Table 1. Crystal data and structure refinement for **3**.

| | | |
|---|---|---|
| Empirical formula | C22 H21 Mo N3 O3 Sn | |
| Formula weight | 590.05 | |
| Temperature | 293(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Rhombohedral | |
| Space group | R-3 | |
| Unit cell dimensions | a = 36.287(8) Å | α= 90°. |
| | b = 36.287(8) Å | β= 90°. |
| | c = 9.125(3) Å | γ = 120°. |
| Volume | 10406(5) $Å^3$ | |
| Z | 18 | |
| Density (calculated) | 1.695 $Mg/m^3$ | |
| Absorption coefficient | 1.649 $mm^{-1}$ | |
| F(000) | 5220 | |
| Crystal size | 0.03 x 0.05 x 0.1 $mm^3$ | |
| Theta range for data collection | 1.12 to 23.37°. | |
| Index ranges | -40<=h<=32, -40<=k<=40, -9<=l<=10 | |
| Reflections collected | 15837 | |
| Independent reflections | 3360 [R(int) = 0.0740] | |
| Completeness to theta = 23.37° | 99.6 % | |
| Absorption correction | SADABS | |
| Max. and min. transmission | 1.000 and 0.8118 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 3360 / 0 / 272 | |
| Goodness-of-fit on $F^2$ | 1.000 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0471, wR2 = 0.1350 | |
| R indices (all data) | R1 = 0.0853, wR2 = 0.1571 | |
| Largest diff. peak and hole | 2.656 and -0.534 $e.Å^{-3}$ | |

Table 2. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) For **3**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Mo(1) | 7517(1) | 2151(1) | 6037(1) | 47(1) |
| Sn(1) | 8165(1) | 2421(1) | 9367(1) | 49(1) |
| C(1) | 6957(4) | 1722(4) | 5671(12) | 57(3) |
| O(1) | 6595(3) | 1455(3) | 5393(10) | 87(3) |
| C(2) | 7644(4) | 2001(4) | 4147(12) | 52(3) |
| O(2) | 7709(3) | 1890(3) | 3017(9) | 76(3) |
| C(3) | 7335(4) | 2506(4) | 5018(12) | 58(3) |
| O(3) | 7205(3) | 2694(3) | 4405(9) | 88(3) |
| C(4) | 8554(4) | 2469(4) | 11176(14) | 74(4) |
| C(5) | 9011(7) | 2691(7) | 10860(30) | 191(14) |
| C(6) | 9212(10) | 2545(10) | 10760(50) | 340(30) |
| C(7) | 9708(7) | 2763(8) | 10500(30) | 223(12) |
| N(1) | 7339(3) | 2310(3) | 8321(9) | 46(2) |
| N(2) | 8198(3) | 2741(3) | 6351(10) | 48(2) |
| N(3) | 7710(3) | 1708(3) | 7218(9) | 48(2) |
| C(11) | 7572(3) | 2417(3) | 9586(12) | 45(3) |
| C(12) | 7432(4) | 2516(4) | 10844(12) | 56(3) |
| C(13) | 7058(4) | 2514(4) | 10895(14) | 74(4) |
| C(14) | 6819(4) | 2402(4) | 9622(13) | 65(4) |
| C(15) | 6966(4) | 2305(4) | 8412(13) | 57(3) |
| C(21) | 8428(3) | 2856(3) | 7566(12) | 46(3) |
| C(22) | 8805(4) | 3249(4) | 7680(13) | 66(3) |
| C(23) | 8953(4) | 3524(4) | 6477(16) | 80(4) |
| C(24) | 8716(5) | 3390(4) | 5209(14) | 74(4) |
| C(25) | 8347(4) | 3005(4) | 5198(13) | 61(3) |
| C(31) | 7974(3) | 1813(3) | 8370(11) | 47(3) |
| C(32) | 8096(4) | 1544(4) | 8921(13) | 61(3) |
| C(33) | 7952(4) | 1161(5) | 8344(14) | 70(4) |
| C(34) | 7673(4) | 1036(4) | 7202(13) | 65(3) |
| C(35) | 7556(4) | 1312(4) | 6654(12) | 60(3) |

Table 3. Bond lengths [Å] and angles [°] for **3**.

| | |
|---|---|
| Mo(1)-C(1) | 1.872(13) |
| Mo(1)-C(2) | 1.932(12) |
| Mo(1)-C(3) | 1.952(13) |
| Mo(1)-N(3) | 2.315(9) |
| Mo(1)-N(1) | 2.339(8) |
| Mo(1)-N(2) | 2.342(9) |
| Sn(1)-C(4) | 2.124(11) |
| Sn(1)-C(21) | 2.144(10) |
| Sn(1)-C(11) | 2.154(10) |
| Sn(1)-C(31) | 2.157(11) |
| C(1)-O(1) | 1.206(13) |
| C(2)-O(2) | 1.173(12) |
| C(3)-O(3) | 1.151(12) |
| C(4)-C(5) | 1.46(2) |
| C(5)-C(6) | 1.10(3) |
| C(6)-C(7) | 1.58(3) |
| N(1)-C(15) | 1.346(13) |
| N(1)-C(11) | 1.366(12) |
| N(2)-C(21) | 1.323(12) |
| N(2)-C(25) | 1.341(13) |
| N(3)-C(31) | 1.343(12) |
| N(3)-C(35) | 1.356(13) |
| C(11)-C(12) | 1.373(14) |
| C(12)-C(13) | 1.357(15) |
| C(13)-C(14) | 1.383(15) |
| C(14)-C(15) | 1.348(14) |
| C(21)-C(22) | 1.401(15) |
| C(22)-C(23) | 1.397(16) |
| C(23)-C(24) | 1.377(17) |
| C(24)-C(25) | 1.369(16) |
| C(31)-C(32) | 1.352(14) |
| C(32)-C(33) | 1.325(16) |
| C(33)-C(34) | 1.362(16) |
| C(34)-C(35) | 1.364(15) |

| | |
|---|---|
| C(1)-Mo(1)-C(2) | 85.4(5) |
| C(1)-Mo(1)-C(3) | 83.0(5) |
| C(2)-Mo(1)-C(3) | 88.1(5) |
| C(1)-Mo(1)-N(3) | 94.9(4) |
| C(2)-Mo(1)-N(3) | 91.3(4) |
| C(3)-Mo(1)-N(3) | 177.9(4) |
| C(1)-Mo(1)-N(1) | 92.6(4) |
| C(2)-Mo(1)-N(1) | 177.9(4) |
| C(3)-Mo(1)-N(1) | 92.0(4) |
| N(3)-Mo(1)-N(1) | 88.5(3) |
| C(1)-Mo(1)-N(2) | 173.4(4) |
| C(2)-Mo(1)-N(2) | 94.3(4) |
| C(3)-Mo(1)-N(2) | 90.3(4) |
| N(3)-Mo(1)-N(2) | 91.8(3) |
| N(1)-Mo(1)-N(2) | 87.8(3) |
| C(4)-Sn(1)-C(21) | 120.1(5) |
| C(4)-Sn(1)-C(11) | 123.4(5) |
| C(21)-Sn(1)-C(11) | 98.7(4) |
| C(4)-Sn(1)-C(31) | 105.7(4) |
| C(21)-Sn(1)-C(31) | 103.2(4) |
| C(11)-Sn(1)-C(31) | 103.0(4) |
| O(1)-C(1)-Mo(1) | 177.4(10) |
| O(2)-C(2)-Mo(1) | 176.7(11) |
| O(3)-C(3)-Mo(1) | 176.0(11) |
| C(5)-C(4)-Sn(1) | 114.6(12) |
| C(6)-C(5)-C(4) | 126(3) |
| C(5)-C(6)-C(7) | 130(3) |
| C(15)-N(1)-C(11) | 115.2(9) |
| C(15)-N(1)-Mo(1) | 116.8(7) |
| C(11)-N(1)-Mo(1) | 127.9(7) |
| C(21)-N(2)-C(25) | 117.8(9) |
| C(21)-N(2)-Mo(1) | 127.0(7) |
| C(25)-N(2)-Mo(1) | 114.9(8) |
| C(31)-N(3)-C(35) | 117.0(9) |
| C(31)-N(3)-Mo(1) | 126.0(7) |

| | |
|---|---|
| C(35)-N(3)-Mo(1) | 116.9(8) |
| N(1)-C(11)-C(12) | 121.6(10) |
| N(1)-C(11)-Sn(1) | 113.4(7) |
| C(12)-C(11)-Sn(1) | 125.0(8) |
| C(13)-C(12)-C(11) | 121.8(11) |
| C(12)-C(13)-C(14) | 116.8(11) |
| C(15)-C(14)-C(13) | 119.6(11) |
| N(1)-C(15)-C(14) | 124.9(11) |
| N(2)-C(21)-C(22) | 121.6(10) |
| N(2)-C(21)-Sn(1) | 115.5(7) |
| C(22)-C(21)-Sn(1) | 122.8(9) |
| C(23)-C(22)-C(21) | 119.9(11) |
| C(24)-C(23)-C(22) | 117.4(12) |
| C(25)-C(24)-C(23) | 119.0(12) |
| N(2)-C(25)-C(24) | 124.3(12) |
| N(3)-C(31)-C(32) | 122.1(10) |
| N(3)-C(31)-Sn(1) | 116.0(7) |
| C(32)-C(31)-Sn(1) | 121.9(9) |
| C(33)-C(32)-C(31) | 120.6(12) |
| C(32)-C(33)-C(34) | 119.4(12) |
| C(33)-C(34)-C(35) | 119.1(12) |
| N(3)-C(35)-C(34) | 121.7(11) |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Mo(1) | 49(1) | 50(1) | 42(1) | -1(1) | -5(1) | 26(1) |
| Sn(1) | 46(1) | 50(1) | 49(1) | 3(1) | -6(1) | 22(1) |
| C(1) | 58(8) | 74(9) | 46(7) | 1(6) | -3(6) | 37(7) |
| O(1) | 60(6) | 89(7) | 85(6) | -13(5) | -18(5) | 17(6) |
| C(2) | 50(7) | 58(8) | 44(7) | 6(6) | -8(6) | 24(6) |
| O(2) | 84(7) | 102(7) | 46(5) | -14(5) | -12(5) | 49(6) |
| C(3) | 70(8) | 60(8) | 44(7) | -13(6) | -19(6) | 32(7) |
| O(3) | 131(9) | 103(7) | 62(6) | -6(5) | -30(6) | 82(7) |
| C(4) | 84(10) | 77(9) | 74(9) | -13(7) | -42(8) | 49(8) |
| C(5) | 98(17) | 120(19) | 310(40) | 40(20) | -120(20) | 23(14) |
| C(6) | 190(30) | 200(30) | 630(80) | 110(40) | 250(40) | 110(30) |
| C(7) | 160(20) | 250(30) | 250(30) | 50(30) | 50(20) | 100(20) |
| N(1) | 46(6) | 46(6) | 44(5) | 0(4) | -5(4) | 21(5) |
| N(2) | 57(6) | 41(5) | 49(6) | 2(5) | 6(5) | 27(5) |
| N(3) | 51(6) | 43(6) | 47(6) | 5(4) | 11(5) | 21(5) |
| C(11) | 45(6) | 36(6) | 50(7) | 3(5) | -6(6) | 17(5) |
| C(12) | 61(8) | 63(8) | 40(7) | 3(6) | 0(6) | 27(7) |
| C(13) | 69(9) | 101(11) | 56(8) | -7(7) | 0(7) | 44(9) |
| C(14) | 59(8) | 97(10) | 53(8) | -7(7) | 9(7) | 49(8) |
| C(15) | 46(7) | 65(8) | 61(8) | 3(6) | -13(6) | 29(7) |
| C(21) | 50(7) | 34(6) | 49(7) | 2(5) | 4(6) | 18(6) |
| C(22) | 70(9) | 67(9) | 54(8) | -5(7) | -3(7) | 29(7) |
| C(23) | 90(11) | 44(8) | 86(10) | 8(8) | 21(9) | 19(7) |
| C(24) | 102(11) | 63(9) | 57(8) | 20(7) | 17(8) | 42(9) |
| C(25) | 79(9) | 41(7) | 61(8) | 12(6) | 10(7) | 27(7) |
| C(31) | 41(6) | 53(7) | 45(7) | 0(5) | -1(5) | 22(6) |
| C(32) | 70(9) | 53(8) | 72(9) | 7(7) | -3(7) | 39(7) |
| C(33) | 80(10) | 91(11) | 63(9) | 16(8) | 1(7) | 62(9) |
| C(34) | 90(10) | 49(8) | 67(8) | 2(7) | 13(8) | 44(8) |
| C(35) | 73(9) | 55(8) | 55(8) | -1(6) | 1(6) | 34(7) |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(4A) | 8503 | 2617 | 11966 | 89 |
| H(4B) | 8469 | 2184 | 11519 | 89 |
| H(5A) | 9046 | 2843 | 9951 | 229 |
| H(5B) | 9143 | 2906 | 11616 | 229 |
| H(6A) | 9085 | 2341 | 9964 | 404 |
| H(6B) | 9158 | 2378 | 11641 | 404 |
| H(7A) | 9821 | 3057 | 10248 | 334 |
| H(7B) | 9763 | 2621 | 9711 | 334 |
| H(7C) | 9843 | 2742 | 11375 | 334 |
| H(12) | 7599 | 2585 | 11685 | 68 |
| H(13) | 6966 | 2585 | 11745 | 89 |
| H(14) | 6559 | 2394 | 9601 | 78 |
| H(15) | 6797 | 2228 | 7576 | 69 |
| H(22) | 8956 | 3327 | 8555 | 79 |
| H(23) | 9201 | 3787 | 6531 | 96 |
| H(24) | 8805 | 3559 | 4372 | 88 |
| H(25) | 8190 | 2921 | 4336 | 74 |
| H(32) | 8282 | 1629 | 9712 | 73 |
| H(33) | 8040 | 979 | 8716 | 84 |
| H(34) | 7564 | 765 | 6801 | 78 |
| H(35) | 7366 | 1227 | 5874 | 72 |

Table 6. Crystal data and structure refinement for **5**.

| | | |
|---|---|---|
| Empirical formula | C13 H24 Mo O3 S3 Sn | |
| Formula weight | 539.13 | |
| Temperature | 293(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P2(1)/n | |
| Unit cell dimensions | a = 8.7421(4) Å | α= 90°. |
| | b = 17.3956(9) Å | β= 92.5430(10)°. |
| | c = 13.4689(7) Å | γ = 90°. |
| Volume | 2046.25(18) Å$^3$ | |
| Z | 4 | |
| Density (calculated) | 1.750 Mg/m$^3$ | |
| Absorption coefficient | 2.144 mm$^{-1}$ | |
| F(000) | 1064 | |
| Crystal size | 0.12 x 0.14 x 0.21 mm$^3$ | |
| Theta range for data collection | 1.91 to 23.27°. | |
| Index ranges | -9<=h<=9, -19<=k<=11, -14<=l<=14 | |
| Reflections collected | 9156 | |
| Independent reflections | 2944 [R(int) = 0.0240] | |
| Completeness to theta = 23.27∞ | 99.9 % | |
| Absorption correction | SADABS | |
| Max. and min. transmission | 1.0000 and 0.5949 | |
| Refinement method | Full-matrix least-squares on F$^2$ | |
| Data / restraints / parameters | 2944 / 0 / 195 | |
| Goodness-of-fit on F$^2$ | 1.039 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0297, wR2 = 0.0719 | |
| R indices (all data) | R1 = 0.0355, wR2 = 0.0755 | |
| Extinction coefficient | 0.00058(16) | |
| Largest diff. peak and hole | 1.268 and -0.948 e. Å$^{-3}$ | |

Table 7. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters ($Å^2$x $10^3$) For **5**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Mo(1) | 1407(1) | 1847(1) | 2837(1) | 47(1) |
| Sn(1) | 1509(1) | 1560(1) | 5789(1) | 74(1) |
| S(1) | 2108(2) | 603(1) | 3796(1) | 62(1) |
| S(2) | -918(2) | 1956(1) | 3968(1) | 67(1) |
| S(3) | 2961(1) | 2657(1) | 4145(1) | 56(1) |
| C(1) | 753(6) | 2712(3) | 2021(4) | 64(1) |
| O(1) | 414(5) | 3210(2) | 1472(3) | 92(1) |
| C(2) | 3219(6) | 1853(3) | 2059(4) | 63(1) |
| O(2) | 4287(5) | 1881(3) | 1588(3) | 108(2) |
| C(3) | 434(6) | 1210(3) | 1806(4) | 71(1) |
| O(3) | -80(6) | 838(3) | 1156(3) | 116(2) |
| C(4) | 2981(6) | 782(3) | 5007(4) | 72(1) |
| C(5) | -723(6) | 1378(4) | 5066(4) | 82(2) |
| C(6) | 2158(6) | 2694(3) | 5339(4) | 70(1) |
| C(7) | 3628(8) | 133(3) | 3172(4) | 92(2) |
| C(8) | -2621(7) | 1545(4) | 3362(6) | 109(2) |
| C(9) | 2813(7) | 3658(3) | 3804(5) | 85(2) |
| C(11) | 1506(13) | 1360(6) | 7350(5) | 153(4) |
| C(12) | 2416(11) | 845(6) | 7819(5) | 144(3) |
| C(13) | 2398(16) | 796(7) | 8937(7) | 216(7) |
| C(14) | 3317(17) | 469(10) | 9464(9) | 269(9) |

Table 8. Bond lengths [Å] and angles [°] for **5**.

| | |
|---|---|
| Mo(1)-C(1) | 1.935(5) |
| Mo(1)-C(2) | 1.938(6) |
| Mo(1)-C(3) | 1.943(5) |
| Mo(1)-S(1) | 2.5804(12) |
| Mo(1)-S(3) | 2.5924(12) |
| Mo(1)-S(2) | 2.5995(14) |
| Sn(1)-C(11) | 2.132(6) |
| Sn(1)-C(6) | 2.146(5) |
| Sn(1)-C(5) | 2.165(6) |
| Sn(1)-C(4) | 2.173(5) |
| S(1)-C(4) | 1.797(5) |
| S(1)-C(7) | 1.799(6) |
| S(2)-C(5) | 1.791(5) |
| S(2)-C(8) | 1.814(6) |
| S(3)-C(6) | 1.785(5) |
| S(3)-C(9) | 1.804(5) |
| C(1)-O(1) | 1.168(6) |
| C(2)-O(2) | 1.153(6) |
| C(3)-O(3) | 1.163(6) |
| C(11)-C(12) | 1.337(10) |
| C(12)-C(13) | 1.510(11) |
| C(13)-C(14) | 1.191(16) |
| | |
| C(1)-Mo(1)-C(2) | 85.2(2) |
| C(1)-Mo(1)-C(3) | 85.9(2) |
| C(2)-Mo(1)-C(3) | 87.6(2) |
| C(1)-Mo(1)-S(1) | 173.91(15) |
| C(2)-Mo(1)-S(1) | 95.34(16) |
| C(3)-Mo(1)-S(1) | 88.04(15) |
| C(1)-Mo(1)-S(3) | 95.49(15) |
| C(2)-Mo(1)-S(3) | 87.04(15) |
| C(3)-Mo(1)-S(3) | 174.35(18) |
| S(1)-Mo(1)-S(3) | 90.60(4) |
| C(1)-Mo(1)-S(2) | 93.26(16) |

| | |
|---|---|
| C(2)-Mo(1)-S(2) | 174.49(15) |
| C(3)-Mo(1)-S(2) | 97.56(18) |
| S(1)-Mo(1)-S(2) | 86.73(4) |
| S(3)-Mo(1)-S(2) | 87.83(4) |
| C(11)-Sn(1)-C(6) | 116.1(3) |
| C(11)-Sn(1)-C(5) | 112.3(3) |
| C(6)-Sn(1)-C(5) | 104.6(2) |
| C(11)-Sn(1)-C(4) | 113.8(3) |
| C(6)-Sn(1)-C(4) | 105.5(2) |
| C(5)-Sn(1)-C(4) | 103.4(2) |
| C(4)-S(1)-C(7) | 102.1(3) |
| C(4)-S(1)-Mo(1) | 112.98(18) |
| C(7)-S(1)-Mo(1) | 108.1(2) |
| C(5)-S(2)-C(8) | 101.3(3) |
| C(5)-S(2)-Mo(1) | 113.3(2) |
| C(8)-S(2)-Mo(1) | 110.8(3) |
| C(6)-S(3)-C(9) | 99.7(3) |
| C(6)-S(3)-Mo(1) | 114.61(18) |
| C(9)-S(3)-Mo(1) | 108.8(2) |
| O(1)-C(1)-Mo(1) | 175.0(5) |
| O(2)-C(2)-Mo(1) | 177.7(5) |
| O(3)-C(3)-Mo(1) | 176.2(5) |
| S(1)-C(4)-Sn(1) | 108.2(2) |
| S(2)-C(5)-Sn(1) | 109.9(3) |
| S(3)-C(6)-Sn(1) | 109.9(3) |
| C(12)-C(11)-Sn(1) | 123.3(6) |
| C(11)-C(12)-C(13) | 118.5(10) |
| C(14)-C(13)-C(12) | 125.8(17) |

Symmetry transformations used to generate equivalent atoms:

Table 9. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| Mo(1) | 49(1) | 49(1) | 44(1) | 4(1) | 0(1) | -3(1) |
| Sn(1) | 84(1) | 93(1) | 47(1) | 12(1) | 10(1) | 3(1) |
| S(1) | 70(1) | 50(1) | 64(1) | 6(1) | -8(1) | -1(1) |
| S(2) | 52(1) | 74(1) | 75(1) | 11(1) | 7(1) | 0(1) |
| S(3) | 54(1) | 60(1) | 55(1) | -4(1) | 4(1) | -2(1) |
| C(1) | 70(3) | 61(3) | 60(3) | 4(3) | -4(2) | -2(2) |
| O(1) | 117(3) | 68(2) | 87(3) | 27(2) | -21(2) | 1(2) |
| C(2) | 65(3) | 76(3) | 49(3) | 1(2) | 4(3) | -7(3) |
| O(2) | 86(3) | 159(5) | 82(3) | -2(3) | 35(3) | -13(3) |
| C(3) | 82(4) | 65(3) | 65(3) | 5(3) | -13(3) | -10(3) |
| O(3) | 147(4) | 109(4) | 90(3) | -22(3) | -35(3) | -30(3) |
| C(4) | 83(4) | 71(3) | 63(3) | 12(3) | -10(3) | 9(3) |
| C(5) | 71(4) | 96(4) | 81(4) | 22(3) | 21(3) | -8(3) |
| C(6) | 75(3) | 80(4) | 55(3) | -10(3) | 7(3) | 2(3) |
| C(7) | 118(5) | 66(4) | 91(4) | -18(3) | -6(4) | 24(3) |
| C(8) | 57(4) | 147(7) | 123(6) | 13(5) | -9(4) | -25(4) |
| C(9) | 97(4) | 63(4) | 93(4) | 2(3) | 0(3) | -9(3) |
| C(11) | 219(10) | 185(9) | 59(4) | 41(5) | 34(5) | 67(8) |
| C(12) | 160(8) | 187(9) | 86(5) | 58(6) | 18(5) | 10(7) |
| C(13) | 291(16) | 225(13) | 123(8) | 99(8) | -101(9) | -119(12) |
| C(14) | 284(19) | 360(20) | 157(11) | 99(13) | -43(11) | -138(17) |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(4A) | 3984 | 1010 | 4945 | 87 |
| H(4B) | 3102 | 303 | 5371 | 87 |
| H(5A) | -845 | 840 | 4892 | 98 |
| H(5B) | -1515 | 1514 | 5517 | 98 |
| H(6A) | 1267 | 3027 | 5314 | 84 |
| H(6B) | 2902 | 2905 | 5821 | 84 |
| H(7A) | 4501 | 467 | 3165 | 138 |
| H(7B) | 3289 | 14 | 2502 | 138 |
| H(7C) | 3904 | -333 | 3517 | 138 |
| H(8A) | -2471 | 1004 | 3266 | 164 |
| H(8B) | -2815 | 1789 | 2729 | 164 |
| H(8C) | -3480 | 1624 | 3770 | 164 |
| H(9A) | 1763 | 3818 | 3816 | 127 |
| H(9B) | 3174 | 3728 | 3147 | 127 |
| H(9C) | 3423 | 3961 | 4267 | 127 |
| H(11A) | 1714 | 1851 | 7669 | 184 |
| H(11B) | 464 | 1223 | 7498 | 184 |
| H(12A) | 3460 | 953 | 7645 | 173 |
| H(12B) | 2154 | 342 | 7553 | 173 |
| H(13A) | 2383 | 1322 | 9173 | 259 |
| H(13B) | 1413 | 576 | 9083 | 259 |
| H(14A) | 3476 | -39 | 9212 | 404 |
| H(14B) | 2948 | 438 | 10123 | 404 |
| H(14C) | 4266 | 747 | 9480 | 404 |

Table 11. Torsion angles [°] for **5**.

| | |
|---|---|
| C(1)-Mo(1)-S(1)-C(4) | -166.8(15) |
| C(2)-Mo(1)-S(1)-C(4) | 98.1(3) |
| C(3)-Mo(1)-S(1)-C(4) | -174.4(3) |
| S(3)-Mo(1)-S(1)-C(4) | 11.0(2) |
| S(2)-Mo(1)-S(1)-C(4) | -76.8(2) |
| C(1)-Mo(1)-S(1)-C(7) | 80.9(15) |
| C(2)-Mo(1)-S(1)-C(7) | -14.1(3) |
| C(3)-Mo(1)-S(1)-C(7) | 73.3(3) |
| S(3)-Mo(1)-S(1)-C(7) | -101.2(2) |
| S(2)-Mo(1)-S(1)-C(7) | 171.0(2) |
| C(1)-Mo(1)-S(2)-C(5) | -168.4(3) |
| C(2)-Mo(1)-S(2)-C(5) | -94.5(16) |
| C(3)-Mo(1)-S(2)-C(5) | 105.3(3) |
| S(1)-Mo(1)-S(2)-C(5) | 17.8(2) |
| S(3)-Mo(1)-S(2)-C(5) | -73.0(2) |
| C(1)-Mo(1)-S(2)-C(8) | 78.6(3) |
| C(2)-Mo(1)-S(2)-C(8) | 152.5(16) |
| C(3)-Mo(1)-S(2)-C(8) | -7.7(3) |
| S(1)-Mo(1)-S(2)-C(8) | -95.3(3) |
| S(3)-Mo(1)-S(2)-C(8) | 174.0(3) |
| C(1)-Mo(1)-S(3)-C(6) | 111.1(3) |
| C(2)-Mo(1)-S(3)-C(6) | -164.0(3) |
| C(3)-Mo(1)-S(3)-C(6) | -144.6(16) |
| S(1)-Mo(1)-S(3)-C(6) | -68.7(2) |
| S(2)-Mo(1)-S(3)-C(6) | 18.0(2) |
| C(1)-Mo(1)-S(3)-C(9) | 0.6(3) |
| C(2)-Mo(1)-S(3)-C(9) | 85.5(3) |
| C(3)-Mo(1)-S(3)-C(9) | 104.8(16) |
| S(1)-Mo(1)-S(3)-C(9) | -179.2(2) |
| S(2)-Mo(1)-S(3)-C(9) | -92.5(2) |
| C(2)-Mo(1)-C(1)-O(1) | 33(5) |
| C(3)-Mo(1)-C(1)-O(1) | -55(5) |
| S(1)-Mo(1)-C(1)-O(1) | -63(6) |
| S(3)-Mo(1)-C(1)-O(1) | 119(5) |

| | |
|---|---|
| S(2)-Mo(1)-C(1)-O(1) | -152(5) |
| C(1)-Mo(1)-C(2)-O(2) | 22(12) |
| C(3)-Mo(1)-C(2)-O(2) | 108(12) |
| S(1)-Mo(1)-C(2)-O(2) | -165(12) |
| S(3)-Mo(1)-C(2)-O(2) | -74(12) |
| S(2)-Mo(1)-C(2)-O(2) | -53(13) |
| C(1)-Mo(1)-C(3)-O(3) | 74(8) |
| C(2)-Mo(1)-C(3)-O(3) | -11(8) |
| S(1)-Mo(1)-C(3)-O(3) | -107(8) |
| S(3)-Mo(1)-C(3)-O(3) | -31(9) |
| S(2)-Mo(1)-C(3)-O(3) | 167(8) |
| C(7)-S(1)-C(4)-Sn(1) | 168.2(3) |
| Mo(1)-S(1)-C(4)-Sn(1) | 52.3(3) |
| C(11)-Sn(1)-C(4)-S(1) | 146.2(4) |
| C(6)-Sn(1)-C(4)-S(1) | -85.4(3) |
| C(5)-Sn(1)-C(4)-S(1) | 24.1(3) |
| C(8)-S(2)-C(5)-Sn(1) | 167.2(4) |
| Mo(1)-S(2)-C(5)-Sn(1) | 48.5(3) |
| C(11)-Sn(1)-C(5)-S(2) | 152.8(4) |
| C(6)-Sn(1)-C(5)-S(2) | 26.0(4) |
| C(4)-Sn(1)-C(5)-S(2) | -84.2(3) |
| C(9)-S(3)-C(6)-Sn(1) | 162.6(3) |
| Mo(1)-S(3)-C(6)-Sn(1) | 46.7(3) |
| C(11)-Sn(1)-C(6)-S(3) | 152.8(4) |
| C(5)-Sn(1)-C(6)-S(3) | -82.8(3) |
| C(4)-Sn(1)-C(6)-S(3) | 25.9(3) |
| C(6)-Sn(1)-C(11)-C(12) | -117.5(10) |
| C(5)-Sn(1)-C(11)-C(12) | 122.2(10) |
| C(4)-Sn(1)-C(11)-C(12) | 5.2(11) |
| Sn(1)-C(11)-C(12)-C(13) | 175.5(7) |
| C(11)-C(12)-C(13)-C(14) | -166.1(15) |

Symmetry transformations used to generate equivalent atoms:

Table 12. Crystal data and structure refinement for **6**.

| | | |
|---|---|---|
| Empirical formula | C13 H24 O3 S3 Sn W | |
| Formula weight | 627.04 | |
| Temperature | 299(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Monoclinic | |
| Space group | P2(1)/n | |
| Unit cell dimensions | a = 14.1071(10) Å | α= 90°. |
| | b = 10.0682(7) Å | β= 113.7970(10)°. |
| | c = 15.4943(12) Å | γ = 90°. |
| Volume | 2013.6(3) $Å^3$ | |
| Z | 4 | |
| Density (calculated) | 2.068 $Mg/m^3$ | |
| Absorption coefficient | 7.264 $mm^{-1}$ | |
| F(000) | 1192 | |
| Crystal size | 0.14 x 0.16 x 0.17 $mm^3$ | |
| Theta range for data collection | 1.65 to 23.30°. | |
| Index ranges | -15<=h<=12, -11<=k<=11, -17<=l<=17 | |
| Reflections collected | 9007 | |
| Independent reflections | 2904 [R(int) = 0.0300] | |
| Completeness to theta = 23.30∞ | 99.8 % | |
| Absorption correction | SADABS | |
| Max. and min. transmission | 1.0000 and 0.4904 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 2904 / 0 / 195 | |
| Goodness-of-fit on $F^2$ | 1.043 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0268, wR2 = 0.0656 | |
| R indices (all data) | R1 = 0.0313, wR2 = 0.0689 | |
| Extinction coefficient | 0.00267(13) | |
| Largest diff. peak and hole | 1.079 and -0.668 e. $Å^{-3}$ | |

Table 13. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters (Å $^2$x $10^3$) For **6**. U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| W(1) | 9277(1) | 7285(1) | 1493(1) | 59(1) |
| Sn(1) | 7295(1) | 9956(1) | -229(1) | 73(1) |
| S(1) | 8789(1) | 7588(2) | -274(1) | 68(1) |
| S(2) | 7341(1) | 7594(2) | 1184(2) | 75(1) |
| S(3) | 9409(1) | 9825(2) | 1669(1) | 70(1) |
| C(1) | 9736(5) | 7037(7) | 2848(5) | 74(2) |
| O(1) | 10017(4) | 6803(7) | 3649(4) | 104(2) |
| C(2) | 9138(5) | 5368(9) | 1427(5) | 76(2) |
| O(2) | 9095(4) | 4192(6) | 1423(4) | 104(2) |
| C(3) | 10683(5) | 7088(7) | 1629(4) | 69(2) |
| O(3) | 11553(4) | 6973(6) | 1710(4) | 100(2) |
| C(4) | 7503(5) | 8189(7) | -905(5) | 75(2) |
| C(5) | 6843(5) | 9242(7) | 860(5) | 80(2) |
| C(6) | 8854(5) | 10714(7) | 564(5) | 78(2) |
| C(11) | 6276(8) | 11309(9) | -1227(7) | 117(3) |
| C(12) | 5871(14) | 12373(17) | -845(16) | 215(9) |
| C(13) | 6427(17) | 13126(17) | -138(15) | 259(13) |
| C(14) | 6223(12) | 14362(16) | 187(17) | 280(12) |
| C(21) | 8719(7) | 6009(9) | -837(6) | 105(3) |
| C(22) | 7193(8) | 7356(11) | 2272(8) | 140(4) |
| C(23) | 10762(6) | 10303(9) | 2073(5) | 97(2) |

Table 14. Bond lengths [Å] and angles [°] for **6**.

| | |
|---|---|
| W(1)-C(3) | 1.918(7) |
| W(1)-C(2) | 1.938(9) |
| W(1)-C(1) | 1.948(8) |
| W(1)-S(1) | 2.5570(17) |
| W(1)-S(3) | 2.5701(18) |
| W(1)-S(2) | 2.5946(18) |
| Sn(1)-C(11) | 2.126(8) |
| Sn(1)-C(4) | 2.144(7) |
| Sn(1)-C(5) | 2.154(7) |
| Sn(1)-C(6) | 2.180(6) |
| S(1)-C(4) | 1.784(7) |
| S(1)-C(21) | 1.797(8) |
| S(2)-C(5) | 1.793(7) |
| S(2)-C(22) | 1.797(9) |
| S(3)-C(6) | 1.806(7) |
| S(3)-C(23) | 1.818(7) |
| C(1)-O(1) | 1.164(8) |
| C(2)-O(2) | 1.186(9) |
| C(3)-O(3) | 1.189(8) |
| C(11)-C(12) | 1.45(2) |
| C(12)-C(13) | 1.30(2) |
| C(13)-C(14) | 1.41(2) |
| | |
| C(3)-W(1)-C(2) | 88.9(3) |
| C(3)-W(1)-C(1) | 89.4(3) |
| C(2)-W(1)-C(1) | 84.9(3) |
| C(3)-W(1)-S(1) | 87.15(19) |
| C(2)-W(1)-S(1) | 94.9(2) |
| C(1)-W(1)-S(1) | 176.5(2) |
| C(3)-W(1)-S(3) | 93.7(2) |
| C(2)-W(1)-S(3) | 175.9(2) |
| C(1)-W(1)-S(3) | 92.0(2) |
| S(1)-W(1)-S(3) | 88.37(6) |
| C(3)-W(1)-S(2) | 175.9(2) |

| | |
|---|---|
| C(2)-W(1)-S(2) | 91.8(2) |
| C(1)-W(1)-S(2) | 94.7(2) |
| S(1)-W(1)-S(2) | 88.81(6) |
| S(3)-W(1)-S(2) | 85.75(6) |
| C(11)-Sn(1)-C(4) | 111.2(3) |
| C(11)-Sn(1)-C(5) | 117.2(4) |
| C(4)-Sn(1)-C(5) | 104.4(3) |
| C(11)-Sn(1)-C(6) | 114.7(3) |
| C(4)-Sn(1)-C(6) | 105.1(3) |
| C(5)-Sn(1)-C(6) | 103.0(3) |
| C(4)-S(1)-C(21) | 100.9(4) |
| C(4)-S(1)-W(1) | 113.8(2) |
| C(21)-S(1)-W(1) | 110.6(3) |
| C(5)-S(2)-C(22) | 101.8(4) |
| C(5)-S(2)-W(1) | 115.3(2) |
| C(22)-S(2)-W(1) | 108.9(3) |
| C(6)-S(3)-C(23) | 100.9(3) |
| C(6)-S(3)-W(1) | 114.1(2) |
| C(23)-S(3)-W(1) | 108.7(3) |
| O(1)-C(1)-W(1) | 175.6(7) |
| O(2)-C(2)-W(1) | 176.9(6) |
| O(3)-C(3)-W(1) | 179.5(7) |
| S(1)-C(4)-Sn(1) | 108.8(3) |
| S(2)-C(5)-Sn(1) | 108.8(3) |
| S(3)-C(6)-Sn(1) | 108.3(3) |
| C(12)-C(11)-Sn(1) | 116.1(9) |
| C(13)-C(12)-C(11) | 124.8(17) |
| C(12)-C(13)-C(14) | 132(2) |

Symmetry transformations used to generate equivalent atoms:

Table 15. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **6**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|---|---|---|---|---|---|---|
| W(1) | 53(1) | 69(1) | 49(1) | 0(1) | 14(1) | -3(1) |
| Sn(1) | 71(1) | 71(1) | 64(1) | 7(1) | 14(1) | 2(1) |
| S(1) | 61(1) | 87(1) | 51(1) | -3(1) | 18(1) | -5(1) |
| S(2) | 60(1) | 79(1) | 83(1) | 13(1) | 27(1) | -3(1) |
| S(3) | 75(1) | 74(1) | 56(1) | -9(1) | 21(1) | -14(1) |
| C(1) | 55(4) | 95(5) | 63(5) | 7(4) | 14(3) | 5(3) |
| O(1) | 91(4) | 157(5) | 59(3) | 22(3) | 23(3) | 10(4) |
| C(2) | 60(4) | 99(6) | 65(4) | 4(4) | 21(3) | 5(4) |
| O(2) | 104(4) | 70(4) | 130(5) | 2(3) | 40(4) | 0(3) |
| C(3) | 61(4) | 89(5) | 46(4) | -9(3) | 10(3) | -6(3) |
| O(3) | 65(3) | 147(5) | 84(4) | -22(3) | 26(3) | -2(3) |
| C(4) | 69(4) | 86(5) | 57(4) | -8(3) | 11(3) | -5(4) |
| C(5) | 75(4) | 81(5) | 84(5) | 3(4) | 33(4) | 4(4) |
| C(6) | 81(5) | 67(4) | 75(5) | -1(3) | 20(4) | -13(3) |
| C(11) | 124(7) | 88(6) | 96(6) | 21(5) | 1(5) | 23(6) |
| C(12) | 182(16) | 162(16) | 240(20) | 103(15) | 19(15) | 62(12) |
| C(13) | 280(30) | 146(14) | 210(20) | -31(13) | -38(17) | 96(15) |
| C(14) | 175(15) | 133(13) | 470(40) | -52(17) | 63(18) | 1(11) |
| C(21) | 112(6) | 119(7) | 76(5) | -27(5) | 30(5) | 22(5) |
| C(22) | 113(8) | 213(12) | 117(8) | 77(8) | 72(7) | 38(7) |
| C(23) | 93(6) | 110(6) | 70(5) | -7(4) | 13(4) | -36(5) |

Table 16. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters (Å $^2$x 10 $^3$) for **6**.

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(4A) | 7006 | 7519 | -911 | 91 |
| H(4B) | 7391 | 8383 | -1553 | 91 |
| H(5A) | 7117 | 9823 | 1405 | 95 |
| H(5B) | 6094 | 9234 | 633 | 95 |
| H(6A) | 9276 | 10584 | 208 | 94 |
| H(6B) | 8828 | 11657 | 681 | 94 |
| H(11A) | 6640 | 11702 | -1578 | 140 |
| H(11B) | 5696 | 10811 | -1671 | 140 |
| H(12A) | 5509 | 12968 | -1367 | 258 |
| H(12B) | 5345 | 11974 | -673 | 258 |
| H(13A) | 7059 | 13278 | -230 | 310 |
| H(13B) | 6623 | 12547 | 407 | 310 |
| H(14A) | 5748 | 14862 | -336 | 420 |
| H(14B) | 6858 | 14848 | 483 | 420 |
| H(14C) | 5923 | 14213 | 636 | 420 |
| H(21A) | 8289 | 5416 | -666 | 157 |
| H(21B) | 9402 | 5640 | -636 | 157 |
| H(21C) | 8428 | 6126 | -1509 | 157 |
| H(22A) | 7632 | 7968 | 2737 | 209 |
| H(22B) | 7383 | 6462 | 2490 | 209 |
| H(22C) | 6484 | 7509 | 2171 | 209 |
| H(23A) | 11053 | 9917 | 1667 | 145 |
| H(23B) | 11136 | 9991 | 2706 | 145 |
| H(23C) | 10813 | 11253 | 2059 | 145 |

Table 17. Torsion angles [°] for **6**.

| | |
|---|---|
| C(3)-W(1)-S(1)-C(4) | -167.7(3) |
| C(2)-W(1)-S(1)-C(4) | 103.6(3) |
| C(1)-W(1)-S(1)-C(4) | -170(4) |
| S(3)-W(1)-S(1)-C(4) | -73.9(3) |
| S(2)-W(1)-S(1)-C(4) | 11.9(3) |
| C(3)-W(1)-S(1)-C(21) | 79.6(4) |
| C(2)-W(1)-S(1)-C(21) | -9.1(4) |
| C(1)-W(1)-S(1)-C(21) | 77(4) |
| S(3)-W(1)-S(1)-C(21) | 173.4(3) |
| S(2)-W(1)-S(1)-C(21) | -100.8(3) |
| C(3)-W(1)-S(2)-C(5) | -64(3) |
| C(2)-W(1)-S(2)-C(5) | -165.0(3) |
| C(1)-W(1)-S(2)-C(5) | 110.0(4) |
| S(1)-W(1)-S(2)-C(5) | -70.1(3) |
| S(3)-W(1)-S(2)-C(5) | 18.3(3) |
| C(3)-W(1)-S(2)-C(22) | -178(3) |
| C(2)-W(1)-S(2)-C(22) | 81.4(5) |
| C(1)-W(1)-S(2)-C(22) | -3.7(5) |
| S(1)-W(1)-S(2)-C(22) | 176.2(4) |
| S(3)-W(1)-S(2)-C(22) | -95.3(4) |
| C(3)-W(1)-S(3)-C(6) | 100.3(3) |
| C(2)-W(1)-S(3)-C(6) | -129(3) |
| C(1)-W(1)-S(3)-C(6) | -170.2(3) |
| S(1)-W(1)-S(3)-C(6) | 13.2(3) |
| S(2)-W(1)-S(3)-C(6) | -75.7(3) |
| C(3)-W(1)-S(3)-C(23) | -11.4(3) |
| C(2)-W(1)-S(3)-C(23) | 119(3) |
| C(1)-W(1)-S(3)-C(23) | 78.1(3) |
| S(1)-W(1)-S(3)-C(23) | -98.4(3) |
| S(2)-W(1)-S(3)-C(23) | 172.7(3) |
| C(3)-W(1)-C(1)-O(1) | -75(8) |
| C(2)-W(1)-C(1)-O(1) | 14(8) |
| S(1)-W(1)-C(1)-O(1) | -73(10) |
| S(3)-W(1)-C(1)-O(1) | -169(8) |

| | |
|---|---|
| S(2)-W(1)-C(1)-O(1) | 105(8) |
| C(3)-W(1)-C(2)-O(2) | 47(12) |
| C(1)-W(1)-C(2)-O(2) | -42(12) |
| S(1)-W(1)-C(2)-O(2) | 134(12) |
| S(3)-W(1)-C(2)-O(2) | -83(12) |
| S(2)-W(1)-C(2)-O(2) | -137(12) |
| C(2)-W(1)-C(3)-O(3) | 143(91) |
| C(1)-W(1)-C(3)-O(3) | -132(91) |
| S(1)-W(1)-C(3)-O(3) | 48(91) |
| S(3)-W(1)-C(3)-O(3) | -40(91) |
| S(2)-W(1)-C(3)-O(3) | 42(93) |
| C(21)-S(1)-C(4)-Sn(1) | 171.3(4) |
| W(1)-S(1)-C(4)-Sn(1) | 52.8(4) |
| C(11)-Sn(1)-C(4)-S(1) | 146.9(4) |
| C(5)-Sn(1)-C(4)-S(1) | -85.8(4) |
| C(6)-Sn(1)-C(4)-S(1) | 22.2(4) |
| C(22)-S(2)-C(5)-Sn(1) | 165.5(5) |
| W(1)-S(2)-C(5)-Sn(1) | 47.8(4) |
| C(11)-Sn(1)-C(5)-S(2) | 149.2(4) |
| C(4)-Sn(1)-C(5)-S(2) | 25.7(4) |
| C(6)-Sn(1)-C(5)-S(2) | -83.9(4) |
| C(23)-S(3)-C(6)-Sn(1) | 167.0(4) |
| W(1)-S(3)-C(6)-Sn(1) | 50.7(4) |
| C(11)-Sn(1)-C(6)-S(3) | 155.2(4) |
| C(4)-Sn(1)-C(6)-S(3) | -82.3(4) |
| C(5)-Sn(1)-C(6)-S(3) | 26.7(4) |
| C(4)-Sn(1)-C(11)-C(12) | 165.0(11) |
| C(5)-Sn(1)-C(11)-C(12) | 45.1(12) |
| C(6)-Sn(1)-C(11)-C(12) | -76.0(12) |
| Sn(1)-C(11)-C(12)-C(13) | 48(3) |
| C(11)-C(12)-C(13)-C(14) | 163(2) |

Symmetry transformations used to generate equivalent atoms: