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Data collection and crystal parameters for the $(Cl_4\text{-cat})(O)MoFe_3S_3(P^nPr_3)_3(CO)_5$ (**IVa**), the $(Cl_4\text{-cat})(O)MoFe_3S_3(P^nPr_3)_2(CO)_6$ (**Va**), the $(Cl_4\text{-cat})(Pyr)MoFe_3S_3(P^nPr_3)_2(CO)_6$ (**VIa**) and the $(Cl_4\text{-cat})(P^nPr_3)MoFe_3S_3(P^nPr_3)_2(CO)_6]$ (**VII**) clusters.

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| Compound | IVa | Va | VIa | VII |
|---|---|---|---|---|
| Empirical formula | C41H70Cl4Fe3MoO8P3S3 | C30H42Cl4Fe3MoO9P2S3 | C35H47Cl4Fe3MoNO8P2S3 | C39H63Cl4Fe3MoO8P3S3 |
| Formula weight | 1285.35 | 1110.05 | 1173.15 | 1254.27 |
| Wavelength, Å | 0.71073 | 0.71073 | 0.71073 | 0.71073 |
| Crystal system | Monoclinic | Monoclinic | Orthorhombic | Monoclinic |
| Sspace group | P2(1)/c | P2(1)/c | Pca2(1) | C2/c |
| Unit cell dimensions, Å, deg. | a = 22.043(7) | a = 23.8913(3) | a = 15.91300(10) | a = 47.0226(4) |
| | b = 15.462(5) | b = 11.6557(2) | b = 18.0227(2) | b = 12.67300(10) |
| | c = 17.260(7) | c = 16.1777(2) | c = 16.31820(10) | c = 18.69580(10) |
| | beta = 106.811(12) | beta = 104.8390(10) | | beta = 97.3650(10) |
| Volume, Å^3 | 5631(3) | 4354.76(11) | 4679.98(7) | 11049.23(14) |
| Z | 4 | 4 | 4 | 8 |
| Calculated density, Mg/m^3 | 1.516 | 1.693 | 1.665 | 1.508 |
| Absorption coefficient, mm^-1 | 1.405 | 1.769 | 1.650 | 1.430 |
| F(000) | 2644 | 2240 | 2376 | 5136 |
| Crystal size, mm | 0.34 x 0.34 x 0.02 | 0.03 x 0.16 x 0.20 | 0.40 x 0.40 x 0.20 | 0.08 x 0.10 x 0.28 |
| Theta range for data collection, deg. | 0.96 to 26.47 | 2.22 to 26.68 | 2.26 to 26.43 | 2.20 to 26.43 |
| Limiting indices | -27<=h<=26 | -29<=h<=29 | -19<=h<=19 | -58<=h<=58 |
| | -19<=k<=18 | -14<=k<=14 | -22<=k<=22 | -15<=k<=15 |
| | -21<=l<=21 | -20<=l<=20 | -20<=l<=20 | -23<=l<=23 |
| Reflections collected / unique | 54252 / 11606 | 38515 / 8916 | 45092 / 9585 | 97633 / 11247 |
| R(int) | 0.1044 | 0.0422 | 0.0212 | 0.0588 |
| Completeness to theta | 99.6 | 96.9 | 99.6 | 98.9 |
| Data / restraints / parameters | 11606 / 0 / 553 | 8916 / 0 / 469 | 9585 / 1 / 514 | 11247 / 0 / 551 |
| Goodness-of-fit on F^2 | 1.016 | 1.091 | 1.003 | 1.211 |
| Final R indices [I>2sigma(I)] | R1 = 0.0912, wR2 = 0.2342 | R1 = 0.0450, wR2 = 0.1347 | R1 = 0.0218, wR2 = 0.0561 | R1 = 0.0464, wR2 = 0.1409 |
| R indices (all data) | R1 = 0.1263, wR2 = 0.2751 | R1 = 0.0647, wR2 = 0.1509 | R1 = 0.0230, wR2 = 0.0571 | R1 = 0.0589, wR2 = 0.1536 |

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

| | |
|---|---|
| Empirical formula | C41 H70 Cl4 Fe3 Mo O8 P3 S3 |
| Formula weight | 1285.35 |
| Temperature | 158(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/c |
| Unit cell dimensions | a = 22.043(7) A alpha = 90 deg.<br>b = 15.462(5) A beta = 106.811(12) deg.<br>c = 17.260(7) A gamma = 90 deg. |
| Volume | 5631(3) A^3 |
| Z, Calculated density | 4, 1.516 Mg/m^3 |
| Absorption coefficient | 1.405 mm^-1 |
| F(000) | 2644 |
| Crystal size | 0.34 x 0.34 x 0.02 mm |
| Theta range for data collection | 0.96 to 26.47 deg. |
| Limiting indices | -27<=h<=26, -19<=k<=18, -21<=l<=21 |
| Reflections collected / unique | 54252 / 11606 [R(int) = 0.1044] |
| Completeness to theta = 26.47 | 99.6 % |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 11606 / 0 / 553 |
| Goodness-of-fit on F^2 | 1.016 |
| Final R indices [I>2sigma(I)] | R1 = 0.0912, wR2 = 0.2342 |
| R indices (all data) | R1 = 0.1263, wR2 = 0.2751 |
| Largest diff. peak and hole | 5.624 and -1.244 e.A^-3 |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Mo(1) | 1749(1) | 9074(1) | 2115(1) | 38(1) |
| Fe(1) | 1955(1) | 10179(1) | 4099(1) | 41(1) |
| Fe(2) | 1424(1) | 10597(1) | 2618(1) | 39(1) |
| Fe(3) | 2342(1) | 11340(1) | 2240(1) | 40(1) |
| S(1) | 1615(1) | 10469(1) | 1434(1) | 40(1) |
| S(2) | 2471(1) | 10178(1) | 3114(1) | 37(1) |
| S(3) | 1263(1) | 9352(1) | 3182(1) | 41(1) |
| P(1) | 2470(1) | 9212(1) | 5007(1) | 44(1) |
| P(2) | 405(1) | 10890(1) | 2020(1) | 47(1) |
| P(3) | 3127(1) | 11170(1) | 1695(1) | 42(1) |
| Cl(1) | 3542(1) | 8549(1) | 911(1) | 51(1) |
| Cl(2) | 4450(1) | 7069(2) | 1809(1) | 68(1) |
| Cl(3) | 4146(1) | 6065(2) | 3221(1) | 70(1) |
| Cl(4) | 2915(1) | 6524(1) | 3707(1) | 59(1) |
| O(1) | 1083(2) | 8661(3) | 1470(3) | 48(1) |
| O(2) | 2491(2) | 8896(3) | 1644(3) | 39(1) |
| O(3) | 2224(2) | 8062(3) | 2778(3) | 43(1) |
| O(4) | 2763(3) | 11584(4) | 4962(3) | 69(2) |
| O(5) | 950(3) | 10414(5) | 4869(4) | 78(2) |
| O(6) | 1468(3) | 12224(4) | 3494(3) | 58(1) |
| O(7) | 1811(3) | 12778(4) | 1164(3) | 59(1) |
| O(8) | 3031(3) | 12515(4) | 3521(3) | 66(2) |
| C(1) | 2877(3) | 8236(4) | 1966(4) | 39(2) |
| C(2) | 3400(3) | 8008(4) | 1718(4) | 41(2) |
| C(3) | 3791(3) | 7348(5) | 2109(4) | 48(2) |
| C(4) | 3652(4) | 6886(5) | 2729(4) | 49(2) |
| C(5) | 3112(3) | 7090(5) | 2963(4) | 47(2) |
| C(6) | 2732(3) | 7785(4) | 2584(4) | 38(2) |
| C(7) | 2430(4) | 11024(5) | 4622(4) | 49(2) |
| C(8) | 1369(4) | 10308(5) | 4589(4) | 53(2) |
| C(9) | 1458(3) | 11583(5) | 3144(5) | 50(2) |
| C(10) | 2023(3) | 12212(5) | 1582(5) | 48(2) |
| C(11) | 2770(3) | 12044(5) | 3026(4) | 46(2) |
| C(12) | 1985(4) | 8318(6) | 5204(5) | 73(3) |
| C(13) | 1716(4) | 7678(6) | 4543(5) | 69(2) |
| C(14) | 1561(4) | 6831(6) | 4826(5) | 70(3) |
| C(15) | 2803(5) | 9621(6) | 6012(5) | 70(3) |
| C(16) | 2418(5) | 10090(7) | 6430(5) | 75(3) |
| C(17) | 2741(5) | 10479(6) | 7228(5) | 76(3) |
| C(18) | 3107(4) | 8652(5) | 4743(5) | 61(2) |
| C(19) | 3690(4) | 9130(6) | 4821(6) | 72(3) |
| C(20) | 4192(4) | 8552(7) | 4587(6) | 78(3) |
| C(21) | -47(4) | 11035(6) | 2739(5) | 57(2) |
| C(22) | -739(4) | 11303(6) | 2419(5) | 65(2) |
| C(23) | -1028(4) | 11394(7) | 3127(7) | 84(3) |
| C(24) | -41(4) | 10104(5) | 1260(5) | 59(2) |

| | | | | |
|---|---|---|---|---|
| C(25) | -246(3) | 9254(6) | 1559(5) | 59(2) |
| C(26) | -493(4) | 8633(6) | 856(5) | 70(2) |
| C(27) | 268(4) | 11865(5) | 1393(5) | 60(2) |
| C(28) | 426(4) | 12730(5) | 1778(5) | 64(2) |
| C(29) | 246(5) | 13441(6) | 1146(6) | 80(3) |
| C(30) | 2897(3) | 10697(5) | 679(4) | 45(2) |
| C(31) | 2429(4) | 11196(5) | 12(4) | 53(2) |
| C(32) | 2234(4) | 10717(6) | -781(5) | 64(2) |
| C(33) | 3758(3) | 10448(5) | 2237(4) | 48(2) |
| C(34) | 4141(4) | 10728(6) | 3076(5) | 64(2) |
| C(35) | 4707(4) | 10125(6) | 3394(6) | 75(3) |
| C(36) | 3519(3) | 12184(5) | 1609(4) | 51(2) |
| C(37) | 4053(4) | 12149(6) | 1200(5) | 65(2) |
| C(38) | 4371(5) | 13023(7) | 1191(7) | 87(3) |
| C(39) | 4758(10) | 4087(15) | 4385(14) | 201(9) |
| C(40) | 4754(7) | 4769(12) | 4917(12) | 161(7) |
| C(41) | 4416(11) | 3557(15) | 3965(14) | 219(10) |

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

| | | | |
|---|---|---|---|
| Mo(1)-O(1) | 1.691(4) | C(16)-C(17) | 1.483(11) |
| Mo(1)-O(3) | 2.040(5) | C(18)-C(19) | 1.455(12) |
| Mo(1)-O(2) | 2.045(4) | C(19)-C(20) | 1.564(12) |
| Mo(1)-S(3) | 2.4204(19) | C(21)-C(22) | 1.524(11) |
| Mo(1)-S(1) | 2.4339(19) | C(22)-C(23) | 1.538(12) |
| Mo(1)-S(2) | 2.6145(18) | C(24)-C(25) | 1.527(11) |
| Mo(1)-Fe(2) | 2.6790(13) | C(25)-C(26) | 1.519(11) |
| Fe(1)-C(8) | 1.748(8) | C(27)-C(28) | 1.488(12) |
| Fe(1)-C(7) | 1.754(8) | C(28)-C(29) | 1.519(11) |
| Fe(1)-P(1) | 2.224(2) | C(30)-C(31) | 1.515(10) |
| Fe(1)-S(3) | 2.251(2) | C(31)-C(32) | 1.506(10) |
| Fe(1)-S(2) | 2.304(2) | C(33)-C(34) | 1.513(10) |
| Fe(1)-Fe(2) | 2.5669(16) | C(34)-C(35) | 1.527(11) |
| Fe(2)-C(9) | 1.765(9) | C(36)-C(37) | 1.539(11) |
| Fe(2)-S(1) | 2.212(2) | C(37)-C(38) | 1.526(12) |
| Fe(2)-S(3) | 2.231(2) | C(39)-C(41) | 1.20(2) |
| Fe(2)-P(2) | 2.232(2) | C(39)-C(40) | 1.40(2) |
| Fe(2)-S(2) | 2.310(2) | C(40)-C(40)#1 | 1.26(3) |
| Fe(2)-Fe(3) | 2.5696(15) | | |
| Fe(3)-C(10) | 1.773(8) | O(1)-Mo(1)-O(3) | 106.7(2) |
| Fe(3)-C(11) | 1.782(8) | O(1)-Mo(1)-O(2) | 109.3(2) |
| Fe(3)-P(3) | 2.211(2) | O(3)-Mo(1)-O(2) | 77.27(18) |
| Fe(3)-S(1) | 2.243(2) | O(1)-Mo(1)-S(3) | 94.44(17) |
| Fe(3)-S(2) | 2.309(2) | O(3)-Mo(1)-S(3) | 88.77(13) |
| P(1)-C(15) | 1.793(8) | O(2)-Mo(1)-S(3) | 155.05(13) |
| P(1)-C(18) | 1.818(8) | O(1)-Mo(1)-S(1) | 93.16(18) |
| P(1)-C(12) | 1.838(9) | O(3)-Mo(1)-S(1) | 157.19(14) |
| P(2)-C(21) | 1.815(8) | O(2)-Mo(1)-S(1) | 85.77(13) |
| P(2)-C(27) | 1.829(8) | S(3)-Mo(1)-S(1) | 101.01(7) |
| P(2)-C(24) | 1.849(8) | O(1)-Mo(1)-S(2) | 158.16(18) |
| P(3)-C(36) | 1.816(8) | O(3)-Mo(1)-S(2) | 90.89(13) |
| P(3)-C(33) | 1.818(7) | O(2)-Mo(1)-S(2) | 86.84(13) |
| P(3)-C(30) | 1.830(7) | S(3)-Mo(1)-S(2) | 72.63(6) |
| Cl(1)-C(2) | 1.728(7) | S(1)-Mo(1)-S(2) | 72.84(6) |
| Cl(2)-C(3) | 1.733(8) | O(1)-Mo(1)-Fe(2) | 106.43(17) |
| Cl(3)-C(4) | 1.727(7) | O(3)-Mo(1)-Fe(2) | 129.41(13) |
| Cl(4)-C(5) | 1.711(8) | O(2)-Mo(1)-Fe(2) | 124.50(13) |
| O(2)-C(1) | 1.341(8) | S(3)-Mo(1)-Fe(2) | 51.58(5) |
| O(3)-C(6) | 1.328(8) | S(1)-Mo(1)-Fe(2) | 50.99(5) |
| O(4)-C(7) | 1.176(9) | S(2)-Mo(1)-Fe(2) | 51.73(4) |
| O(5)-C(8) | 1.172(9) | C(8)-Fe(1)-C(7) | 94.6(4) |
| O(6)-C(9) | 1.156(9) | C(8)-Fe(1)-P(1) | 91.9(3) |
| O(7)-C(10) | 1.144(8) | C(7)-Fe(1)-P(1) | 91.8(2) |
| O(8)-C(11) | 1.144(8) | C(8)-Fe(1)-S(3) | 87.7(3) |
| C(1)-C(6) | 1.387(9) | C(7)-Fe(1)-S(3) | 165.4(2) |
| C(1)-C(2) | 1.388(9) | P(1)-Fe(1)-S(3) | 102.58(8) |
| C(2)-C(3) | 1.379(10) | C(8)-Fe(1)-S(2) | 161.7(2) |
| C(3)-C(4) | 1.391(10) | C(7)-Fe(1)-S(2) | 91.8(2) |
| C(4)-C(5) | 1.400(10) | P(1)-Fe(1)-S(2) | 105.05(8) |
| C(5)-C(6) | 1.402(9) | S(3)-Fe(1)-S(2) | 81.92(7) |
| C(12)-C(13) | 1.497(12) | C(8)-Fe(1)-Fe(2) | 105.4(2) |
| C(13)-C(14) | 1.472(11) | C(7)-Fe(1)-Fe(2) | 110.9(2) |
| C(15)-C(16) | 1.455(12) | P(1)-Fe(1)-Fe(2) | 149.72(7) |

| | | | |
|---|---|---|---|
| S(3)-Fe(1)-Fe(2) | 54.69(6) | C(15)-P(1)-C(18) | 106.3(4) |
| S(2)-Fe(1)-Fe(2) | 56.31(5) | C(15)-P(1)-C(12) | 100.5(4) |
| C(9)-Fe(2)-S(1) | 124.2(3) | C(18)-P(1)-C(12) | 102.7(4) |
| C(9)-Fe(2)-S(3) | 120.6(2) | C(15)-P(1)-Fe(1) | 115.3(3) |
| S(1)-Fe(2)-S(3) | 114.96(8) | C(18)-P(1)-Fe(1) | 114.7(3) |
| C(9)-Fe(2)-P(2) | 87.1(2) | C(12)-P(1)-Fe(1) | 115.5(3) |
| S(1)-Fe(2)-P(2) | 91.49(8) | C(21)-P(2)-C(27) | 105.9(4) |
| S(3)-Fe(2)-P(2) | 96.36(8) | C(21)-P(2)-C(24) | 106.4(4) |
| C(9)-Fe(2)-S(2) | 99.4(2) | C(27)-P(2)-C(24) | 99.2(4) |
| S(1)-Fe(2)-S(2) | 83.12(7) | C(21)-P(2)-Fe(2) | 112.7(3) |
| S(3)-Fe(2)-S(2) | 82.21(7) | C(27)-P(2)-Fe(2) | 114.3(3) |
| P(2)-Fe(2)-S(2) | 173.17(8) | C(24)-P(2)-Fe(2) | 116.9(3) |
| C(9)-Fe(2)-Fe(1) | 76.5(2) | C(36)-P(3)-C(33) | 105.1(3) |
| S(1)-Fe(2)-Fe(1) | 138.05(7) | C(36)-P(3)-C(30) | 105.9(3) |
| S(3)-Fe(2)-Fe(1) | 55.43(5) | C(33)-P(3)-C(30) | 101.9(3) |
| P(2)-Fe(2)-Fe(1) | 128.29(7) | C(36)-P(3)-Fe(3) | 112.4(3) |
| S(2)-Fe(2)-Fe(1) | 56.08(5) | C(33)-P(3)-Fe(3) | 115.5(3) |
| C(9)-Fe(2)-Fe(3) | 80.0(2) | C(30)-P(3)-Fe(3) | 114.9(2) |
| S(1)-Fe(2)-Fe(3) | 55.33(5) | C(1)-O(2)-Mo(1) | 115.0(4) |
| S(3)-Fe(2)-Fe(3) | 137.08(6) | C(6)-O(3)-Mo(1) | 116.1(4) |
| P(2)-Fe(2)-Fe(3) | 123.62(7) | O(2)-C(1)-C(6) | 116.2(6) |
| S(2)-Fe(2)-Fe(3) | 56.18(5) | O(2)-C(1)-C(2) | 123.4(6) |
| Fe(1)-Fe(2)-Fe(3) | 101.54(5) | C(6)-C(1)-C(2) | 120.4(6) |
| C(9)-Fe(2)-Mo(1) | 162.0(2) | C(3)-C(2)-C(1) | 119.6(6) |
| S(1)-Fe(2)-Mo(1) | 58.76(5) | C(3)-C(2)-Cl(1) | 121.4(5) |
| S(3)-Fe(2)-Mo(1) | 58.22(5) | C(1)-C(2)-Cl(1) | 119.0(5) |
| P(2)-Fe(2)-Mo(1) | 110.87(7) | C(2)-C(3)-C(4) | 120.8(7) |
| S(2)-Fe(2)-Mo(1) | 62.69(5) | C(2)-C(3)-Cl(2) | 119.7(6) |
| Fe(1)-Fe(2)-Mo(1) | 90.76(4) | C(4)-C(3)-Cl(2) | 119.4(6) |
| Fe(3)-Fe(2)-Mo(1) | 90.38(4) | C(3)-C(4)-C(5) | 120.0(7) |
| C(10)-Fe(3)-C(11) | 92.7(3) | C(3)-C(4)-Cl(3) | 120.6(6) |
| C(10)-Fe(3)-P(3) | 91.8(2) | C(5)-C(4)-Cl(3) | 119.4(6) |
| C(11)-Fe(3)-P(3) | 95.1(2) | C(4)-C(5)-C(6) | 118.8(7) |
| C(10)-Fe(3)-S(1) | 88.8(3) | C(4)-C(5)-Cl(4) | 121.6(5) |
| C(11)-Fe(3)-S(1) | 164.9(2) | C(6)-C(5)-Cl(4) | 119.6(6) |
| P(3)-Fe(3)-S(1) | 99.87(8) | O(3)-C(6)-C(1) | 115.4(6) |
| C(10)-Fe(3)-S(2) | 164.1(2) | O(3)-C(6)-C(5) | 124.2(6) |
| C(11)-Fe(3)-S(2) | 92.3(2) | C(1)-C(6)-C(5) | 120.3(6) |
| P(3)-Fe(3)-S(2) | 102.81(8) | O(4)-C(7)-Fe(1) | 177.9(7) |
| S(1)-Fe(3)-S(2) | 82.49(7) | O(5)-C(8)-Fe(1) | 175.4(7) |
| C(10)-Fe(3)-Fe(2) | 107.9(2) | O(6)-C(9)-Fe(2) | 178.5(7) |
| C(11)-Fe(3)-Fe(2) | 111.2(2) | O(7)-C(10)-Fe(3) | 178.9(7) |
| P(3)-Fe(3)-Fe(2) | 145.65(7) | O(8)-C(11)-Fe(3) | 177.9(7) |
| S(1)-Fe(3)-Fe(2) | 54.22(6) | C(13)-C(12)-P(1) | 118.6(7) |
| S(2)-Fe(3)-Fe(2) | 56.22(5) | C(14)-C(13)-C(12) | 114.5(8) |
| Fe(2)-S(1)-Fe(3) | 70.45(6) | C(16)-C(15)-P(1) | 121.4(7) |
| Fe(2)-S(1)-Mo(1) | 70.24(6) | C(15)-C(16)-C(17) | 118.1(8) |
| Fe(3)-S(1)-Mo(1) | 105.50(7) | C(19)-C(18)-P(1) | 117.4(7) |
| Fe(1)-S(2)-Fe(3) | 119.21(8) | C(18)-C(19)-C(20) | 110.9(8) |
| Fe(1)-S(2)-Fe(2) | 67.60(6) | C(22)-C(21)-P(2) | 118.4(6) |
| Fe(3)-S(2)-Fe(2) | 67.60(6) | C(21)-C(22)-C(23) | 109.8(8) |
| Fe(1)-S(2)-Mo(1) | 98.62(7) | C(25)-C(24)-P(2) | 118.4(6) |
| Fe(3)-S(2)-Mo(1) | 98.12(7) | C(26)-C(25)-C(24) | 110.2(7) |
| Fe(2)-S(2)-Mo(1) | 65.57(5) | C(28)-C(27)-P(2) | 120.0(6) |
| Fe(2)-S(3)-Fe(1) | 69.88(7) | C(27)-C(28)-C(29) | 110.6(7) |
| Fe(2)-S(3)-Mo(1) | 70.20(6) | C(31)-C(30)-P(3) | 117.6(5) |
| Fe(1)-S(3)-Mo(1) | 106.11(8) | C(32)-C(31)-C(30) | 113.1(7) |

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| | | | |
|---|---|---|---|
| C(34)-C(33)-P(3) | 116.7(6) | C(38)-C(37)-C(36) | 112.9(8) |
| C(33)-C(34)-C(35) | 109.9(8) | C(41)-C(39)-C(40) | 142(2) |
| C(37)-C(36)-P(3) | 116.9(6) | C(40)#1-C(40)-C(39) | 114(2) |

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **IVa**. 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 |
|---|---|---|---|---|---|---|
| Mo(1) | 42(1) | 37(1) | 41(1) | -1(1) | 20(1) | -1(1) |
| Fe(1) | 50(1) | 38(1) | 41(1) | -1(1) | 24(1) | 3(1) |
| Fe(2) | 41(1) | 39(1) | 44(1) | 1(1) | 22(1) | 3(1) |
| Fe(3) | 44(1) | 37(1) | 44(1) | 1(1) | 22(1) | 0(1) |
| S(1) | 42(1) | 42(1) | 39(1) | 1(1) | 17(1) | 2(1) |
| S(2) | 41(1) | 37(1) | 40(1) | 0(1) | 19(1) | 0(1) |
| S(3) | 44(1) | 43(1) | 44(1) | 1(1) | 25(1) | 0(1) |
| P(1) | 58(1) | 41(1) | 41(1) | 1(1) | 27(1) | 3(1) |
| P(2) | 44(1) | 46(1) | 55(1) | 3(1) | 22(1) | 4(1) |
| P(3) | 43(1) | 40(1) | 48(1) | 4(1) | 22(1) | 0(1) |
| Cl(1) | 61(1) | 48(1) | 56(1) | -1(1) | 35(1) | 3(1) |
| Cl(2) | 57(1) | 73(2) | 80(1) | -3(1) | 33(1) | 20(1) |
| Cl(3) | 71(1) | 65(1) | 72(1) | 11(1) | 20(1) | 27(1) |
| Cl(4) | 85(1) | 45(1) | 56(1) | 10(1) | 34(1) | 14(1) |
| O(1) | 47(3) | 49(3) | 50(3) | -7(2) | 18(2) | -6(2) |
| O(2) | 42(2) | 39(3) | 42(2) | 0(2) | 21(2) | 5(2) |
| O(3) | 53(3) | 37(3) | 47(3) | -1(2) | 29(2) | -1(2) |
| O(4) | 97(4) | 54(4) | 52(3) | -10(3) | 17(3) | -8(3) |
| O(5) | 76(4) | 104(5) | 72(4) | -9(3) | 50(3) | 11(4) |
| O(6) | 63(3) | 48(3) | 68(3) | -10(3) | 28(3) | 6(3) |
| O(7) | 65(3) | 53(4) | 65(3) | 11(3) | 28(3) | 10(3) |
| O(8) | 84(4) | 60(4) | 59(3) | -8(3) | 28(3) | -13(3) |
| C(1) | 43(3) | 38(4) | 42(3) | -3(3) | 21(3) | -2(3) |
| C(2) | 40(3) | 44(4) | 46(4) | -12(3) | 26(3) | 0(3) |
| C(3) | 53(4) | 44(4) | 51(4) | -14(3) | 20(3) | 3(3) |
| C(4) | 55(4) | 47(4) | 45(4) | -2(3) | 14(3) | 5(3) |
| C(5) | 58(4) | 42(4) | 45(4) | -2(3) | 22(3) | 6(3) |
| C(6) | 50(4) | 33(4) | 37(3) | -3(3) | 21(3) | -2(3) |
| C(7) | 60(5) | 56(5) | 35(4) | 2(3) | 19(3) | 10(4) |
| C(8) | 61(5) | 58(5) | 41(4) | -7(3) | 20(3) | 7(4) |
| C(9) | 49(4) | 52(5) | 55(4) | 11(4) | 26(3) | 15(3) |
| C(10) | 53(4) | 43(4) | 61(5) | 6(4) | 35(4) | 3(3) |
| C(11) | 48(4) | 42(4) | 52(4) | 2(3) | 23(3) | -3(3) |
| C(12) | 77(6) | 76(6) | 62(5) | 7(5) | 14(4) | -11(5) |
| C(13) | 70(6) | 64(6) | 73(6) | 2(5) | 23(4) | 4(4) |
| C(14) | 87(6) | 55(6) | 60(5) | 11(4) | 9(4) | -21(5) |
| C(15) | 95(7) | 60(6) | 53(5) | 0(4) | 17(4) | 25(5) |
| C(16) | 105(7) | 76(7) | 53(5) | -6(4) | 37(5) | 5(5) |
| C(17) | 107(7) | 65(6) | 54(5) | -8(4) | 21(5) | 15(5) |
| C(18) | 73(5) | 53(5) | 62(5) | 3(4) | 28(4) | 8(4) |
| C(19) | 45(5) | 84(7) | 83(6) | 21(5) | 13(4) | -2(4) |
| C(20) | 61(5) | 98(8) | 90(7) | -14(6) | 43(5) | 6(5) |
| C(21) | 56(5) | 59(5) | 66(5) | 3(4) | 32(4) | 1(4) |
| C(22) | 50(5) | 68(6) | 85(6) | 8(5) | 34(4) | 12(4) |
| C(23) | 70(6) | 73(7) | 131(9) | 13(6) | 67(6) | 10(5) |
| C(24) | 43(4) | 58(5) | 77(5) | 15(4) | 16(4) | 4(4) |

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| | | | | | | |
|---|---|---|---|---|---|---|
| C(25) | 42(4) | 72(6) | 66(5) | 5(4) | 22(4) | -1(4) |
| C(26) | 68(6) | 72(6) | 74(6) | 0(5) | 26(5) | -17(5) |
| C(27) | 60(5) | 60(5) | 67(5) | 13(4) | 29(4) | 20(4) |
| C(28) | 69(5) | 53(5) | 81(6) | 10(4) | 41(5) | 6(4) |
| C(29) | 83(6) | 63(6) | 112(8) | 28(5) | 57(6) | 14(5) |
| C(30) | 46(4) | 44(4) | 49(4) | 2(3) | 22(3) | 1(3) |
| C(31) | 63(5) | 55(5) | 44(4) | 2(3) | 21(3) | -10(4) |
| C(32) | 86(6) | 55(5) | 54(5) | 4(4) | 24(4) | 6(4) |
| C(33) | 50(4) | 39(4) | 61(4) | 5(3) | 27(3) | 1(3) |
| C(34) | 53(5) | 71(6) | 66(5) | 8(4) | 16(4) | -8(4) |
| C(35) | 57(5) | 70(6) | 93(7) | 7(5) | 15(5) | 4(4) |
| C(36) | 47(4) | 60(5) | 48(4) | -1(3) | 17(3) | -8(3) |
| C(37) | 56(5) | 70(6) | 78(6) | 3(5) | 32(4) | -17(4) |
| C(38) | 76(6) | 85(8) | 111(8) | 22(6) | 42(6) | -20(5) |

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

| | |
|---|---|
| Empirical formula | C30 H42 Cl4 Fe3 Mo O9 P2 S3 |
| Formula weight | 1110.05 |
| Temperature | 158(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, P2(1)/c |
| Unit cell dimensions | a = 23.8913(3)A alpha = 90 deg.<br>b = 11.6557(2)A beta = 104.8390(10)deg.<br>c = 16.1777(2)A gamma = 90 deg. |
| Volume | 4354.76(11) A^3 |
| Z, Calculated density | 4, 1.693 Mg/m^3 |
| Absorption coefficient | 1.769 mm^-1 |
| F(000) | 2240 |
| Crystal size | 0.03 x 0.16 x 0.20 mm |
| Theta range for data collection | 2.22 to 26.68 deg. |
| Limiting indices | -29<=h<=29, -14<=k<=14, -20<=l<=20 |
| Reflections collected / unique | 38515 / 8916 [R(int) = 0.0422] |
| Completeness to theta = 26.68 | 96.9 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 8916 / 0 / 469 |
| Goodness-of-fit on F^2 | 1.091 |
| Final R indices [I>2sigma(I)] | R1 = 0.0450, wR2 = 0.1347 |
| R indices (all data) | R1 = 0.0647, wR2 = 0.1509 |
| Largest diff. peak and hole | 1.615 and -0.775 e.A^-3 |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Mo(1) | 2667(1) | 122(1) | 1579(1) | 27(1) |
| Fe(1) | 2449(1) | -1929(1) | 3286(1) | 29(1) |
| Fe(2) | 3317(1) | -1360(1) | 2676(1) | 29(1) |
| Fe(3) | 3726(1) | 358(1) | 3697(1) | 27(1) |
| S(1) | 2435(1) | -1854(1) | 1896(1) | 30(1) |
| S(2) | 3686(1) | 249(1) | 2297(1) | 31(1) |
| S(3) | 2768(1) | -84(1) | 3224(1) | 25(1) |
| Cl(1) | 2716(1) | 4425(1) | 1387(1) | 47(1) |
| Cl(2) | 1488(1) | 5535(1) | 1166(1) | 56(1) |
| Cl(3) | 421(1) | 3963(1) | 1158(1) | 66(1) |
| Cl(4) | 585(1) | 1288(1) | 1313(1) | 55(1) |
| P(1) | 1497(1) | -1868(1) | 3161(1) | 44(1) |
| P(2) | 3709(1) | 2245(1) | 3851(1) | 31(1) |
| O(1) | 2487(2) | -4424(3) | 3210(2) | 56(1) |
| O(2) | 2753(2) | -1930(3) | 5162(2) | 57(1) |
| O(3) | 3938(2) | -2615(3) | 4218(3) | 61(1) |
| O(4) | 3917(2) | -2821(4) | 1693(3) | 74(1) |
| O(5) | 3712(1) | -89(3) | 5480(2) | 38(1) |
| O(6) | 4990(1) | 318(4) | 4103(2) | 52(1) |
| O(7) | 2703(1) | 1859(2) | 1600(2) | 31(1) |
| O(8) | 1826(1) | 559(2) | 1487(2) | 32(1) |
| O(9) | 2752(2) | -261(3) | 614(2) | 38(1) |
| C(1) | 2452(2) | -3453(4) | 3234(3) | 37(1) |
| C(2) | 2637(2) | -1935(4) | 4438(3) | 40(1) |
| C(3) | 3680(2) | -2095(4) | 3639(3) | 41(1) |
| C(4) | 3674(2) | -2258(4) | 2069(3) | 45(1) |
| C(5) | 3719(2) | 70(4) | 4781(3) | 30(1) |
| C(6) | 4491(2) | 343(4) | 3941(3) | 37(1) |
| C(7) | 2191(2) | 2396(4) | 1476(3) | 29(1) |
| C(8) | 2118(2) | 3588(4) | 1387(3) | 34(1) |
| C(9) | 1574(2) | 4060(4) | 1289(3) | 38(1) |
| C(10) | 1098(2) | 3368(4) | 1273(3) | 39(1) |
| C(11) | 1169(2) | 2167(4) | 1341(3) | 37(1) |
| C(12) | 1710(2) | 1691(4) | 1430(3) | 30(1) |
| C(13) | 1079(2) | -1731(5) | 2022(4) | 51(1) |
| C(14) | 418(3) | -1682(6) | 1833(4) | 65(2) |
| C(15) | 172(3) | -1774(8) | 863(5) | 87(2) |
| C(16) | 1204(3) | -800(7) | 3724(5) | 77(2) |
| C(17) | 1317(3) | 314(8) | 3524(5) | 86(2) |
| C(18) | 838(3) | 1201(6) | 3798(5) | 79(2) |
| C(19) | 1165(3) | -3242(8) | 3438(6) | 99(3) |
| C(20) | 1399(3) | -3648(7) | 4311(6) | 87(3) |
| C(21) | 1063(3) | -4712(7) | 4439(6) | 96(3) |
| C(22) | 4168(2) | 2745(4) | 4873(3) | 42(1) |
| C(23) | 4153(3) | 4022(5) | 5032(4) | 57(2) |
| C(24) | 4641(3) | 4349(6) | 5823(4) | 76(2) |

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| | | | | |
|---|---|---|---|---|
| C(25) | 3947(2) | 3061(4) | 3026(3) | 39(1) |
| C(26) | 4562(2) | 2866(5) | 2964(3) | 48(1) |
| C(27) | 4712(3) | 3661(6) | 2303(4) | 61(2) |
| C(28) | 2991(2) | 2851(4) | 3757(3) | 36(1) |
| C(29) | 2695(2) | 2503(4) | 4437(3) | 37(1) |
| C(30) | 2091(2) | 3067(4) | 4264(3) | 44(1) |

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

| | | | |
|---|---|---|---|
| Mo(1)-O(9) | 1.685(3) | C(20)-C(21) | 1.519(10) |
| Mo(1)-O(7) | 2.027(3) | C(22)-C(23) | 1.513(7) |
| Mo(1)-O(8) | 2.042(3) | C(23)-C(24) | 1.542(9) |
| Mo(1)-S(2) | 2.4186(12) | C(25)-C(26) | 1.517(7) |
| Mo(1)-S(1) | 2.4542(11) | C(26)-C(27) | 1.524(7) |
| Mo(1)-S(3) | 2.6198(11) | C(28)-C(29) | 1.509(6) |
| Mo(1)-Fe(2) | 2.6710(7) | C(29)-C(30) | 1.544(7) |
| Fe(1)-C(1) | 1.779(5) | | |
| Fe(1)-C(2) | 1.802(5) | O(9)-Mo(1)-O(7) | 105.39(14) |
| Fe(1)-P(1) | 2.2333(15) | O(9)-Mo(1)-O(8) | 110.37(14) |
| Fe(1)-S(1) | 2.2408(12) | O(7)-Mo(1)-O(8) | 77.81(12) |
| Fe(1)-S(3) | 2.2925(12) | O(9)-Mo(1)-S(2) | 96.66(12) |
| Fe(1)-Fe(2) | 2.6002(9) | O(7)-Mo(1)-S(2) | 84.12(9) |
| Fe(2)-C(4) | 1.791(5) | O(8)-Mo(1)-S(2) | 150.57(9) |
| Fe(2)-C(3) | 1.794(5) | O(9)-Mo(1)-S(1) | 91.86(11) |
| Fe(2)-S(2) | 2.2244(13) | O(7)-Mo(1)-S(1) | 160.93(9) |
| Fe(2)-S(1) | 2.2379(13) | O(8)-Mo(1)-S(1) | 88.73(8) |
| Fe(2)-S(3) | 2.3059(11) | S(2)-Mo(1)-S(1) | 102.19(4) |
| Fe(2)-Fe(3) | 2.6204(9) | O(9)-Mo(1)-S(3) | 156.20(11) |
| Fe(3)-C(6) | 1.767(5) | O(7)-Mo(1)-S(3) | 94.70(9) |
| Fe(3)-C(5) | 1.789(5) | O(8)-Mo(1)-S(3) | 85.97(9) |
| Fe(3)-P(2) | 2.2153(13) | S(2)-Mo(1)-S(3) | 72.46(4) |
| Fe(3)-S(2) | 2.2465(12) | S(1)-Mo(1)-S(3) | 70.66(3) |
| Fe(3)-S(3) | 2.2790(12) | O(9)-Mo(1)-Fe(2) | 104.88(11) |
| Cl(1)-C(8) | 1.731(5) | O(7)-Mo(1)-Fe(2) | 128.33(8) |
| Cl(2)-C(9) | 1.737(5) | O(8)-Mo(1)-Fe(2) | 127.15(8) |
| Cl(3)-C(10) | 1.724(5) | S(2)-Mo(1)-Fe(2) | 51.54(3) |
| Cl(4)-C(11) | 1.723(5) | S(1)-Mo(1)-Fe(2) | 51.57(3) |
| P(1)-C(16) | 1.789(7) | S(3)-Mo(1)-Fe(2) | 51.66(3) |
| P(1)-C(13) | 1.864(6) | C(1)-Fe(1)-C(2) | 92.4(2) |
| P(1)-C(19) | 1.891(8) | C(1)-Fe(1)-P(1) | 92.49(16) |
| P(2)-C(28) | 1.823(5) | C(2)-Fe(1)-P(1) | 94.11(18) |
| P(2)-C(22) | 1.830(5) | C(1)-Fe(1)-S(1) | 89.45(15) |
| P(2)-C(25) | 1.844(5) | C(2)-Fe(1)-S(1) | 166.83(17) |
| O(1)-C(1) | 1.136(6) | P(1)-Fe(1)-S(1) | 98.84(5) |
| O(2)-C(2) | 1.132(6) | C(1)-Fe(1)-S(3) | 158.39(16) |
| O(3)-C(3) | 1.153(6) | C(2)-Fe(1)-S(3) | 92.98(15) |
| O(4)-C(4) | 1.149(6) | P(1)-Fe(1)-S(3) | 107.96(5) |
| O(5)-C(5) | 1.151(5) | S(1)-Fe(1)-S(3) | 80.76(4) |
| O(6)-C(6) | 1.154(6) | C(1)-Fe(1)-Fe(2) | 102.91(16) |
| O(7)-C(7) | 1.344(5) | C(2)-Fe(1)-Fe(2) | 112.49(17) |
| O(8)-C(12) | 1.347(5) | P(1)-Fe(1)-Fe(2) | 148.26(5) |
| C(7)-C(12) | 1.400(6) | S(1)-Fe(1)-Fe(2) | 54.46(3) |
| C(7)-C(8) | 1.403(6) | S(3)-Fe(1)-Fe(2) | 55.81(3) |
| C(8)-C(9) | 1.382(7) | C(4)-Fe(2)-C(3) | 90.3(2) |
| C(9)-C(10) | 1.390(7) | C(4)-Fe(2)-S(2) | 93.65(18) |
| C(10)-C(11) | 1.412(7) | C(3)-Fe(2)-S(2) | 120.33(17) |
| C(11)-C(12) | 1.378(6) | C(4)-Fe(2)-S(1) | 93.02(17) |
| C(13)-C(14) | 1.532(8) | C(3)-Fe(2)-S(1) | 122.82(17) |
| C(14)-C(15) | 1.532(9) | S(2)-Fe(2)-S(1) | 116.37(5) |
| C(16)-C(17) | 1.382(10) | C(4)-Fe(2)-S(3) | 169.80(18) |
| C(17)-C(18) | 1.684(10) | C(3)-Fe(2)-S(3) | 99.81(16) |
| C(19)-C(20) | 1.459(10) | S(2)-Fe(2)-S(3) | 82.32(4) |

| | | | |
|---|---|---|---|
| S(1)-Fe(2)-S(3) | 80.53(4) | C(16)-P(1)-Fe(1) | 119.9(3) |
| C(4)-Fe(2)-Fe(1) | 126.60(18) | C(13)-P(1)-Fe(1) | |
| C(3)-Fe(2)-Fe(1) | 78.66(17) | 111.56(17) | |
| S(2)-Fe(2)-Fe(1) | 136.92(4) | C(19)-P(1)-Fe(1) | 115.5(2) |
| S(1)-Fe(2)-Fe(1) | 54.56(3) | C(28)-P(2)-C(22) | 106.6(2) |
| S(3)-Fe(2)-Fe(1) | 55.32(3) | C(28)-P(2)-C(25) | 101.4(2) |
| C(4)-Fe(2)-Fe(3) | 129.70(17) | C(22)-P(2)-C(25) | 105.4(2) |
| C(3)-Fe(2)-Fe(3) | 78.38(16) | C(28)-P(2)-Fe(3) | 114.84(15) |
| S(2)-Fe(2)-Fe(3) | 54.51(3) | C(22)-P(2)-Fe(3) | 113.15(18) |
| S(1)-Fe(2)-Fe(3) | 134.20(4) | C(25)-P(2)-Fe(3) | 114.39(16) |
| S(3)-Fe(2)-Fe(3) | 54.66(3) | C(7)-O(7)-Mo(1) | 115.5(3) |
| Fe(1)-Fe(2)-Fe(3) | 99.22(3) | C(12)-O(8)-Mo(1) | 115.5(3) |
| C(4)-Fe(2)-Mo(1) | 106.90(18) | O(1)-C(1)-Fe(1) | 176.1(5) |
| C(3)-Fe(2)-Mo(1) | 162.72(16) | O(2)-C(2)-Fe(1) | 179.5(5) |
| S(2)-Fe(2)-Mo(1) | 58.36(3) | O(3)-C(3)-Fe(2) | 174.4(5) |
| S(1)-Fe(2)-Mo(1) | 59.21(3) | O(4)-C(4)-Fe(2) | 178.1(5) |
| S(3)-Fe(2)-Mo(1) | 63.02(3) | O(5)-C(5)-Fe(3) | 178.5(4) |
| Fe(1)-Fe(2)-Mo(1) | 91.85(2) | O(6)-C(6)-Fe(3) | 179.0(5) |
| Fe(3)-Fe(2)-Mo(1) | 89.08(2) | O(7)-C(7)-C(12) | 116.1(4) |
| C(6)-Fe(3)-C(5) | 92.8(2) | O(7)-C(7)-C(8) | 124.2(4) |
| C(6)-Fe(3)-P(2) | 91.88(17) | C(12)-C(7)-C(8) | 119.8(4) |
| C(5)-Fe(3)-P(2) | 94.05(14) | C(9)-C(8)-C(7) | 119.7(4) |
| C(6)-Fe(3)-S(2) | 89.93(14) | C(9)-C(8)-Cl(1) | 121.9(3) |
| C(5)-Fe(3)-S(2) | 165.69(14) | C(7)-C(8)-Cl(1) | 118.5(4) |
| P(2)-Fe(3)-S(2) | 99.91(5) | C(8)-C(9)-C(10) | 120.8(4) |
| C(6)-Fe(3)-S(3) | 164.73(17) | C(8)-C(9)-Cl(2) | 119.2(4) |
| C(5)-Fe(3)-S(3) | 91.51(14) | C(10)-C(9)-Cl(2) | 120.0(4) |
| P(2)-Fe(3)-S(3) | 102.43(5) | C(9)-C(10)-C(11) | 119.5(4) |
| S(2)-Fe(3)-S(3) | 82.45(4) | C(9)-C(10)-Cl(3) | 120.6(4) |
| C(6)-Fe(3)-Fe(2) | 109.30(16) | C(11)-C(10)-Cl(3) | 119.9(4) |
| C(5)-Fe(3)-Fe(2) | 112.28(14) | C(12)-C(11)-C(10) | 119.9(4) |
| P(2)-Fe(3)-Fe(2) | 144.56(4) | C(12)-C(11)-Cl(4) | 119.7(4) |
| S(2)-Fe(3)-Fe(2) | 53.73(3) | C(10)-C(11)-Cl(4) | 120.4(4) |
| S(3)-Fe(3)-Fe(2) | 55.63(3) | O(8)-C(12)-C(11) | 124.9(4) |
| Fe(2)-S(1)-Fe(1) | 70.98(4) | O(8)-C(12)-C(7) | 114.8(4) |
| Fe(2)-S(1)-Mo(1) | 69.22(3) | C(11)-C(12)-C(7) | 120.3(4) |
| Fe(1)-S(1)-Mo(1) | 107.46(4) | C(14)-C(13)-P(1) | 117.7(4) |
| Fe(2)-S(2)-Fe(3) | 71.76(4) | C(15)-C(14)-C(13) | 107.9(6) |
| Fe(2)-S(2)-Mo(1) | 70.10(4) | C(17)-C(16)-P(1) | 114.1(6) |
| Fe(3)-S(2)-Mo(1) | 105.37(4) | C(16)-C(17)-C(18) | 109.1(6) |
| Fe(3)-S(3)-Fe(1) | 120.88(5) | C(20)-C(19)-P(1) | 115.2(6) |
| Fe(3)-S(3)-Fe(2) | 69.71(4) | C(19)-C(20)-C(21) | 108.2(7) |
| Fe(1)-S(3)-Fe(2) | 68.87(4) | C(23)-C(22)-P(2) | 115.6(4) |
| Fe(3)-S(3)-Mo(1) | 98.28(4) | C(22)-C(23)-C(24) | 109.8(5) |
| Fe(1)-S(3)-Mo(1) | 100.68(4) | C(26)-C(25)-P(2) | 117.0(4) |
| Fe(2)-S(3)-Mo(1) | 65.31(3) | C(25)-C(26)-C(27) | 111.1(5) |
| C(16)-P(1)-C(13) | 105.3(3) | C(29)-C(28)-P(2) | 116.2(3) |
| C(16)-P(1)-C(19) | 103.0(4) | C(28)-C(29)-C(30) | 110.1(4) |
| C(13)-P(1)-C(19) | 99.0(3) | | |

Symmetry transformations used to generate equivalent atoms:

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Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **Va**.
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 |
|---|---|---|---|---|---|---|
| Mo(1) | 27(1) | 26(1) | 27(1) | 1(1) | 6(1) | 0(1) |
| Fe(1) | 34(1) | 22(1) | 33(1) | 2(1) | 11(1) | 3(1) |
| Fe(2) | 29(1) | 27(1) | 31(1) | -2(1) | 9(1) | 7(1) |
| Fe(3) | 24(1) | 32(1) | 27(1) | 0(1) | 7(1) | 3(1) |
| S(1) | 34(1) | 24(1) | 32(1) | -2(1) | 8(1) | 1(1) |
| S(2) | 26(1) | 39(1) | 29(1) | -1(1) | 9(1) | 1(1) |
| S(3) | 26(1) | 23(1) | 28(1) | 0(1) | 8(1) | 3(1) |
| Cl(1) | 54(1) | 29(1) | 60(1) | 1(1) | 21(1) | -10(1) |
| Cl(2) | 74(1) | 29(1) | 63(1) | -2(1) | 11(1) | 11(1) |
| Cl(3) | 44(1) | 57(1) | 95(1) | -1(1) | 17(1) | 21(1) |
| Cl(4) | 28(1) | 51(1) | 83(1) | 4(1) | 12(1) | -4(1) |
| P(1) | 39(1) | 38(1) | 60(1) | 14(1) | 22(1) | 12(1) |
| P(2) | 28(1) | 31(1) | 32(1) | 1(1) | 7(1) | -5(1) |
| O(1) | 84(3) | 28(2) | 62(2) | 6(2) | 29(2) | 9(2) |
| O(2) | 87(3) | 53(2) | 33(2) | 3(2) | 17(2) | -20(2) |
| O(3) | 66(3) | 55(2) | 57(2) | 15(2) | 7(2) | 25(2) |
| O(4) | 57(3) | 84(3) | 84(3) | -48(3) | 23(2) | 12(2) |
| O(5) | 34(2) | 49(2) | 32(2) | 7(2) | 7(1) | 2(2) |
| O(6) | 24(2) | 88(3) | 43(2) | 0(2) | 7(2) | 7(2) |
| O(7) | 28(2) | 26(2) | 39(2) | 2(1) | 9(1) | -1(1) |
| O(8) | 27(2) | 25(2) | 41(2) | 6(1) | 6(1) | 0(1) |
| O(9) | 41(2) | 40(2) | 32(2) | -5(1) | 9(1) | 0(2) |
| C(1) | 44(3) | 30(2) | 38(2) | 3(2) | 13(2) | 8(2) |
| C(2) | 54(3) | 29(2) | 39(3) | 4(2) | 16(2) | -7(2) |
| C(3) | 40(3) | 31(2) | 48(3) | 6(2) | 6(2) | 12(2) |
| C(4) | 38(3) | 47(3) | 49(3) | -12(2) | 8(2) | 2(2) |
| C(5) | 21(2) | 31(2) | 36(2) | 0(2) | 5(2) | 3(2) |
| C(6) | 34(3) | 53(3) | 23(2) | 1(2) | 9(2) | 6(2) |
| C(7) | 29(2) | 30(2) | 29(2) | 3(2) | 6(2) | -1(2) |
| C(8) | 44(3) | 26(2) | 32(2) | -1(2) | 11(2) | -4(2) |
| C(9) | 47(3) | 26(2) | 40(3) | -1(2) | 9(2) | 7(2) |
| C(10) | 35(3) | 39(3) | 43(3) | -2(2) | 7(2) | 14(2) |
| C(11) | 28(2) | 39(3) | 43(3) | 1(2) | 10(2) | -1(2) |
| C(12) | 32(2) | 28(2) | 30(2) | 4(2) | 6(2) | -1(2) |
| C(13) | 39(3) | 41(3) | 76(4) | -11(3) | 22(3) | -7(2) |
| C(14) | 46(4) | 63(4) | 83(5) | 11(3) | 9(3) | -17(3) |
| C(15) | 66(5) | 110(6) | 73(5) | -8(4) | -2(4) | -38(4) |
| C(16) | 69(5) | 93(5) | 67(4) | -12(4) | 12(4) | 14(4) |
| C(17) | 62(5) | 122(7) | 76(5) | -16(5) | 22(4) | -47(5) |
| C(18) | 68(5) | 55(4) | 110(6) | -12(4) | 18(4) | 16(3) |
| C(19) | 40(4) | 125(7) | 132(7) | 82(6) | 22(4) | 13(4) |
| C(20) | 54(4) | 82(5) | 131(7) | 57(5) | 36(4) | 15(4) |
| C(21) | 50(4) | 108(6) | 130(7) | 51(5) | 23(5) | 3(4) |
| C(22) | 44(3) | 47(3) | 32(2) | -2(2) | 7(2) | -14(2) |
| C(23) | 65(4) | 53(3) | 56(3) | -17(3) | 20(3) | -23(3) |
| C(24) | 90(5) | 85(5) | 60(4) | -34(4) | 31(4) | -44(4) |
| C(25) | 43(3) | 36(3) | 37(3) | 3(2) | 12(2) | -7(2) |

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| | | | | | | |
|---|---|---|---|---|---|---|
| C(26) | 38(3) | 65(4) | 39(3) | 3(2) | 8(2) | -24(3) |
| C(27) | 58(4) | 79(4) | 45(3) | 11(3) | 12(3) | -20(3) |
| C(28) | 38(3) | 24(2) | 47(3) | 1(2) | 10(2) | -4(2) |
| C(29) | 41(3) | 34(2) | 39(3) | -4(2) | 13(2) | -1(2) |
| C(30) | 39(3) | 42(3) | 53(3) | -3(2) | 17(2) | 1(2) |

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

| | |
|---|---|
| Empirical formula | C35 H47 Cl4 Fe3 Mo N O8 P2 S3 |
| Formula weight | 1173.15 |
| Temperature | 158(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Orthorhombic, Pca2(1) |
| Unit cell dimensions | a = 15.91300(10) A alpha = 90 deg.<br>b = 18.0227(2) A beta = 90 deg.<br>c = 16.31820(10) A gamma = 90 deg. |
| Volume | 4679.98(7) A^3 |
| Z, Calculated density | 4, 1.665 Mg/m^3 |
| Absorption coefficient | 1.650 mm^-1 |
| F(000) | 2376 |
| Crystal size | 0.40 x 0.40 x 0.20 mm |
| Theta range for data collection | 2.26 to 26.43 deg. |
| Limiting indices | -19<=h<=19, -22<=k<=22, -20<=l<=20 |
| Reflections collected / unique | 45092 / 9585 [R(int) = 0.0212] |
| Completeness to theta = 26.43 | 99.6 % |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 9585 / 1 / 514 |
| Goodness-of-fit on F^2 | 1.003 |
| Final R indices [I>2sigma(I)] | R1 = 0.0218, wR2 = 0.0561 |
| R indices (all data) | R1 = 0.0230, wR2 = 0.0571 |
| Absolute structure parameter | 0.016(9) |
| Largest diff. peak and hole | 1.109 and -0.737 e.A^-3 |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Mo(1) | 5562(1) | 7810(1) | 5865(1) | 14(1) |
| Fe(1) | 4114(1) | 7652(1) | 4930(1) | 20(1) |
| Fe(2) | 5631(1) | 7492(1) | 4266(1) | 18(1) |
| Fe(3) | 5784(1) | 6334(1) | 5222(1) | 16(1) |
| Cl(1) | 6713(1) | 7075(1) | 8627(1) | 27(1) |
| Cl(2) | 6038(1) | 8106(1) | 10031(1) | 37(1) |
| Cl(3) | 4882(1) | 9457(1) | 9594(1) | 36(1) |
| Cl(4) | 4320(1) | 9703(1) | 7764(1) | 37(1) |
| S(1) | 5086(1) | 8511(1) | 4751(1) | 20(1) |
| S(2) | 6670(1) | 7301(1) | 5133(1) | 17(1) |
| S(3) | 4641(1) | 6853(1) | 5898(1) | 18(1) |
| P(1) | 3400(1) | 8323(1) | 3969(1) | 36(1) |
| P(2) | 6341(1) | 5758(1) | 6328(1) | 19(1) |
| O(1) | 2978(1) | 8223(1) | 6203(1) | 30(1) |
| O(2) | 3241(2) | 6330(2) | 4299(2) | 52(1) |
| O(3) | 4891(2) | 6325(1) | 3246(1) | 37(1) |
| O(4) | 6518(2) | 8017(1) | 2813(1) | 39(1) |
| O(5) | 7001(1) | 5664(1) | 4098(1) | 35(1) |
| O(6) | 4601(2) | 5137(1) | 4849(2) | 37(1) |
| O(7) | 6078(1) | 7504(1) | 6978(1) | 17(1) |
| O(8) | 4939(1) | 8521(1) | 6637(1) | 18(1) |
| N(1) | 6529(1) | 8785(1) | 6091(1) | 18(1) |
| C(1) | 3416(2) | 8013(2) | 5703(2) | 22(1) |
| C(2) | 3594(2) | 6846(2) | 4532(2) | 34(1) |
| C(3) | 5175(2) | 6765(2) | 3688(2) | 27(1) |
| C(4) | 6175(2) | 7815(2) | 3389(2) | 26(1) |
| C(5) | 6528(2) | 5924(2) | 4542(2) | 23(1) |
| C(6) | 5051(2) | 5611(2) | 4997(2) | 24(1) |
| C(7) | 5802(2) | 7919(1) | 7599(2) | 17(1) |
| C(8) | 6049(2) | 7803(1) | 8409(2) | 19(1) |
| C(9) | 5754(2) | 8273(2) | 9029(2) | 23(1) |
| C(10) | 5218(2) | 8859(2) | 8835(2) | 23(1) |
| C(11) | 4959(2) | 8970(2) | 8026(2) | 21(1) |
| C(12) | 5212(2) | 8480(1) | 7410(2) | 17(1) |
| C(13) | 7319(2) | 8659(1) | 6339(2) | 22(1) |
| C(14) | 7885(2) | 9221(2) | 6510(2) | 26(1) |
| C(15) | 7634(2) | 9943(2) | 6411(2) | 28(1) |
| C(16) | 6816(2) | 10088(2) | 6172(2) | 28(1) |
| C(17) | 6278(2) | 9496(2) | 6027(2) | 24(1) |
| C(18) | 3141(2) | 9256(2) | 4310(2) | 32(1) |
| C(19) | 2824(4) | 9813(3) | 3701(3) | 83(2) |
| C(20) | 2455(3) | 10496(2) | 4021(3) | 54(1) |
| C(21) | 3991(4) | 8523(3) | 2955(3) | 79(2) |
| C(22) | 3970(4) | 7913(3) | 2385(3) | 72(1) |
| C(23) | 4590(4) | 8140(5) | 1692(3) | 120(3) |
| C(24) | 2390(3) | 7920(3) | 3678(3) | 64(2) |

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| | | | | |
|---|---|---|---|---|
| C(25) | 1774(2) | 7782(2) | 4371(4) | 65(2) |
| C(26) | 1085(3) | 7236(3) | 4120(5) | 92(2) |
| C(27) | 5673(2) | 5744(2) | 7237(2) | 23(1) |
| C(28) | 4879(2) | 5268(2) | 7153(2) | 28(1) |
| C(29) | 4272(2) | 5396(2) | 7850(2) | 43(1) |
| C(30) | 6571(2) | 4778(2) | 6120(2) | 28(1) |
| C(31) | 6950(2) | 4340(2) | 6834(2) | 36(1) |
| C(32) | 7263(3) | 3587(2) | 6562(3) | 64(1) |
| C(33) | 7344(2) | 6134(2) | 6692(2) | 22(1) |
| C(34) | 8092(2) | 6022(2) | 6110(2) | 29(1) |
| C(35) | 8856(2) | 6452(2) | 6396(2) | 35(1) |

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

| | | | |
|---|---|---|---|
| Mo(1)-O(8) | 2.0531(18) | C(15)-C(16) | 1.384(4) |
| Mo(1)-O(7) | 2.0684(18) | C(16)-C(17) | 1.388(4) |
| Mo(1)-S(3) | 2.2651(6) | C(18)-C(19) | 1.502(5) |
| Mo(1)-S(2) | 2.3179(6) | C(19)-C(20) | 1.460(6) |
| Mo(1)-S(1) | 2.3395(6) | C(21)-C(22) | 1.441(7) |
| Mo(1)-N(1) | 2.364(2) | C(22)-C(23) | 1.556(8) |
| Mo(1)-Fe(2) | 2.6737(4) | C(24)-C(25) | 1.517(8) |
| Mo(1)-Fe(1) | 2.7793(4) | C(25)-C(26) | 1.529(6) |
| Mo(1)-Fe(3) | 2.8819(4) | C(27)-C(28) | 1.533(4) |
| Fe(1)-C(2) | 1.793(3) | C(28)-C(29) | 1.510(5) |
| Fe(1)-C(1) | 1.803(3) | C(30)-C(31) | 1.531(4) |
| Fe(1)-S(1) | 2.2077(8) | C(31)-C(32) | 1.511(5) |
| Fe(1)-P(1) | 2.2828(9) | C(33)-C(34) | 1.536(4) |
| Fe(1)-S(3) | 2.2968(8) | C(34)-C(35) | 1.516(4) |
| Fe(1)-Fe(2) | 2.6615(5) | | |
| Fe(2)-C(3) | 1.770(3) | O(8)-Mo(1)-O(7) | 79.60(7) |
| Fe(2)-C(4) | 1.773(3) | O(8)-Mo(1)-S(3) | 98.53(5) |
| Fe(2)-S(1) | 2.1800(7) | O(7)-Mo(1)-S(3) | 91.89(5) |
| Fe(2)-S(2) | 2.2030(7) | O(8)-Mo(1)-S(2) | 158.55(5) |
| Fe(2)-Fe(3) | 2.6155(5) | O(7)-Mo(1)-S(2) | 92.58(5) |
| Fe(3)-C(5) | 1.782(3) | S(3)-Mo(1)-S(2) | 101.69(2) |
| Fe(3)-C(6) | 1.787(3) | O(8)-Mo(1)-S(1) | 89.03(5) |
| Fe(3)-S(2) | 2.2455(7) | O(7)-Mo(1)-S(1) | 162.74(5) |
| Fe(3)-P(2) | 2.2626(8) | S(3)-Mo(1)-S(1) | 102.70(2) |
| Fe(3)-S(3) | 2.3244(7) | S(2)-Mo(1)-S(1) | 93.42(2) |
| Cl(1)-C(8) | 1.722(3) | O(8)-Mo(1)-N(1) | 75.81(7) |
| Cl(2)-C(9) | 1.723(3) | O(7)-Mo(1)-N(1) | 78.64(7) |
| Cl(3)-C(10) | 1.727(3) | S(3)-Mo(1)-N(1) | 169.65(5) |
| Cl(4)-C(11) | 1.721(3) | S(2)-Mo(1)-N(1) | 83.10(6) |
| P(1)-C(18) | 1.818(3) | S(1)-Mo(1)-N(1) | 86.03(5) |
| P(1)-C(24) | 1.827(4) | O(8)-Mo(1)-Fe(2) | 138.81(5) |
| P(1)-C(21) | 1.936(6) | O(7)-Mo(1)-Fe(2) | 141.59(5) |
| P(2)-C(27) | 1.826(3) | S(3)-Mo(1)-Fe(2) | 83.44(2) |
| P(2)-C(30) | 1.835(3) | S(2)-Mo(1)-Fe(2) | 51.781(17) |
| P(2)-C(33) | 1.833(3) | S(1)-Mo(1)-Fe(2) | 51.020(18) |
| O(1)-C(1) | 1.137(3) | N(1)-Mo(1)-Fe(2) | 106.57(5) |
| O(2)-C(2) | 1.151(4) | O(8)-Mo(1)-Fe(1) | 90.00(5) |
| O(3)-C(3) | 1.162(4) | O(7)-Mo(1)-Fe(1) | 141.64(5) |
| O(4)-C(4) | 1.144(4) | S(3)-Mo(1)-Fe(1) | 52.986(19) |
| O(5)-C(5) | 1.145(3) | S(2)-Mo(1)-Fe(1) | 107.881(19) |
| O(6)-C(6) | 1.141(4) | S(1)-Mo(1)-Fe(1) | 50.201(19) |
| O(7)-C(7) | 1.334(3) | N(1)-Mo(1)-Fe(1) | 134.55(5) |
| O(8)-C(12) | 1.335(3) | Fe(2)-Mo(1)-Fe(1) | 58.390(12) |
| N(1)-C(13) | 1.340(3) | O(8)-Mo(1)-Fe(3) | 149.23(5) |
| N(1)-C(17) | 1.346(3) | O(7)-Mo(1)-Fe(3) | 91.44(5) |
| C(7)-C(8) | 1.395(4) | S(3)-Mo(1)-Fe(3) | 52.026(17) |
| C(7)-C(12) | 1.413(4) | S(2)-Mo(1)-Fe(3) | 49.730(17) |
| C(8)-C(9) | 1.400(4) | S(1)-Mo(1)-Fe(3) | 104.763(19) |
| C(9)-C(10) | 1.394(4) | N(1)-Mo(1)-Fe(3) | 131.53(5) |
| C(10)-C(11) | 1.397(4) | Fe(2)-Mo(1)-Fe(3) | 56.020(11) |
| C(11)-C(12) | 1.398(4) | Fe(1)-Mo(1)-Fe(3) | 78.869(12) |
| C(13)-C(14) | 1.384(4) | C(2)-Fe(1)-C(1) | 105.16(13) |
| C(14)-C(15) | 1.371(4) | C(2)-Fe(1)-S(1) | 147.52(11) |

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| | | | |
|---|---|---|---|
| C(1)-Fe(1)-S(1) | 105.72(9) | S(2)-Fe(3)-Mo(1) | 51.962(17) |
| C(2)-Fe(1)-P(1) | 87.21(11) | P(2)-Fe(3)-Mo(1) | 100.41(2) |
| C(1)-Fe(1)-P(1) | 89.02(9) | S(3)-Fe(3)-Mo(1) | 50.189(16) |
| S(1)-Fe(1)-P(1) | 83.49(3) | Fe(2)-Fe(3)-Mo(1) | 57.959(12) |
| C(2)-Fe(1)-S(3) | 84.77(11) | Fe(2)-S(1)-Fe(1) | 74.68(2) |
| C(1)-Fe(1)-S(3) | 88.31(9) | Fe(2)-S(1)-Mo(1) | 72.44(2) |
| S(1)-Fe(1)-S(3) | 105.94(3) | Fe(1)-S(1)-Mo(1) | 75.29(2) |
| P(1)-Fe(1)-S(3) | 170.57(4) | Fe(2)-S(2)-Fe(3) | 72.02(2) |
| C(2)-Fe(1)-Fe(2) | 100.55(10) | Fe(2)-S(2)-Mo(1) | 72.46(2) |
| C(1)-Fe(1)-Fe(2) | 152.00(9) | Fe(3)-S(2)-Mo(1) | 78.31(2) |
| S(1)-Fe(1)-Fe(2) | 52.18(2) | Mo(1)-S(3)-Fe(1) | 75.07(2) |
| P(1)-Fe(1)-Fe(2) | 103.26(3) | Mo(1)-S(3)-Fe(3) | 77.78(2) |
| S(3)-Fe(1)-Fe(2) | 83.12(2) | Fe(1)-S(3)-Fe(3) | 102.21(3) |
| C(2)-Fe(1)-Mo(1) | 131.62(10) | C(18)-P(1)-C(24) | 104.36(16) |
| C(1)-Fe(1)-Mo(1) | 95.15(8) | C(18)-P(1)-C(21) | 101.53(19) |
| S(1)-Fe(1)-Mo(1) | 54.505(18) | C(24)-P(1)-C(21) | 106.2(2) |
| P(1)-Fe(1)-Mo(1) | 137.36(3) | C(18)-P(1)-Fe(1) | 113.06(10) |
| S(3)-Fe(1)-Mo(1) | 51.948(16) | C(24)-P(1)-Fe(1) | 113.93(17) |
| Fe(2)-Fe(1)-Mo(1) | 58.819(12) | C(21)-P(1)-Fe(1) | 116.35(13) |
| C(3)-Fe(2)-C(4) | 90.73(14) | C(27)-P(2)-C(30) | 104.66(13) |
| C(3)-Fe(2)-S(1) | 130.86(10) | C(27)-P(2)-C(33) | 104.37(13) |
| C(4)-Fe(2)-S(1) | 102.13(10) | C(30)-P(2)-C(33) | 104.01(13) |
| C(3)-Fe(2)-S(2) | 122.33(10) | C(27)-P(2)-Fe(3) | 115.30(10) |
| C(4)-Fe(2)-S(2) | 101.75(10) | C(30)-P(2)-Fe(3) | 111.86(10) |
| S(1)-Fe(2)-S(2) | 101.34(3) | C(33)-P(2)-Fe(3) | 115.43(9) |
| C(3)-Fe(2)-Fe(3) | 76.47(10) | C(7)-O(7)-Mo(1) | 112.69(15) |
| C(4)-Fe(2)-Fe(3) | 134.30(10) | C(12)-O(8)-Mo(1) | 112.75(15) |
| S(1)-Fe(2)-Fe(3) | 119.54(2) | C(13)-N(1)-C(17) | 117.6(2) |
| S(2)-Fe(2)-Fe(3) | 54.75(2) | C(13)-N(1)-Mo(1) | 122.06(17) |
| C(3)-Fe(2)-Fe(1) | 85.75(10) | C(17)-N(1)-Mo(1) | 120.16(18) |
| C(4)-Fe(2)-Fe(1) | 137.40(10) | O(1)-C(1)-Fe(1) | 178.0(2) |
| S(1)-Fe(2)-Fe(1) | 53.13(2) | O(2)-C(2)-Fe(1) | 177.6(3) |
| S(2)-Fe(2)-Fe(1) | 115.87(2) | O(3)-C(3)-Fe(2) | 173.9(3) |
| Fe(3)-Fe(2)-Fe(1) | 85.935(16) | O(4)-C(4)-Fe(2) | 178.8(3) |
| C(3)-Fe(2)-Mo(1) | 131.51(10) | O(5)-C(5)-Fe(3) | 179.2(3) |
| C(4)-Fe(2)-Mo(1) | 137.53(10) | O(6)-C(6)-Fe(3) | 178.2(3) |
| S(1)-Fe(2)-Mo(1) | 56.537(19) | O(7)-C(7)-C(8) | 122.8(2) |
| S(2)-Fe(2)-Mo(1) | 55.757(18) | O(7)-C(7)-C(12) | 117.0(2) |
| Fe(3)-Fe(2)-Mo(1) | 66.022(13) | C(8)-C(7)-C(12) | 120.1(2) |
| Fe(1)-Fe(2)-Mo(1) | 62.791(12) | C(7)-C(8)-C(9) | 120.0(2) |
| C(5)-Fe(3)-C(6) | 90.18(12) | C(7)-C(8)-Cl(1) | 118.9(2) |
| C(5)-Fe(3)-S(2) | 82.28(9) | C(9)-C(8)-Cl(1) | 121.1(2) |
| C(6)-Fe(3)-S(2) | 164.22(9) | C(10)-C(9)-C(8) | 120.0(2) |
| C(5)-Fe(3)-P(2) | 92.65(9) | C(10)-C(9)-Cl(2) | 120.5(2) |
| C(6)-Fe(3)-P(2) | 94.85(9) | C(8)-C(9)-Cl(2) | 119.5(2) |
| S(2)-Fe(3)-P(2) | 99.31(3) | C(9)-C(10)-C(11) | 120.2(2) |
| C(5)-Fe(3)-S(3) | 169.12(9) | C(9)-C(10)-Cl(3) | 120.0(2) |
| C(6)-Fe(3)-S(3) | 83.10(9) | C(11)-C(10)-Cl(3) | 119.8(2) |
| S(2)-Fe(3)-S(3) | 102.09(3) | C(12)-C(11)-C(10) | 120.3(2) |
| P(2)-Fe(3)-S(3) | 96.42(3) | C(12)-C(11)-Cl(4) | 118.4(2) |
| C(5)-Fe(3)-Fe(2) | 91.28(9) | C(10)-C(11)-Cl(4) | 121.3(2) |
| C(6)-Fe(3)-Fe(2) | 113.47(9) | O(8)-C(12)-C(11) | 123.4(2) |
| S(2)-Fe(3)-Fe(2) | 53.238(19) | O(8)-C(12)-C(7) | 117.6(2) |
| P(2)-Fe(3)-Fe(2) | 151.39(2) | C(11)-C(12)-C(7) | 119.0(2) |
| S(3)-Fe(3)-Fe(2) | 83.63(2) | N(1)-C(13)-C(14) | 123.1(2) |
| C(5)-Fe(3)-Mo(1) | 133.72(9) | C(15)-C(14)-C(13) | 118.8(3) |
| C(6)-Fe(3)-Mo(1) | 131.91(9) | C(14)-C(15)-C(16) | 119.1(3) |

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| | | | |
|---|---|---|---|
| C(17)-C(16)-C(15) | 118.9(3) | C(26)-C(25)-C(24) | 111.6(4) |
| N(1)-C(17)-C(16) | 122.4(3) | C(28)-C(27)-P(2) | 114.5(2) |
| C(19)-C(18)-P(1) | 119.5(3) | C(29)-C(28)-C(27) | 112.0(3) |
| C(20)-C(19)-C(18) | 117.5(4) | C(31)-C(30)-P(2) | 115.8(2) |
| C(22)-C(21)-P(1) | 113.5(5) | C(32)-C(31)-C(30) | 111.6(3) |
| C(21)-C(22)-C(23) | 104.8(6) | C(34)-C(33)-P(2) | 115.2(2) |
| C(25)-C(24)-P(1) | 116.1(3) | C(35)-C(34)-C(33) | 111.4(3) |

Symmetry transformations used to generate equivalent atoms:

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Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **VIa**. 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 |
|---|---|---|---|---|---|---|
| Mo(1) | 15(1) | 15(1) | 11(1) | 0(1) | -1(1) | 1(1) |
| Fe(1) | 17(1) | 26(1) | 16(1) | -5(1) | -3(1) | 3(1) |
| Fe(2) | 20(1) | 21(1) | 12(1) | 0(1) | 0(1) | 2(1) |
| Fe(3) | 17(1) | 17(1) | 16(1) | -1(1) | 1(1) | 0(1) |
| Cl(1) | 31(1) | 30(1) | 19(1) | 1(1) | -7(1) | 9(1) |
| Cl(2) | 46(1) | 52(1) | 14(1) | -5(1) | -5(1) | 10(1) |
| Cl(3) | 44(1) | 41(1) | 24(1) | -17(1) | 2(1) | 9(1) |
| Cl(4) | 43(1) | 35(1) | 33(1) | -11(1) | -8(1) | 23(1) |
| S(1) | 24(1) | 20(1) | 15(1) | 2(1) | -3(1) | 4(1) |
| S(2) | 16(1) | 19(1) | 16(1) | 0(1) | 1(1) | 1(1) |
| S(3) | 16(1) | 20(1) | 19(1) | -1(1) | 2(1) | -1(1) |
| P(1) | 32(1) | 60(1) | 17(1) | -9(1) | -9(1) | 25(1) |
| P(2) | 21(1) | 17(1) | 20(1) | 2(1) | 0(1) | 1(1) |
| O(1) | 29(1) | 30(1) | 30(1) | -6(1) | 8(1) | 2(1) |
| O(2) | 37(1) | 47(1) | 74(2) | -30(1) | -16(1) | -3(1) |
| O(3) | 50(1) | 34(1) | 26(1) | -10(1) | -2(1) | -5(1) |
| O(4) | 49(1) | 45(1) | 23(1) | 8(1) | 9(1) | -6(1) |
| O(5) | 32(1) | 37(1) | 36(1) | -14(1) | 10(1) | 1(1) |
| O(6) | 36(1) | 33(1) | 41(1) | -9(1) | 0(1) | -12(1) |
| O(7) | 20(1) | 18(1) | 12(1) | -1(1) | 0(1) | 2(1) |
| O(8) | 18(1) | 22(1) | 14(1) | -1(1) | -1(1) | 4(1) |
| N(1) | 23(1) | 17(1) | 14(1) | 1(1) | 0(1) | 0(1) |
| C(1) | 19(1) | 23(1) | 26(2) | -1(1) | -5(1) | 2(1) |
| C(2) | 23(1) | 43(2) | 36(2) | -11(1) | -7(1) | 8(1) |
| C(3) | 30(2) | 30(1) | 20(1) | 0(1) | 2(1) | 1(1) |
| C(4) | 28(2) | 28(2) | 22(2) | 0(1) | -2(1) | 1(1) |
| C(5) | 26(1) | 21(1) | 22(1) | -4(1) | -1(1) | -3(1) |
| C(6) | 26(1) | 27(1) | 20(1) | -1(1) | 1(1) | 3(1) |
| C(7) | 17(1) | 18(1) | 15(1) | 1(1) | 2(1) | -4(1) |
| C(8) | 19(1) | 21(1) | 15(1) | 0(1) | 0(1) | 3(1) |
| C(9) | 24(1) | 29(1) | 15(1) | -3(1) | -1(1) | -2(1) |
| C(10) | 25(1) | 26(1) | 18(1) | -10(1) | 1(1) | 0(1) |
| C(11) | 22(1) | 23(1) | 19(1) | -2(1) | 1(1) | 5(1) |
| C(12) | 15(1) | 20(1) | 16(1) | -1(1) | 1(1) | 0(1) |
| C(13) | 23(1) | 17(1) | 25(1) | 2(1) | -4(1) | 3(1) |
| C(14) | 27(1) | 26(1) | 25(1) | -1(1) | -8(1) | -5(1) |
| C(15) | 34(2) | 24(1) | 25(1) | 1(1) | -4(1) | -7(1) |
| C(16) | 36(2) | 19(1) | 28(1) | 1(1) | -3(1) | 0(1) |
| C(17) | 26(1) | 23(1) | 21(1) | 2(1) | -3(1) | 3(1) |
| C(18) | 25(1) | 40(2) | 30(2) | 9(1) | -3(1) | 6(1) |
| C(19) | 118(4) | 97(4) | 34(2) | 2(2) | -2(2) | 82(4) |
| C(20) | 43(2) | 45(2) | 76(3) | 31(2) | -12(2) | -2(2) |
| C(21) | 95(4) | 104(4) | 40(2) | -11(2) | -26(2) | 71(3) |
| C(22) | 80(3) | 90(4) | 47(3) | -11(2) | -6(2) | 23(3) |
| C(23) | 62(3) | 253(10) | 45(3) | 49(4) | 21(2) | 66(5) |
| C(24) | 48(2) | 75(3) | 69(3) | -46(2) | -41(2) | 36(2) |
| C(25) | 29(2) | 61(3) | 105(4) | -47(3) | -28(2) | 13(2) |

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| | | | | | | |
|---|---|---|---|---|---|---|
| C(26) | 50(3) | 70(3) | 155(6) | -49(4) | -54(3) | 17(2) |
| C(27) | 26(1) | 24(1) | 20(1) | 4(1) | 1(1) | -3(1) |
| C(28) | 29(2) | 25(1) | 29(2) | 5(1) | -1(1) | -6(1) |
| C(29) | 39(2) | 53(2) | 36(2) | 11(2) | 10(2) | -9(2) |
| C(30) | 29(2) | 18(1) | 35(2) | 0(1) | 2(1) | 3(1) |
| C(31) | 39(2) | 26(2) | 44(2) | 6(1) | -1(2) | 7(1) |
| C(32) | 80(3) | 36(2) | 75(3) | 11(2) | 4(2) | 31(2) |
| C(33) | 20(1) | 22(1) | 24(1) | 1(1) | -5(1) | 1(1) |
| C(34) | 22(1) | 31(2) | 33(2) | -1(1) | 1(1) | 3(1) |
| C(35) | 24(2) | 37(2) | 44(2) | 3(1) | -4(1) | -3(1) |

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

| | |
|---|---|
| Empirical formula | C39 H63 Cl4 Fe3 Mo O8 P3 S3 |
| Formula weight | 1254.27 |
| Temperature | 158(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Monoclinic, C2/c |
| Unit cell dimensions | a = 47.0226(4) A alpha = 90 deg.<br>b = 12.67300(10)A beta = 97.3650(10)deg.<br>c = 18.69580(10) A gamma = 90 deg. |
| Volume | 11049.23(14) A^3 |
| Z, Calculated density | 8, 1.508 Mg/m^3 |
| Absorption coefficient | 1.430 mm^-1 |
| F(000) | 5136 |
| Crystal size | 0.08 x 0.10 x 0.28 mm |
| Theta range for data collection | 2.20 to 26.43 deg. |
| Limiting indices | -58<=h<=58, -15<=k<=15, -23<=l<=23 |
| Reflections collected / unique | 97633 / 11247 [R(int) = 0.0588] |
| Completeness to theta = 26.43 | 98.9 % |
| Absorption correction | None |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 11247 / 0 / 551 |
| Goodness-of-fit on F^2 | 1.211 |
| Final R indices [I>2sigma(I)] | R1 = 0.0464, wR2 = 0.1409 |
| R indices (all data) | R1 = 0.0589, wR2 = 0.1536 |
| Largest diff. peak and hole | 3.181 and -1.159 e.A^-3 |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Mo(1) | 1214(1) | 7357(1) | 4244(1) | 21(1) |
| Fe(1) | 1564(1) | 9084(1) | 4630(1) | 21(1) |
| Fe(2) | 1694(1) | 7654(1) | 3637(1) | 22(1) |
| Fe(3) | 1223(1) | 8378(1) | 2877(1) | 21(1) |
| Cl(1) | 173(1) | 6251(1) | 3710(1) | 42(1) |
| Cl(2) | -150(1) | 6406(1) | 5095(1) | 47(1) |
| Cl(3) | 182(1) | 7237(1) | 6555(1) | 45(1) |
| Cl(4) | 841(1) | 7834(1) | 6627(1) | 38(1) |
| S(1) | 1690(1) | 7417(1) | 4796(1) | 24(1) |
| S(2) | 1118(1) | 9093(1) | 3953(1) | 22(1) |
| S(3) | 1325(1) | 6681(1) | 3156(1) | 23(1) |
| P(1) | 1278(1) | 5356(1) | 4683(1) | 37(1) |
| P(2) | 1988(1) | 9388(1) | 5333(1) | 26(1) |
| P(3) | 756(1) | 8263(1) | 2381(1) | 23(1) |
| O(1) | 1287(1) | 10614(2) | 2499(2) | 36(1) |
| O(2) | 1429(1) | 7784(2) | 1514(2) | 40(1) |
| O(3) | 1941(1) | 9324(2) | 2811(2) | 40(1) |
| O(4) | 2171(1) | 6257(3) | 3397(2) | 56(1) |
| O(5) | 1259(1) | 9904(2) | 5802(2) | 37(1) |
| O(6) | 1654(1) | 11093(2) | 3887(2) | 40(1) |
| O(7) | 786(1) | 6902(2) | 3993(1) | 29(1) |
| O(8) | 1070(1) | 7507(2) | 5240(1) | 27(1) |
| C(1) | 1265(1) | 9745(3) | 2651(2) | 27(1) |
| C(2) | 1344(1) | 8006(3) | 2048(2) | 28(1) |
| C(3) | 1834(1) | 8686(3) | 3140(2) | 30(1) |
| C(4) | 1984(1) | 6816(3) | 3494(2) | 32(1) |
| C(5) | 1381(1) | 9551(3) | 5362(2) | 28(1) |
| C(6) | 1625(1) | 10297(3) | 4162(2) | 28(1) |
| C(7) | 639(1) | 6949(3) | 4562(2) | 26(1) |
| C(8) | 350(1) | 6694(3) | 4532(2) | 30(1) |
| C(9) | 207(1) | 6777(3) | 5145(2) | 33(1) |
| C(10) | 356(1) | 7142(3) | 5793(2) | 31(1) |
| C(11) | 646(1) | 7396(3) | 5831(2) | 28(1) |
| C(12) | 789(1) | 7298(3) | 5229(2) | 26(1) |
| C(13) | 1607(1) | 4737(3) | 4418(3) | 39(1) |
| C(14) | 1689(1) | 3625(4) | 4624(3) | 54(1) |
| C(15) | 1977(1) | 3334(4) | 4355(3) | 52(1) |
| C(16) | 1303(1) | 5145(4) | 5669(2) | 45(1) |
| C(17) | 1541(1) | 5672(5) | 6150(3) | 65(2) |
| C(18) | 1541(2) | 5395(7) | 6946(3) | 110(3) |
| C(19) | 986(1) | 4434(4) | 4376(3) | 50(1) |
| C(20) | 925(1) | 4234(5) | 3584(3) | 59(1) |
| C(21) | 677(2) | 3426(6) | 3429(4) | 85(2) |
| C(22) | 2071(1) | 8574(3) | 6141(2) | 33(1) |
| C(23) | 1840(1) | 8542(4) | 6650(2) | 37(1) |
| C(24) | 1953(1) | 7974(5) | 7354(3) | 55(1) |

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| | | | | |
|---|---|---|---|---|
| C(25) | 2312(1) | 9195(3) | 4895(2) | 36(1) |
| C(26) | 2370(1) | 10039(4) | 4341(2) | 39(1) |
| C(27) | 2617(1) | 9674(5) | 3913(3) | 61(2) |
| C(28) | 2009(1) | 10745(3) | 5686(2) | 35(1) |
| C(29) | 2267(1) | 11023(4) | 6235(3) | 45(1) |
| C(30) | 2272(1) | 12204(4) | 6423(3) | 63(2) |
| C(31) | 699(1) | 8887(3) | 1487(2) | 32(1) |
| C(32) | 393(1) | 8846(4) | 1098(2) | 42(1) |
| C(33) | 377(1) | 9353(6) | 347(3) | 70(2) |
| C(34) | 492(1) | 8906(3) | 2882(2) | 28(1) |
| C(35) | 508(1) | 10117(3) | 2894(2) | 33(1) |
| C(36) | 301(1) | 10593(4) | 3360(3) | 54(1) |
| C(37) | 613(1) | 6923(3) | 2233(2) | 30(1) |
| C(38) | 741(1) | 6287(3) | 1653(2) | 37(1) |
| C(39) | 611(1) | 5188(4) | 1561(3) | 48(1) |

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

| | | | |
|---|---|---|---|
| Mo(1)-O(8) | 2.068(3) | C(14)-C(15) | 1.546(7) |
| Mo(1)-O(7) | 2.090(2) | C(16)-C(17) | 1.500(7) |
| Mo(1)-S(2) | 2.2979(9) | C(17)-C(18) | 1.528(7) |
| Mo(1)-S(3) | 2.3278(9) | C(19)-C(20) | 1.494(7) |
| Mo(1)-S(1) | 2.3427(9) | C(20)-C(21) | 1.552(7) |
| Mo(1)-P(1) | 2.6706(12) | C(22)-C(23) | 1.533(6) |
| Mo(1)-Fe(2) | 2.6820(6) | C(23)-C(24) | 1.534(6) |
| Mo(1)-Fe(1) | 2.7789(6) | C(25)-C(26) | 1.538(6) |
| Mo(1)-Fe(3) | 2.8704(6) | C(26)-C(27) | 1.561(6) |
| Fe(1)-C(5) | 1.808(4) | C(28)-C(29) | 1.529(6) |
| Fe(1)-C(6) | 1.809(4) | C(29)-C(30) | 1.537(7) |
| Fe(1)-S(1) | 2.2043(10) | C(31)-C(32) | 1.526(6) |
| Fe(1)-P(2) | 2.2732(10) | C(32)-C(33) | 1.536(7) |
| Fe(1)-S(2) | 2.3095(9) | C(34)-C(35) | 1.537(5) |
| Fe(1)-Fe(2) | 2.7193(7) | C(35)-C(36) | 1.514(6) |
| Fe(2)-C(4) | 1.774(4) | C(37)-C(38) | 1.535(5) |
| Fe(2)-C(3) | 1.777(4) | C(38)-C(39) | 1.522(6) |
| Fe(2)-S(1) | 2.1911(10) | | |
| Fe(2)-S(3) | 2.2231(10) | O(8)-Mo(1)-O(7) | 79.12(10) |
| Fe(2)-Fe(3) | 2.6414(7) | O(8)-Mo(1)-S(2) | 92.72(8) |
| Fe(3)-C(2) | 1.781(4) | O(7)-Mo(1)-S(2) | 93.22(8) |
| Fe(3)-C(1) | 1.801(4) | O(8)-Mo(1)-S(3) | 162.96(8) |
| Fe(3)-S(3) | 2.2512(10) | O(7)-Mo(1)-S(3) | 91.16(8) |
| Fe(3)-P(3) | 2.2731(10) | S(2)-Mo(1)-S(3) | 101.84(3) |
| Fe(3)-S(2) | 2.3176(10) | O(8)-Mo(1)-S(1) | 90.23(8) |
| Cl(1)-C(8) | 1.744(4) | O(7)-Mo(1)-S(1) | 161.22(8) |
| Cl(2)-C(9) | 1.736(4) | S(2)-Mo(1)-S(1) | 102.78(3) |
| Cl(3)-C(10) | 1.734(4) | S(3)-Mo(1)-S(1) | 95.05(3) |
| Cl(4)-C(11) | 1.737(4) | O(8)-Mo(1)-P(1) | 81.27(8) |
| P(1)-C(19) | 1.837(5) | O(7)-Mo(1)-P(1) | 82.79(8) |
| P(1)-C(16) | 1.852(5) | S(2)-Mo(1)-P(1) | 173.31(3) |
| P(1)-C(13) | 1.856(4) | S(3)-Mo(1)-P(1) | 83.66(3) |
| P(2)-C(22) | 1.829(4) | S(1)-Mo(1)-P(1) | 80.31(3) |
| P(2)-C(25) | 1.836(4) | O(8)-Mo(1)-Fe(2) | 139.27(7) |
| P(2)-C(28) | 1.840(4) | O(7)-Mo(1)-Fe(2) | 141.59(7) |
| P(3)-C(37) | 1.835(4) | S(2)-Mo(1)-Fe(2) | 85.22(3) |
| P(3)-C(31) | 1.839(4) | S(3)-Mo(1)-Fe(2) | 52.10(2) |
| P(3)-C(34) | 1.840(4) | S(1)-Mo(1)-Fe(2) | 51.16(3) |
| O(1)-C(1) | 1.144(5) | P(1)-Mo(1)-Fe(2) | 101.25(3) |
| O(2)-C(2) | 1.157(5) | O(8)-Mo(1)-Fe(1) | 86.86(7) |
| O(3)-C(3) | 1.170(5) | O(7)-Mo(1)-Fe(1) | 142.96(8) |
| O(4)-C(4) | 1.160(5) | S(2)-Mo(1)-Fe(1) | 53.10(2) |
| O(5)-C(5) | 1.153(5) | S(3)-Mo(1)-Fe(1) | 108.92(3) |
| O(6)-C(6) | 1.148(5) | S(1)-Mo(1)-Fe(1) | 50.10(2) |
| O(7)-C(7) | 1.342(4) | P(1)-Mo(1)-Fe(1) | 128.93(3) |
| O(8)-C(12) | 1.348(4) | Fe(2)-Mo(1)-Fe(1) | 59.699(16) |
| C(7)-C(8) | 1.391(5) | O(8)-Mo(1)-Fe(3) | 143.46(8) |
| C(7)-C(12) | 1.421(5) | O(7)-Mo(1)-Fe(3) | 92.73(7) |
| C(8)-C(9) | 1.404(5) | S(2)-Mo(1)-Fe(3) | 51.85(2) |
| C(9)-C(10) | 1.399(6) | S(3)-Mo(1)-Fe(3) | 50.00(2) |
| C(10)-C(11) | 1.394(5) | S(1)-Mo(1)-Fe(3) | 104.83(3) |
| C(11)-C(12) | 1.388(5) | P(1)-Mo(1)-Fe(3) | 133.45(3) |
| C(13)-C(14) | 1.499(7) | Fe(2)-Mo(1)-Fe(3) | 56.690(16) |

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| | | | |
|---|---|---|---|
| Fe(1)-Mo(1)-Fe(3) | 78.482(16) | S(2)-Fe(3)-Fe(2) | 85.77(3) |
| C(5)-Fe(1)-C(6) | 101.97(17) | C(2)-Fe(3)-Mo(1) | 134.23(13) |
| C(5)-Fe(1)-S(1) | 110.61(13) | C(1)-Fe(3)-Mo(1) | 131.11(12) |
| C(6)-Fe(1)-S(1) | 145.75(12) | S(3)-Fe(3)-Mo(1) | 52.38(3) |
| C(5)-Fe(1)-P(2) | 88.48(12) | P(3)-Fe(3)-Mo(1) | 102.17(3) |
| C(6)-Fe(1)-P(2) | 87.46(12) | S(2)-Fe(3)-Mo(1) | 51.24(2) |
| S(1)-Fe(1)-P(2) | 83.23(4) | Fe(2)-Fe(3)-Mo(1) | 58.055(16) |
| C(5)-Fe(1)-S(2) | 85.64(12) | Fe(2)-S(1)-Fe(1) | 76.44(3) |
| C(6)-Fe(1)-S(2) | 85.37(12) | Fe(2)-S(1)-Mo(1) | 72.45(3) |
| S(1)-Fe(1)-S(2) | 106.89(4) | Fe(1)-S(1)-Mo(1) | 75.28(3) |
| P(2)-Fe(1)-S(2) | 169.59(4) | Mo(1)-S(2)-Fe(1) | 74.19(3) |
| C(5)-Fe(1)-Fe(2) | 154.91(13) | Mo(1)-S(2)-Fe(3) | 76.91(3) |
| C(6)-Fe(1)-Fe(2) | 99.97(12) | Fe(1)-S(2)-Fe(3) | 101.16(4) |
| S(1)-Fe(1)-Fe(2) | 51.56(3) | Fe(2)-S(3)-Fe(3) | 72.36(3) |
| P(2)-Fe(1)-Fe(2) | 104.57(3) | Fe(2)-S(3)-Mo(1) | 72.18(3) |
| S(2)-Fe(1)-Fe(2) | 84.14(3) | Fe(3)-S(3)-Mo(1) | 77.62(3) |
| C(5)-Fe(1)-Mo(1) | 97.46(12) | C(19)-P(1)-C(16) | 99.7(2) |
| C(6)-Fe(1)-Mo(1) | 131.96(11) | C(19)-P(1)-C(13) | 105.2(2) |
| S(1)-Fe(1)-Mo(1) | 54.62(3) | C(16)-P(1)-C(13) | 104.8(2) |
| P(2)-Fe(1)-Mo(1) | 136.86(3) | C(19)-P(1)-Mo(1) | 117.23(18) |
| S(2)-Fe(1)-Mo(1) | 52.71(2) | C(16)-P(1)-Mo(1) | 115.63(16) |
| Fe(2)-Fe(1)-Mo(1) | 58.380(16) | C(13)-P(1)-Mo(1) | 112.61(15) |
| C(4)-Fe(2)-C(3) | 91.20(18) | C(22)-P(2)-C(25) | 101.07(19) |
| C(4)-Fe(2)-S(1) | 99.86(13) | C(22)-P(2)-C(28) | 103.6(2) |
| C(3)-Fe(2)-S(1) | 131.93(13) | C(25)-P(2)-C(28) | 106.0(2) |
| C(4)-Fe(2)-S(3) | 100.52(13) | C(22)-P(2)-Fe(1) | 117.40(13) |
| C(3)-Fe(2)-S(3) | 121.22(13) | C(25)-P(2)-Fe(1) | 115.72(14) |
| S(1)-Fe(2)-S(3) | 102.59(4) | C(28)-P(2)-Fe(1) | 111.61(14) |
| C(4)-Fe(2)-Fe(3) | 137.37(13) | C(37)-P(3)-C(31) | 104.61(19) |
| C(3)-Fe(2)-Fe(3) | 78.67(13) | C(37)-P(3)-C(34) | 103.20(17) |
| S(1)-Fe(2)-Fe(3) | 117.77(3) | C(31)-P(3)-C(34) | 104.04(18) |
| S(3)-Fe(2)-Fe(3) | 54.31(3) | C(37)-P(3)-Fe(3) | 115.87(13) |
| C(4)-Fe(2)-Mo(1) | 133.18(14) | C(31)-P(3)-Fe(3) | 111.37(13) |
| C(3)-Fe(2)-Mo(1) | 135.33(13) | C(34)-P(3)-Fe(3) | 116.38(13) |
| S(1)-Fe(2)-Mo(1) | 56.39(3) | C(7)-O(7)-Mo(1) | 112.8(2) |
| S(3)-Fe(2)-Mo(1) | 55.72(3) | C(12)-O(8)-Mo(1) | 113.7(2) |
| Fe(3)-Fe(2)-Mo(1) | 65.255(17) | O(1)-C(1)-Fe(3) | 178.5(4) |
| C(4)-Fe(2)-Fe(1) | 138.18(13) | O(2)-C(2)-Fe(3) | 178.1(4) |
| C(3)-Fe(2)-Fe(1) | 89.75(13) | O(3)-C(3)-Fe(2) | 175.4(4) |
| S(1)-Fe(2)-Fe(1) | 52.00(3) | O(4)-C(4)-Fe(2) | 179.0(4) |
| S(3)-Fe(2)-Fe(1) | 114.42(3) | O(5)-C(5)-Fe(1) | 175.7(4) |
| Fe(3)-Fe(2)-Fe(1) | 83.62(2) | O(6)-C(6)-Fe(1) | 176.3(4) |
| Mo(1)-Fe(2)-Fe(1) | 61.922(16) | O(7)-C(7)-C(8) | 123.7(3) |
| C(2)-Fe(3)-C(1) | 89.58(17) | O(7)-C(7)-C(12) | 117.5(3) |
| C(2)-Fe(3)-S(3) | 82.28(13) | C(8)-C(7)-C(12) | 118.7(3) |
| C(1)-Fe(3)-S(3) | 161.20(12) | C(7)-C(8)-C(9) | 121.1(4) |
| C(2)-Fe(3)-P(3) | 91.70(12) | C(7)-C(8)-Cl(1) | 117.7(3) |
| C(1)-Fe(3)-P(3) | 95.57(12) | C(9)-C(8)-Cl(1) | 121.3(3) |
| S(3)-Fe(3)-P(3) | 101.56(4) | C(10)-C(9)-C(8) | 119.6(4) |
| C(2)-Fe(3)-S(2) | 170.45(13) | C(10)-C(9)-Cl(2) | 120.6(3) |
| C(1)-Fe(3)-S(2) | 82.53(12) | C(8)-C(9)-Cl(2) | 119.8(3) |
| S(3)-Fe(3)-S(2) | 103.61(4) | C(11)-C(10)-C(9) | 119.8(3) |
| P(3)-Fe(3)-S(2) | 94.42(4) | C(11)-C(10)-Cl(3) | 120.2(3) |
| C(2)-Fe(3)-Fe(2) | 91.98(12) | C(9)-C(10)-Cl(3) | 119.9(3) |
| C(1)-Fe(3)-Fe(2) | 110.43(11) | C(12)-C(11)-C(10) | 120.7(4) |
| S(3)-Fe(3)-Fe(2) | 53.33(3) | C(12)-C(11)-Cl(4) | 117.7(3) |
| P(3)-Fe(3)-Fe(2) | 153.76(3) | C(10)-C(11)-Cl(4) | 121.5(3) |

| | | | |
|---|---|---|---|
| O(8)-C(12)-C(11) | 123.2(4) | C(26)-C(25)-P(2) | 115.9(3) |
| O(8)-C(12)-C(7) | 116.8(3) | C(25)-C(26)-C(27) | 110.3(4) |
| C(11)-C(12)-C(7) | 120.1(3) | C(29)-C(28)-P(2) | 117.2(3) |
| C(14)-C(13)-P(1) | 121.7(3) | C(28)-C(29)-C(30) | 111.5(4) |
| C(13)-C(14)-C(15) | 110.4(4) | C(32)-C(31)-P(3) | 116.0(3) |
| C(17)-C(16)-P(1) | 118.7(3) | C(31)-C(32)-C(33) | 110.8(4) |
| C(16)-C(17)-C(18) | 112.8(4) | C(35)-C(34)-P(3) | 114.4(3) |
| C(20)-C(19)-P(1) | 117.3(4) | C(36)-C(35)-C(34) | 111.7(4) |
| C(19)-C(20)-C(21) | 109.9(5) | C(38)-C(37)-P(3) | 115.1(3) |
| C(23)-C(22)-P(2) | 115.7(3) | C(39)-C(38)-C(37) | 111.8(4) |
| C(22)-C(23)-C(24) | 110.5(4) | | |

Symmetry transformations used to generate equivalent atoms:

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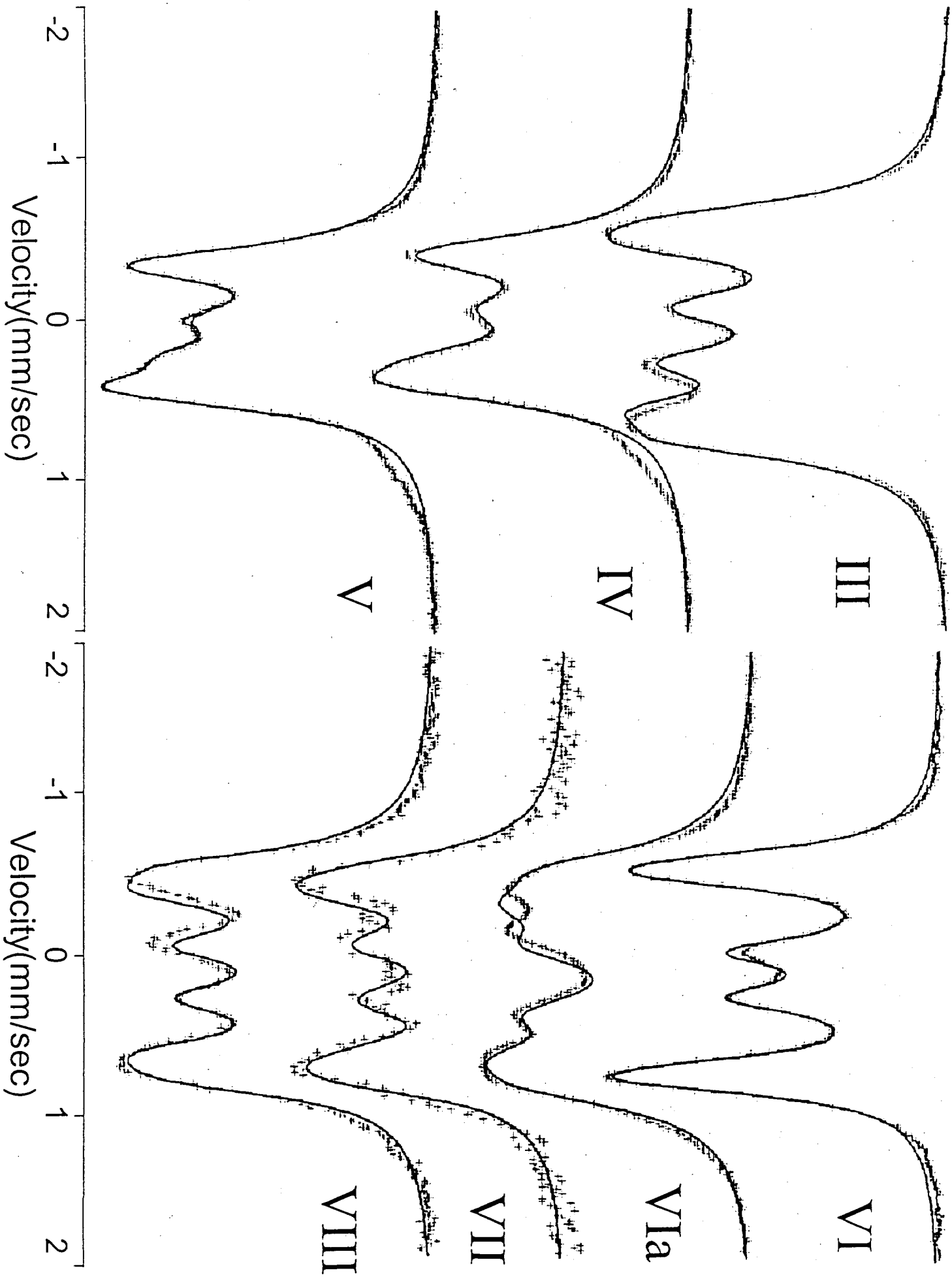
Table 4. Anisotropic displacement parameters (A^2 x 10^3) for VII.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [ h^2 a*^2 U11 + ... + 2 h k a* b* U12 ]

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Mo(1) | 15(1) | 26(1) | 23(1) | 0(1) | 3(1) | -4(1) |
| Fe(1) | 14(1) | 25(1) | 24(1) | -1(1) | 3(1) | -1(1) |
| Fe(2) | 14(1) | 27(1) | 25(1) | 0(1) | 3(1) | 1(1) |
| Fe(3) | 13(1) | 25(1) | 24(1) | 0(1) | 3(1) | 1(1) |
| Cl(1) | 19(1) | 69(1) | 38(1) | 1(1) | 1(1) | -11(1) |
| Cl(2) | 15(1) | 74(1) | 54(1) | 15(1) | 11(1) | 3(1) |
| Cl(3) | 33(1) | 61(1) | 45(1) | 2(1) | 23(1) | 14(1) |
| Cl(4) | 41(1) | 45(1) | 31(1) | -8(1) | 12(1) | -6(1) |
| S(1) | 18(1) | 26(1) | 26(1) | 2(1) | 1(1) | -1(1) |
| S(2) | 14(1) | 28(1) | 25(1) | -1(1) | 2(1) | 2(1) |
| S(3) | 19(1) | 24(1) | 27(1) | -2(1) | 3(1) | -1(1) |
| P(1) | 28(1) | 42(1) | 41(1) | -4(1) | 7(1) | -6(1) |
| P(2) | 15(1) | 33(1) | 30(1) | -2(1) | 1(1) | -4(1) |
| P(3) | 15(1) | 28(1) | 26(1) | -2(1) | 2(1) | 1(1) |
| O(1) | 38(2) | 30(2) | 40(2) | 8(1) | 0(1) | -1(1) |
| O(2) | 39(2) | 50(2) | 33(2) | 1(1) | 13(1) | 6(1) |
| O(3) | 32(2) | 44(2) | 44(2) | 8(1) | 10(1) | -9(1) |
| O(4) | 32(2) | 62(2) | 74(2) | -2(2) | 8(2) | 25(2) |
| O(5) | 33(2) | 47(2) | 34(2) | -7(1) | 12(1) | 4(1) |
| O(6) | 46(2) | 29(2) | 44(2) | 8(1) | 2(1) | -9(1) |
| O(7) | 19(1) | 40(2) | 27(1) | 0(1) | 3(1) | -7(1) |
| O(8) | 20(1) | 38(2) | 25(1) | -1(1) | 5(1) | -7(1) |
| C(1) | 17(2) | 38(2) | 26(2) | -1(2) | 0(1) | -2(2) |
| C(2) | 22(2) | 32(2) | 31(2) | 4(2) | 4(2) | 0(2) |
| C(3) | 22(2) | 34(2) | 35(2) | -3(2) | 5(2) | 0(2) |
| C(4) | 22(2) | 39(2) | 36(2) | 1(2) | 0(2) | 1(2) |
| C(5) | 20(2) | 35(2) | 27(2) | -1(2) | -2(2) | 1(2) |
| C(6) | 20(2) | 34(2) | 29(2) | -6(2) | -2(2) | -4(2) |
| C(7) | 19(2) | 30(2) | 31(2) | 1(2) | 7(2) | 0(2) |
| C(8) | 18(2) | 40(2) | 31(2) | 5(2) | 3(2) | 3(2) |
| C(9) | 20(2) | 38(2) | 43(2) | 11(2) | 10(2) | 6(2) |
| C(10) | 24(2) | 37(2) | 35(2) | 7(2) | 14(2) | 8(2) |
| C(11) | 24(2) | 30(2) | 31(2) | 1(2) | 6(2) | 1(2) |
| C(12) | 20(2) | 25(2) | 33(2) | 2(2) | 8(2) | -1(1) |
| C(13) | 25(2) | 41(2) | 52(3) | -2(2) | 11(2) | -1(2) |
| C(14) | 50(3) | 41(3) | 74(4) | 9(2) | 17(3) | -3(2) |
| C(15) | 42(3) | 40(3) | 73(3) | -6(2) | 7(2) | 6(2) |
| C(16) | 38(3) | 53(3) | 45(3) | 4(2) | 13(2) | -6(2) |
| C(17) | 75(4) | 77(4) | 38(3) | 20(3) | -7(3) | -32(3) |
| C(18) | 143(7) | 134(7) | 45(3) | 35(4) | -19(4) | -90(6) |
| C(19) | 31(2) | 52(3) | 68(3) | -8(2) | 10(2) | -10(2) |
| C(20) | 47(3) | 68(4) | 61(3) | -16(3) | 10(3) | -15(3) |
| C(21) | 73(4) | 107(5) | 77(4) | -39(4) | 19(3) | -51(4) |
| C(22) | 23(2) | 42(2) | 30(2) | -3(2) | -4(2) | 0(2) |
| C(23) | 38(2) | 46(2) | 27(2) | -2(2) | 3(2) | 4(2) |
| C(24) | 67(4) | 66(3) | 33(2) | 8(2) | 9(2) | 16(3) |
| C(25) | 15(2) | 49(2) | 45(2) | -6(2) | 4(2) | -2(2) |

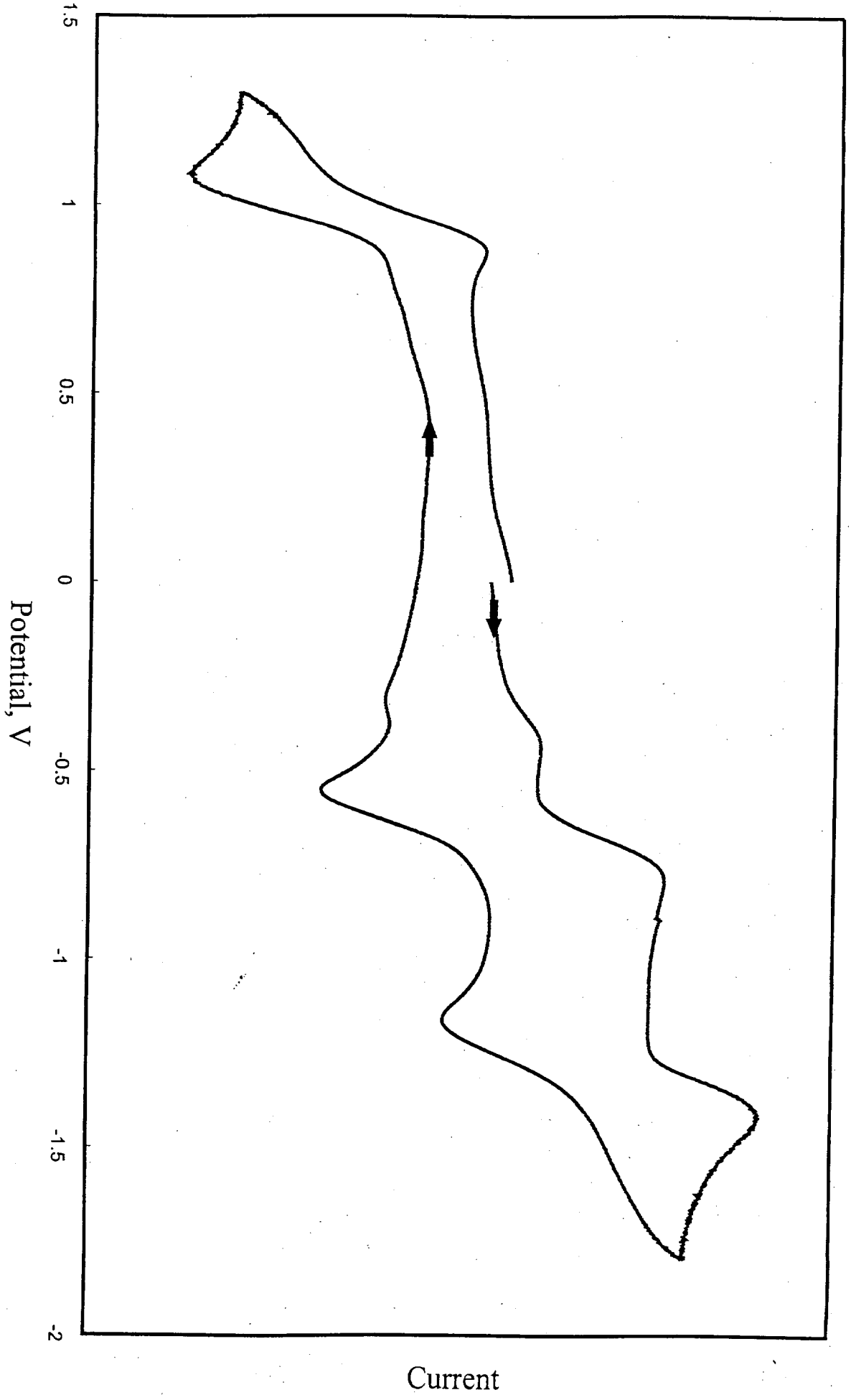
| | | | | | | |
|---|---|---|---|---|---|---|
| C(26) | 22(2) | 52(3) | 46(2) | -8(2) | 9(2) | -10(2) |
| C(27) | 37(3) | 84(4) | 69(3) | 5(3) | 27(3) | -1(3) |
| C(28) | 25(2) | 36(2) | 41(2) | -5(2) | -1(2) | -8(2) |
| C(29) | 30(2) | 59(3) | 46(3) | -12(2) | -1(2) | -16(2) |
| C(30) | 60(4) | 59(3) | 66(4) | -19(3) | -2(3) | -25(3) |
| C(31) | 26(2) | 40(2) | 29(2) | 3(2) | 5(2) | 6(2) |
| C(32) | 30(2) | 54(3) | 40(2) | 0(2) | -5(2) | 10(2) |
| C(33) | 54(3) | 106(5) | 46(3) | 20(3) | -13(3) | 18(3) |
| C(34) | 15(2) | 32(2) | 36(2) | -7(2) | 4(2) | 3(1) |
| C(35) | 20(2) | 34(2) | 45(2) | -3(2) | 4(2) | 8(2) |
| C(36) | 39(3) | 54(3) | 71(3) | -17(3) | 14(2) | 11(2) |
| C(37) | 17(2) | 35(2) | 36(2) | -1(2) | -1(2) | -3(2) |
| C(38) | 25(2) | 38(2) | 48(2) | -12(2) | 3(2) | -3(2) |
| C(39) | 29(2) | 43(3) | 71(3) | -19(2) | 1(2) | -2(2) |

$^{57}$Fe Mossbauer spectra of : $(Cl_4\text{–cat})MoFe_3S_3(Pet_3)_2(CO)_6$(**III**); $(Cl_4\text{–cat})(O)MoFe_3S_3(Pet_3)_3(CO)_5$(**IV**); $(Cl_4\text{–cat})(O)MoFe_3S_3(Pet_3)_2(CO)_6$(**V**); $(Cl_4\text{–cat})(Pyr)MoFe_3S_3(Pet_3)_2(CO)_6$(**VI**); $(Cl_4\text{–cat})(P\,^nPr_3)_2MoFe_3S_3(Pet_3)_2(CO)_6$(**VII**); and $(Cl_4\text{–cat})(Pyr)MoFe_3S_3)(P\,^nPr_3)_3(CO)_4$(**VIII**). The spectra were obtained at 125K in zero applied magnetic field. The source was $^{57}$Co in Rh matrix and the isomer shift is reported vs Fe metal ar ambient temperature. The solid lines represent the fitted spectra as described in the text,

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Cyclic voltammogram of the $(Cl_4\text{-cat})Mo(O)Fe_3S_3(Pet_3)_2(CO)_6$ , (V) cluster.


1.5
1
0.5
0
-0.5
-1
-1.5
-2
Potential, V
Current