cl(3) | 80(1) | 67(1) | 92(1) | 26(1) | 50(1) | 4(1) |
| Cl(4) | 64(1) | 79(1) | 108(1) | 1(1) | -14(1) | -27(1) |
| Ti(1) | 42(1) | 35(1) | 53(1) | 2(1) | 16(1) | -2(1) |
| Ti(2) | 44(1) | 37(1) | 51(1) | 2(1) | 13(1) | -7(1) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 1.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1) | 7095(2) | -2510(2) | 5856(2) | 50 |
| H(4) | 8854(2) | -2914(2) | 8016(2) | 64 |
| H(5) | 9439(2) | -1507(2) | 7916(2) | 68 |
| H(6A) | 7845(3) | -4088(2) | 7493(2) | 108 |
| H(6B) | 7549(3) | -4187(2) | 6564(2) | 108 |
| H(6C) | 6944(3) | -3727(2) | 6957(2) | 108 |
| H(7) | 8025(2) | 2285(2) | 8848(2) | 50 |
| H(10) | 5927(2) | 3227(2) | 7144(2) | 67 |
| H(11) | 5197(2) | 1960(2) | 7334(2) | 72 |
| H(12A) | 7161(3) | 4121(2) | 7461(3) | 105 |
| H(12B) | 8002(3) | 3534(2) | 7706(3) | 105 |
| H(12C) | 7754(3) | 4068(2) | 8358(3) | 105 |
| H(13A) | 6692(3) | 570(3) | 10638(3) | 157 |
| H(13B) | 6238(3) | 1431(3) | 10849(3) | 157 |
| H(13C) | 6907(3) | 1574(3) | 10428(3) | 157 |
| H(14A) | 4977(3) | 2000(3) | 8912(3) | 161 |
| H(14B) | 5867(3) | 2442(3) | 9380(3) | 161 |
| H(14C) | 5201(3) | 2296(3) | 9806(3) | 161 |
| H(15A) | 8774(3) | -1015(3) | 4538(3) | 131 |
| H(15B) | 8646(3) | -1969(3) | 4904(3) | 131 |
| H(15C) | 9452(3) | -1805(3) | 4668(3) | 131 |
| H(16A) | 10261(3) | -1717(3) | 6723(3) | 133 |
| H(16B) | 10345(3) | -2232(3) | 5982(3) | 133 |
| H(16C) | 9540(3) | -2393(3) | 6221(3) | 133 |
| H(17A) | 7167(3) | -1270(3) | 8581(3) | 152 |
| H(17B) | 7138(3) | -1045(3) | 7714(3) | 152 |
| H(17C) | 6596(3) | -1867(3) | 7860(3) | 152 |
| H(18A) | 5112(3) | -192(4) | 7015(3) | 181 |
| H(18B) | 5353(3) | -1225(4) | 6911(3) | 181 |
| H(18C) | 5900(3) | -409(4) | 6763(3) | 181 |
| H(19A) | 7211(3) | 259(3) | 6535(3) | 137 |
| H(19B) | 7123(3) | 1342(3) | 6451(3) | 137 |
| H(19C) | 7572(3) | 892(3) | 7292(3) | 137 |
| H(20A) | 9251(4) | 1797(3) | 6823(3) | 164 |
| H(20B) | 8812(4) | 1827(3) | 7467(3) | 164 |
| H(20C) | 8360(4) | 2274(3) | 6626(3) | 164 |
| H(21A) | 8110(3) | -1184(3) | 9791(3) | 136 |
| H(21B) | 8050(3) | -674(3) | 10544(3) | 136 |
| H(21C) | 8942(3) | -859(3) | 10470(3) | 136 |
| H(22A) | 8749(3) | 1896(3) | 10197(3) | 141 |
| H(22B) | 9356(3) | 1140(3) | 10733(3) | 141 |
| H(22C) | 8464(3) | 1323(3) | 10807(3) | 141 |
| H(23A) | 8693(3) | -105(3) | 8604(3) | 129 |
| H(23B) | 9510(3) | 182(3) | 9319(3) | 129 |
| H(23C) | 8933(3) | 947(3) | 8767(3) | 129 |
| H(24A) | 7136(3) | 834(3) | 4486(3) | 141 |
| H(24B) | 6583(3) | 928(3) | 5031(3) | 141 |
| H(24C) | 6149(3) | 678(3) | 4127(3) | 141 |
| H(25A) | 5932(3) | -1724(4) | 5162(3) | 157 |
| H(25B) | 5367(3) | -986(4) | 4566(3) | 157 |
| H(25C) | 5800(3) | -737(4) | 5471(3) | 157 |
| H(26A) | 7354(3) | -1088(4) | 3849(3) | 191 |
| H(26B) | 6360(3) | -1184(4) | 3517(3) | 191 |
| H(26C) | 6918(3) | -1947(4) | 4087(3) | 191 |
| H(4A) | 5350(2) | 818(2) | 9794(2) | 77 |
| H(8) | 9956(2) | -841(2) | 5612(2) | 73 |




SCHAKAL

(6-Me-TMS- APy)($NEt_2$)($HNEt_2$)**Zr**$Cl_2$ (**2**)

Table 1. Crystal data and structure refinement for 2.

| | |
|---|---|
| Identification code | 511 |
| Empirical formula | C17 H36 Cl2 N4 Si Zr |
| Formula weight | 486.71 |
| Crystal system | Monoclinic |
| Space group | P21/n |
| Unit cell dimensions | a= 10.125(1) A alpha= 90 deg.<br>b= 16.331(1) A beta= 93.90(1) deg.<br>c= 15.276(2) A gamma= 90 deg. |
| Temperature for cell | 293(2) |
| Number of reflecions for cell | 2000 |
| Theta for cell determination | 4 to 24 deg |
| Volume | 2520.1(4) A^3 |
| Z | 4 |
| Density (calculated) | 1.283 Mg/m^3 |
| Absorption coefficient | 0.704 mm^-1 |
| F(000) | 1016 |
| Crystal colour | yellow |
| Crystal description | prism |
| Crystal size | 0.3 x 0.2 x 0.2 mm |
| Temperature | 293(2) K |
| Wavelength | 0.71069 A |
| Radiation type | MoK\a |
| Radiation source | fine-focus sealed tube |
| Monochromator | graphite |
| Measurment device | STOE-IPDS |
| Scan method | LASER SCANNED IMAGING PLATE |
| Standards (intensity + orient.) | 50-200 |
| Intensity after | 360 sec |
| Theta range for data collection | 1.83 to 24.20 deg. |
| Index ranges | -10<=h<=10, 0<=k<=18, 0<=l<=17 |
| Reflections collected | 17342 |
| Independent reflections | 3560 [R(int) = 0.0620] |

| | |
|---|---|
| Reflections unique | 3560 |
| Reflections observed | 2506 |
| Criterion | >2sigma(I) |
| Absorption correction | No |
| Structure solution primary | direct |
| Structure solution secondary | difmap |
| Hydrogen positions | geom |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 3560 / 0 / 234 |
| Final R indices [I>2sigma(I)] | R1 = 0.0536, wR2 = 0.1473 |
| R indices (all data) | R1 = 0.0752, wR2 = 0.1570 |
| Goodness-of-fit on F^2(all) | 0.998 |
| Goodness-of-fit on F^2(obs) | 1.129 |
| Shift/esd(max) | -0.093 |
| Shift/esd(mean) | 0.015 |

Weighting scheme

calc w=1/[\s^2^(Fo^2^)+(0.0907P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3

| | |
|---|---|
| Largest diff. peak and hole | 0.401 and -0.954 e.A^-3 |
| Diff. density rms | 0.079 |
| Data collection | STOE-EXPOSE |
| Cell refinement | STOE-CELL |
| Data reduction | STOE-INTEGRATE |
| Structure solution | SHELXS-86 (Sheldrick, 1990) |
| Structure refinement | SHELXL-93 (Sheldrick, 1993) |
| Molecular graphics | SCHAKAL |
| Publication material | SHELXL-93 |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| C(1) | -938(6) | 4192(4) | 6693(5) | 77(2) |
| C(2) | -2628(6) | 3530(4) | 8077(5) | 80(2) |
| C(3) | -165(7) | 4488(4) | 8624(5) | 92(2) |
| C(4) | 1282(4) | 2760(3) | 7900(4) | 47(1) |
| C(5) | 2238(5) | 3346(3) | 7693(4) | 62(2) |
| C(6) | 3550(6) | 3144(4) | 7786(5) | 84(2) |
| C(7) | 3932(6) | 2379(4) | 8063(6) | 86(2) |
| C(8) | 2996(5) | 1803(4) | 8254(5) | 71(2) |
| C(9) | 3379(7) | 968(4) | 8585(7) | 122(4) |
| C(10) | -1543(6) | 940(4) | 9707(5) | 74(2) |
| C(11) | -2842(7) | 1202(6) | 10035(6) | 109(3) |
| C(12) | -328(9) | 2197(5) | 10009(6) | 101(2) |
| C(13) | 753(9) | 1905(7) | 10625(7) | 142(4) |
| C(14A) | -275(34) | 1966(23) | 5870(25) | 207(15) |
| C(15A) | -1274(30) | 1598(18) | 5117(22) | 169(12) |
| C(16A) | 989(22) | 803(16) | 6151(18) | 144(8) |
| C(17A) | 2128(33) | 1302(20) | 5899(26) | 198(14) |
| C(14B) | -884(13) | 1907(8) | 5875(9) | 58(3) |
| C(15B) | -1830(20) | 1329(14) | 5239(15) | 117(6) |
| C(16B) | 1409(20) | 1341(13) | 6269(13) | 102(5) |
| C(17B) | 1671(25) | 894(16) | 5437(18) | 157(9) |
| N(1) | 1719(3) | 2001(2) | 8174(3) | 52(1) |
| N(2) | -39(3) | 2830(2) | 7881(3) | 44(1) |
| N(3) | -799(4) | 1611(3) | 9337(3) | 60(1) |
| N(4) | -40(5) | 1394(3) | 6483(4) | 71(1) |
| Si(1) | -914(1) | 3747(1) | 7813(1) | 54(1) |
| Cl(1) | -2767(1) | 1379(1) | 7503(1) | 76(1) |
| Cl(2) | 269(2) | 103(1) | 8055(1) | 78(1) |
| Zr(1A) | -463(1) | 1539(1) | 8062(1) | 45(1) |
| Zr(1B) | -404(11) | 1474(7) | 7660(6) | 50(3) |

Table 3. Bond lengths [A] and angles [deg] for 2.

| | |
|---|---|
| C(1)-Si(1) | 1.858(7) |
| C(2)-Si(1) | 1.842(6) |
| C(3)-Si(1) | 1.856(7) |
| C(4)-N(2) | 1.341(6) |
| C(4)-N(1) | 1.373(6) |
| C(4)-C(5) | 1.411(7) |
| C(4)-Zr(1A) | 2.687(5) |
| C(4)-Zr(1B) | 2.716(12) |
| C(5)-C(6) | 1.367(8) |
| C(6)-C(7) | 1.367(9) |
| C(7)-C(8) | 1.381(9) |
| C(8)-N(1) | 1.330(6) |
| C(8)-C(9) | 1.497(8) |
| C(10)-N(3) | 1.464(7) |
| C(10)-C(11) | 1.501(9) |
| C(12)-N(3) | 1.460(9) |
| C(12)-C(13) | 1.474(11) |
| C(14A)-N(4) | 1.33(4) |
| C(14A)-C(15A) | 1.60(4) |
| C(14A)-Zr(1B) | 2.86(4) |
| C(16A)-C(17A) | 1.48(4) |
| C(16A)-N(4) | 1.53(2) |
| C(14B)-N(4) | 1.48(2) |
| C(14B)-C(15B) | 1.62(3) |
| C(14B)-Zr(1B) | 2.83(2) |
| C(16B)-C(17B) | 1.51(3) |
| C(16B)-N(4) | 1.53(2) |
| N(1)-Zr(1A) | 2.329(4) |
| N(1)-Zr(1B) | 2.398(12) |
| N(2)-Si(1) | 1.740(4) |
| N(2)-Zr(1A) | 2.172(4) |
| N(2)-Zr(1B) | 2.266(12) |
| N(3)-Zr(1A) | 2.003(5) |
| N(3)-Zr(1B) | 2.630(10) |
| N(4)-Zr(1B) | 1.864(10) |
| N(4)-Zr(1A) | 2.490(5) |
| Cl(1)-Zr(1B) | 2.393(12) |
| Cl(1)-Zr(1A) | 2.443(2) |
| Cl(2)-Zr(1B) | 2.406(12) |
| Cl(2)-Zr(1A) | 2.461(2) |
| Zr(1A)-Zr(1B) | 0.631(9) |
| | |
| N(2)-C(4)-N(1) | 112.5(4) |
| N(2)-C(4)-C(5) | 129.5(4) |
| N(1)-C(4)-C(5) | 118.0(4) |
| N(2)-C(4)-Zr(1A) | 53.4(2) |
| N(1)-C(4)-Zr(1A) | 60.1(2) |
| C(5)-C(4)-Zr(1A) | 171.4(4) |
| N(2)-C(4)-Zr(1B) | 56.3(3) |
| N(1)-C(4)-Zr(1B) | 61.9(3) |
| C(5)-C(4)-Zr(1B) | 158.2(5) |
| Zr(1A)-C(4)-Zr(1B) | 13.4(2) |
| C(6)-C(5)-C(4) | 119.3(5) |
| C(7)-C(6)-C(5) | 120.4(6) |
| C(6)-C(7)-C(8) | 120.3(5) |
| N(1)-C(8)-C(7) | 119.5(5) |
| N(1)-C(8)-C(9) | 118.7(5) |
| C(7)-C(8)-C(9) | 121.8(5) |
| N(3)-C(10)-C(11) | 113.7(6) |
| N(3)-C(12)-C(13) | 115.4(8) |
| N(4)-C(14A)-C(15A) | 109(3) |
| N(4)-C(14A)-Zr(1B) | 31.7(12) |

| | |
|---|---|
| C(15A)-C(14A)-Zr(1B) | 121(2) |
| C(17A)-C(16A)-N(4) | 107(2) |
| N(4)-C(14B)-C(15B) | 109.9(11) |
| N(4)-C(14B)-Zr(1B) | 36.7(5) |
| C(15B)-C(14B)-Zr(1B) | 119.3(11) |
| C(17B)-C(16B)-N(4) | 116(2) |
| C(8)-N(1)-C(4) | 122.6(4) |
| C(8)-N(1)-Zr(1A) | 147.1(4) |
| C(4)-N(1)-Zr(1A) | 89.2(3) |
| C(8)-N(1)-Zr(1B) | 141.9(5) |
| C(4)-N(1)-Zr(1B) | 87.8(4) |
| Zr(1A)-N(1)-Zr(1B) | 15.3(2) |
| C(4)-N(2)-Si(1) | 125.3(3) |
| C(4)-N(2)-Zr(1A) | 96.9(3) |
| Si(1)-N(2)-Zr(1A) | 137.6(2) |
| C(4)-N(2)-Zr(1B) | 94.2(4) |
| Si(1)-N(2)-Zr(1B) | 139.0(4) |
| Zr(1A)-N(2)-Zr(1B) | 16.2(2) |
| C(12)-N(3)-C(10) | 111.8(6) |
| C(12)-N(3)-Zr(1A) | 130.7(5) |
| C(10)-N(3)-Zr(1A) | 117.3(4) |
| C(12)-N(3)-Zr(1B) | 132.6(5) |
| C(10)-N(3)-Zr(1B) | 115.4(5) |
| Zr(1A)-N(3)-Zr(1B) | 1.8(3) |
| C(14B)-N(4)-C(16B) | 114.9(10) |
| C(14A)-N(4)-C(16A) | 108(2) |
| C(14A)-N(4)-Zr(1B) | 126(2) |
| C(14B)-N(4)-Zr(1B) | 115.0(7) |
| C(16B)-N(4)-Zr(1B) | 117.8(9) |
| C(16A)-N(4)-Zr(1B) | 123.1(11) |
| C(14A)-N(4)-Zr(1A) | 125(2) |
| C(14B)-N(4)-Zr(1A) | 115.1(6) |
| C(16B)-N(4)-Zr(1A) | 116.3(8) |
| C(16A)-N(4)-Zr(1A) | 123.1(10) |
| Zr(1B)-N(4)-Zr(1A) | 2.1(4) |
| N(2)-Si(1)-C(2) | 107.7(2) |
| N(2)-Si(1)-C(3) | 109.8(3) |
| C(2)-Si(1)-C(3) | 109.1(3) |
| N(2)-Si(1)-C(1) | 111.5(3) |
| C(2)-Si(1)-C(1) | 108.9(3) |
| C(3)-Si(1)-C(1) | 109.8(3) |
| Zr(1B)-Cl(1)-Zr(1A) | 15.0(2) |
| Zr(1B)-Cl(2)-Zr(1A) | 14.9(2) |
| Zr(1B)-Zr(1A)-N(3) | 172.3(12) |
| Zr(1B)-Zr(1A)-N(2) | 90.4(12) |
| N(3)-Zr(1A)-N(2) | 96.7(2) |
| Zr(1B)-Zr(1A)-N(1) | 88.5(11) |
| N(3)-Zr(1A)-N(1) | 97.6(2) |
| N(2)-Zr(1A)-N(1) | 60.07(13) |
| Zr(1B)-Zr(1A)-Cl(1) | 78.0(11) |
| N(3)-Zr(1A)-Cl(1) | 97.20(14) |
| N(2)-Zr(1A)-Cl(1) | 104.58(11) |
| N(1)-Zr(1A)-Cl(1) | 159.80(12) |
| Zr(1B)-Zr(1A)-Cl(2) | 77.6(12) |
| N(3)-Zr(1A)-Cl(2) | 97.5(2) |
| N(2)-Zr(1A)-Cl(2) | 149.56(11) |
| N(1)-Zr(1A)-Cl(2) | 91.38(11) |
| Cl(1)-Zr(1A)-Cl(2) | 100.18(7) |
| Zr(1B)-Zr(1A)-N(4) | 6.2(11) |
| N(3)-Zr(1A)-N(4) | 177.9(2) |
| N(2)-Zr(1A)-N(4) | 85.4(2) |
| N(1)-Zr(1A)-N(4) | 82.9(2) |
| Cl(1)-Zr(1A)-N(4) | 82.75(14) |
| Cl(2)-Zr(1A)-N(4) | 80.41(13) |
| Zr(1B)-Zr(1A)-C(4) | 86.0(11) |
| N(3)-Zr(1A)-C(4) | 101.7(2) |

| | |
|---|---|
| N(1)-Zr(1A)-C(4) | 30.72(14) |
| Cl(1)-Zr(1A)-C(4) | 131.73(12) |
| Cl(2)-Zr(1A)-C(4) | 120.44(11) |
| N(4)-Zr(1A)-C(4) | 79.8(2) |
| Zr(1A)-Zr(1B)-N(4) | 171.8(14) |
| Zr(1A)-Zr(1B)-N(2) | 73.4(11) |
| N(4)-Zr(1B)-N(2) | 99.8(5) |
| Zr(1A)-Zr(1B)-Cl(1) | 87.0(11) |
| N(4)-Zr(1B)-Cl(1) | 99.3(5) |
| N(2)-Zr(1B)-Cl(1) | 103.3(5) |
| Zr(1A)-Zr(1B)-N(1) | 76.2(11) |
| N(4)-Zr(1B)-N(1) | 96.3(5) |
| N(2)-Zr(1B)-N(1) | 57.8(3) |
| Cl(1)-Zr(1B)-N(1) | 157.5(5) |
| Zr(1A)-Zr(1B)-Cl(2) | 87.5(12) |
| N(4)-Zr(1B)-Cl(2) | 96.1(4) |
| N(2)-Zr(1B)-Cl(2) | 146.2(5) |
| Cl(1)-Zr(1B)-Cl(2) | 103.2(4) |
| N(1)-Zr(1B)-Cl(2) | 91.1(4) |
| Zr(1A)-Zr(1B)-N(3) | 5.9(9) |
| N(4)-Zr(1B)-N(3) | 177.2(6) |
| N(2)-Zr(1B)-N(3) | 78.8(3) |
| Cl(1)-Zr(1B)-N(3) | 83.5(3) |
| N(1)-Zr(1B)-N(3) | 80.9(3) |
| Cl(2)-Zr(1B)-N(3) | 83.9(4) |
| Zr(1A)-Zr(1B)-C(4) | 80.6(11) |
| N(4)-Zr(1B)-C(4) | 91.2(5) |
| N(2)-Zr(1B)-C(4) | 29.5(2) |
| Cl(1)-Zr(1B)-C(4) | 132.7(5) |
| N(1)-Zr(1B)-C(4) | 30.3(2) |
| Cl(2)-Zr(1B)-C(4) | 121.4(5) |
| N(3)-Zr(1B)-C(4) | 86.5(3) |
| Zr(1A)-Zr(1B)-C(14B) | 151.2(14) |
| N(4)-Zr(1B)-C(14B) | 28.3(4) |
| N(2)-Zr(1B)-C(14B) | 85.1(5) |
| Cl(1)-Zr(1B)-C(14B) | 79.3(4) |
| N(1)-Zr(1B)-C(14B) | 108.5(5) |
| Cl(2)-Zr(1B)-C(14B) | 120.2(4) |
| N(3)-Zr(1B)-C(14B) | 153.0(6) |
| C(4)-Zr(1B)-C(14B) | 90.1(4) |
| Zr(1A)-Zr(1B)-C(14A) | 154(2) |
| N(4)-Zr(1B)-C(14A) | 22.1(8) |
| N(2)-Zr(1B)-C(14A) | 81.4(8) |
| Cl(1)-Zr(1B)-C(14A) | 91.9(8) |
| N(1)-Zr(1B)-C(14A) | 96.5(8) |
| Cl(2)-Zr(1B)-C(14A) | 118.1(8) |
| N(3)-Zr(1B)-C(14A) | 158.0(9) |
| C(4)-Zr(1B)-C(14A) | 80.9(8) |

---

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 2. 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 |
|---|---|---|---|---|---|---|
| C(1) | 64(3) | 57(4) | 108(6) | 23(4) | -17(3) | 3(3) |
| C(2) | 47(3) | 85(5) | 109(6) | -2(4) | 6(3) | 21(3) |
| C(3) | 96(5) | 58(4) | 118(7) | -23(4) | -13(4) | 12(3) |
| C(4) | 43(3) | 36(3) | 63(4) | 6(2) | 2(2) | -5(2) |
| C(5) | 40(3) | 45(3) | 100(5) | 17(3) | 5(3) | 2(2) |
| C(6) | 54(4) | 72(4) | 127(7) | 23(4) | 9(3) | -17(3) |
| C(7) | 43(3) | 75(5) | 141(7) | 7(4) | 9(3) | 14(3) |
| C(8) | 38(3) | 53(3) | 120(6) | 16(4) | -8(3) | 2(2) |
| C(9) | 71(4) | 67(5) | 228(12) | 46(6) | 17(5) | 38(4) |
| C(10) | 64(4) | 80(5) | 78(5) | 9(4) | -1(3) | -3(3) |
| C(11) | 86(5) | 142(8) | 102(8) | 14(6) | 19(4) | -14(5) |
| C(12) | 124(6) | 93(6) | 85(6) | 0(4) | 1(5) | -23(5) |
| C(13) | 116(7) | 168(10) | 133(10) | 49(8) | -49(6) | -43(7) |
| N(1) | 23(2) | 38(2) | 94(4) | 13(2) | 8(2) | 3(2) |
| N(2) | 26(2) | 38(2) | 67(3) | 5(2) | 3(2) | 2(1) |
| N(3) | 61(3) | 52(3) | 67(4) | 2(2) | 2(2) | -14(2) |
| N(4) | 83(4) | 72(4) | 59(4) | -3(3) | 14(3) | 10(3) |
| Si(1) | 53(1) | 35(1) | 75(1) | 1(1) | -2(1) | 8(1) |
| Cl(1) | 54(1) | 81(1) | 90(1) | 6(1) | -14(1) | -14(1) |
| Cl(2) | 95(1) | 33(1) | 107(2) | 3(1) | 4(1) | 7(1) |
| Zr(1A) | 40(1) | 32(1) | 63(1) | 5(1) | 1(1) | -2(1) |
| Zr(1B) | 53(4) | 36(3) | 60(7) | -7(5) | 3(5) | -5(2) |

Table 5. Hydrogen coordinates ( x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 2.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1A) | -48(6) | 4307(4) | 6549(5) | 116 |
| H(1B) | -1442(6) | 4691(4) | 6674(5) | 116 |
| H(1C) | -1335(6) | 3810(4) | 6277(5) | 116 |
| H(2A) | -3021(6) | 3147(4) | 7659(5) | 120 |
| H(2B) | -3130(6) | 4029(4) | 8054(5) | 120 |
| H(2C) | -2625(6) | 3301(4) | 8656(5) | 120 |
| H(3A) | 729(7) | 4603(4) | 8489(5) | 138 |
| H(3B) | -166(7) | 4258(4) | 9202(5) | 138 |
| H(3C) | -670(7) | 4986(4) | 8599(5) | 138 |
| H(5) | 1977(5) | 3863(3) | 7496(4) | 74 |
| H(6) | 4186(6) | 3529(4) | 7659(5) | 101 |
| H(7) | 4827(6) | 2246(4) | 8124(6) | 103 |
| H(9A) | 2596(7) | 657(4) | 8678(7) | 183 |
| H(9B) | 3908(7) | 1016(4) | 9129(7) | 183 |
| H(9C) | 3880(7) | 693(4) | 8161(7) | 183 |
| H(10A) | -1006(6) | 697(4) | 10189(5) | 89 |
| H(10B) | -1706(6) | 522(4) | 9262(5) | 89 |
| H(11A) | -3278(7) | 736(6) | 10267(6) | 164 |
| H(11B) | -3389(7) | 1432(6) | 9559(6) | 164 |
| H(11C) | -2689(7) | 1606(6) | 10487(6) | 164 |
| H(12A) | -1068(9) | 2354(5) | 10343(6) | 121 |
| H(12B) | -29(9) | 2685(5) | 9719(6) | 121 |
| H(13A) | 999(9) | 2330(7) | 11038(7) | 212 |
| H(13B) | 1504(9) | 1761(7) | 10305(7) | 212 |
| H(13C) | 462(9) | 1432(7) | 10933(7) | 212 |
| H(14A) | 546(34) | 2127(23) | 5627(25) | 249 |
| H(14B) | -661(34) | 2447(23) | 6126(25) | 249 |
| H(15A) | -1465(30) | 2007(18) | 4674(22) | 254 |
| H(15B) | -2080(30) | 1436(18) | 5364(22) | 254 |
| H(15C) | -877(30) | 1131(18) | 4858(22) | 254 |
| H(16A) | 1273(22) | 417(16) | 6608(18) | 173 |
| H(16B) | 613(22) | 499(16) | 5649(18) | 173 |
| H(17A) | 2792(33) | 948(20) | 5685(26) | 298 |
| H(17B) | 2494(33) | 1598(20) | 6401(26) | 298 |
| H(17C) | 1835(33) | 1682(20) | 5447(26) | 298 |
| H(14C) | -332(13) | 2243(8) | 5525(9) | 70 |
| H(14D) | -1424(13) | 2267(8) | 6208(9) | 70 |
| H(15D) | -2374(20) | 1665(14) | 4845(15) | 175 |
| H(15E) | -2383(20) | 1003(14) | 5587(15) | 175 |
| H(15F) | -1293(20) | 977(14) | 4906(15) | 175 |
| H(16C) | 1753(20) | 1894(13) | 6230(13) | 122 |
| H(16D) | 1902(20) | 1073(13) | 6754(13) | 122 |
| H(17D) | 2605(25) | 890(16) | 5363(18) | 236 |
| H(17E) | 1218(25) | 1165(16) | 4946(18) | 236 |
| H(17F) | 1356(25) | 341(16) | 5471(18) | 236 |
| HN4 | -1083(55) | 744(37) | 5816(45) | 85 |