

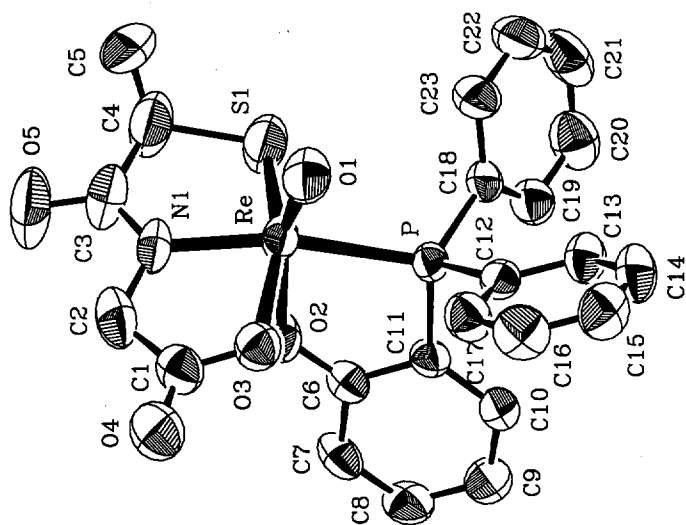


Table 1. Summary of Crystal, Intensity Collection and Refinement Data

|                                                              |                                                                      |
|--------------------------------------------------------------|----------------------------------------------------------------------|
| Empirical formula                                            | C <sub>39</sub> H <sub>55</sub> N <sub>2</sub> O <sub>5</sub> P Re S |
| Formula weight                                               | 881.08                                                               |
| Temperature, K                                               | 298                                                                  |
| Wavelength                                                   | Mo Ka 0.71073                                                        |
| Space group                                                  | P2 <sub>1</sub> /n                                                   |
| a (Å)                                                        | 8.910(3)                                                             |
| b (Å)                                                        | 23.466(8)                                                            |
| c (Å)                                                        | 19.114(8)                                                            |
| β, deg                                                       | 89.99(1)                                                             |
| V, (Å <sup>3</sup> )                                         | 3996(1)                                                              |
| Z                                                            | 4                                                                    |
| D <sub>calcd</sub> /D <sub>measd</sub> (Mg m <sup>-3</sup> ) | 1.464/1.44                                                           |
| abs coeff(μ), mm <sup>-1</sup>                               | 3.176                                                                |
| Abs.correction mode Tmin/Tmax                                | 0.82/1.00                                                            |
| Scan mode / speed (deg/min)                                  | θ-2θ/2.7                                                             |
| Scan range (2θ deg)                                          | 2.3+ α <sub>1</sub> α <sub>2</sub> separation                        |
| θ range(deg)                                                 | 1.74 to 24.00                                                        |
| Reflections collected                                        | 6710                                                                 |
| Independent reflections                                      | 6268 [R(int) = 0.0186]                                               |
| Range of h,k,l                                               | 0→10, 0→26, -21→21                                                   |
| F(000)                                                       | 1796                                                                 |
| [Δ/σ] <sub>max</sub>                                         | 0.105                                                                |
| W <sup>(1)</sup>                                             | a=0.0118 b=7.8702                                                    |
| [Δρ] <sub>max</sub> /[Δρ] <sub>min</sub> (e/Å <sup>3</sup> ) | 0.706 and -0.575                                                     |
| Refinement method                                            | Full-matrix least-squares on F <sup>2</sup>                          |
| Data / restraints / parameters                               | 6268 / 0 / 504                                                       |
| Goodness-of-fit on F <sup>2</sup>                            | 1.186                                                                |
| R indices [5045 refs I>2σ(I)] <sup>(2)</sup>                 | R1 = 0.0410, wR2 = 0.0918                                            |
| R indices (all data)                                         | R1 = 0.0579, wR2 = 0.1002                                            |

(1)  $W=1/[\sigma^2(F_o^2)+(a*P)^2+b*P]$  and  $P=\frac{1}{2}(\text{Max}(F_o^2,0)+2*F_c^2)/3$   
(2) R1 based on F's, wR2 based on F<sup>2</sup>

Table 2. Positional ( $\times 10^3$ ) and equivalent thermal parameters ( $\times 10^3$ ) of the non-H atoms. E.s.d.'s in parentheses. U(eq) is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

|       | x         | y         | z        | U(eq)   |
|-------|-----------|-----------|----------|---------|
| Re(1) | 1528(1)   | -1251(1)  | 2584(1)  | 48(1)   |
| O(1)  | 3364(5)   | -1342(3)  | 2386(3)  | 80(2)   |
| O(2)  | -783(5)   | -1179(2)  | 2480(2)  | 57(1)   |
| O(3)  | 933(6)    | -2083(2)  | 2244(2)  | 63(1)   |
| P     | 1201(2)   | -750(1)   | 1478(1)  | 42(1)   |
| S(1)  | 1523(3)   | -429(1)   | 3234(1)  | 81(1)   |
| N(1)  | 1085(7)   | -1632(3)  | 3500(3)  | 59(2)   |
| C(1)  | 533(8)    | -2433(3)  | 2732(4)  | 63(2)   |
| O(4)  | 142(8)    | -2926(2)  | 2617(3)  | 92(2)   |
| C(2)  | 549(11)   | -2215(4)  | 3464(5)  | 75(2)   |
| C(3)  | 1087(11)  | -1354(4)  | 4116(4)  | 84(3)   |
| O(5)  | 677(10)   | -1579(3)  | 4663(3)  | 121(3)  |
| C(4)  | 1632(15)  | -738(4)   | 4117(4)  | 102(3)  |
| C(5)  | 3009(32)  | -695(11)  | 4436(12) | 133(15) |
| C(5A) | 1122(48)  | -376(11)  | 4635(10) | 157(18) |
| C(6)  | -1520(7)  | -1029(3)  | 1915(3)  | 46(1)   |
| C(7)  | -3093(7)  | -1083(3)  | 1865(4)  | 55(2)   |
| C(8)  | -3826(8)  | -943(3)   | 1271(4)  | 63(2)   |
| C(9)  | -3078(9)  | -741(4)   | 695(4)   | 68(2)   |
| C(10) | -1548(8)  | -676(3)   | 723(4)   | 56(2)   |
| C(11) | -763(6)   | -811(3)   | 1331(3)  | 45(1)   |
| C(12) | 2148(7)   | -1038(3)  | 705(3)   | 45(1)   |
| C(13) | 2579(8)   | -688(3)   | 151(3)   | 57(2)   |
| C(14) | 3264(10)  | -917(4)   | -425(4)  | 71(2)   |
| C(15) | 3502(10)  | -1494(4)  | -467(5)  | 73(2)   |
| C(16) | 3095(10)  | -1836(4)  | 67(5)    | 75(2)   |
| C(17) | 2410(9)   | -1617(3)  | 660(4)   | 62(2)   |
| C(18) | 1675(7)   | 10(3)     | 1470(3)  | 44(1)   |
| C(19) | 689(9)    | 420(3)    | 1222(4)  | 57(2)   |
| C(20) | 1102(12)  | 982(4)    | 1205(5)  | 75(2)   |
| C(21) | 2474(13)  | 1148(4)   | 1445(5)  | 83(3)   |
| C(22) | 3433(12)  | 746(4)    | 1707(5)  | 79(3)   |
| C(23) | 3071(8)   | 176(3)    | 1724(4)  | 62(2)   |
| N(2)  | 793(8)    | -1753(3)  | 6805(3)  | 76(2)   |
| C(31) | 1376(13)  | -1232(4)  | 7149(6)  | 107(3)  |
| C(32) | 1814(14)  | -751(5)   | 6698(6)  | 117(4)  |
| C(33) | 2667(17)  | -286(5)   | 7093(7)  | 138(5)  |
| C(34) | 3186(19)  | 167(6)    | 6615(8)  | 181(7)  |
| C(35) | 188(13)   | -2172(5)  | 7345(5)  | 112(3)  |
| C(36) | 1252(13)  | -2423(5)  | 7849(5)  | 106(3)  |
| C(37) | 457(14)   | -2777(5)  | 8382(5)  | 114(4)  |
| C(38) | 1347(18)  | -3126(5)  | 8847(6)  | 143(5)  |
| C(39) | 2113(14)  | -2012(5)  | 6389(6)  | 118(4)  |
| C(40) | 1909(20)  | -2505(6)  | 6002(8)  | 168(7)  |
| C(41) | 3334(25)  | -2724(10) | 5633(12) | 223(9)  |
| C(42) | 3768(27)  | -2601(10) | 5169(11) | 297(15) |
| C(43) | -429(14)  | -1644(5)  | 6285(6)  | 117(4)  |
| C(44) | -1753(14) | -1342(6)  | 6545(7)  | 135(5)  |
| C(45) | -2901(23) | -1264(8)  | 5915(10) | 194(7)  |
| C(46) | -4083(31) | -978(11)  | 6072(13) | 257(11) |

Table 3. Anisotropic thermal parameters ( $\times 10^{-3}$ ) of the non-H atoms.  
E.s.d.'s in parentheses

|       | U11     | U22     | U33     | U23     | U13     | U12     |
|-------|---------|---------|---------|---------|---------|---------|
| Re(1) | 45(1)   | 60(1)   | 40(1)   | 12(1)   | -2(1)   | 2(1)    |
| O(1)  | 42(3)   | 124(5)  | 75(4)   | 30(3)   | -4(2)   | 6(3)    |
| O(2)  | 42(2)   | 78(3)   | 51(3)   | 19(2)   | 8(2)    | 5(2)    |
| O(3)  | 73(3)   | 54(3)   | 60(3)   | 3(2)    | -3(2)   | 0(2)    |
| P     | 35(1)   | 52(1)   | 38(1)   | 8(1)    | 0(1)    | -1(1)   |
| S(1)  | 124(2)  | 69(1)   | 51(1)   | 5(1)    | -8(1)   | -7(1)   |
| N(1)  | 66(4)   | 73(4)   | 39(3)   | 18(3)   | 2(3)    | 9(3)    |
| C(1)  | 59(4)   | 53(4)   | 76(5)   | 13(4)   | -5(4)   | 3(4)    |
| O(4)  | 120(5)  | 61(4)   | 96(4)   | 11(3)   | -8(4)   | -8(3)   |
| C(2)  | 85(6)   | 70(6)   | 71(6)   | 30(4)   | 8(5)    | 4(5)    |
| C(3)  | 115(7)  | 90(7)   | 46(4)   | 15(4)   | -2(4)   | 16(5)   |
| O(5)  | 199(8)  | 113(5)  | 50(3)   | 22(3)   | 20(4)   | 5(5)    |
| C(4)  | 171(11) | 88(7)   | 48(5)   | 9(4)    | -9(6)   | -16(7)  |
| C(5)  | 149(25) | 149(23) | 101(18) | 48(16)  | -77(17) | -64(19) |
| C(5A) | 311(52) | 115(19) | 44(11)  | -3(11)  | 51(18)  | -2(23)  |
| C(6)  | 40(3)   | 41(3)   | 56(4)   | 4(3)    | 5(3)    | 0(3)    |
| C(7)  | 40(4)   | 55(4)   | 71(5)   | 12(3)   | 13(3)   | -5(3)   |
| C(8)  | 40(4)   | 66(5)   | 84(6)   | 12(4)   | -11(4)  | 1(3)    |
| C(9)  | 55(4)   | 78(5)   | 70(5)   | 17(4)   | -16(4)  | -5(4)   |
| C(10) | 52(4)   | 65(5)   | 51(4)   | 13(4)   | -3(3)   | -13(4)  |
| C(11) | 33(3)   | 51(4)   | 51(4)   | 9(3)    | -3(3)   | -4(3)   |
| C(12) | 37(3)   | 57(4)   | 42(3)   | 1(3)    | 0(3)    | -2(3)   |
| C(13) | 58(4)   | 67(5)   | 47(4)   | 4(3)    | 9(3)    | 5(4)    |
| C(14) | 74(5)   | 84(6)   | 54(5)   | 14(4)   | 19(4)   | 4(4)    |
| C(15) | 69(5)   | 91(6)   | 58(5)   | -10(5)  | 15(4)   | 9(5)    |
| C(16) | 85(6)   | 58(5)   | 82(6)   | -15(5)  | 7(5)    | 5(4)    |
| C(17) | 68(5)   | 54(5)   | 63(5)   | 2(4)    | 4(4)    | -3(4)   |
| C(18) | 46(3)   | 50(4)   | 36(3)   | 2(3)    | 8(3)    | -3(3)   |
| C(19) | 62(5)   | 57(5)   | 52(4)   | 0(3)    | 4(4)    | -4(4)   |
| C(20) | 94(7)   | 57(5)   | 75(6)   | 3(4)    | 14(5)   | 1(5)    |
| C(21) | 120(8)  | 50(5)   | 80(6)   | -8(4)   | 31(6)   | -26(5)  |
| C(22) | 75(6)   | 94(7)   | 68(5)   | -13(5)  | 12(5)   | -35(6)  |
| C(23) | 53(4)   | 72(5)   | 61(4)   | -1(4)   | 5(3)    | -13(4)  |
| N(2)  | 78(4)   | 91(5)   | 59(4)   | -10(4)  | 5(3)    | -5(4)   |
| C(31) | 110(8)  | 110(8)  | 102(8)  | -27(7)  | 8(6)    | -20(7)  |
| C(32) | 131(10) | 109(9)  | 110(8)  | 8(7)    | 12(7)   | -9(8)   |
| C(33) | 166(13) | 103(9)  | 146(11) | -23(8)  | 38(10)  | -31(9)  |
| C(34) | 227(18) | 118(11) | 199(16) | -22(11) | 89(14)  | -46(11) |
| C(35) | 107(8)  | 135(9)  | 93(7)   | -18(7)  | 13(6)   | -10(7)  |
| C(36) | 120(9)  | 112(8)  | 86(7)   | 11(6)   | 12(6)   | -11(7)  |
| C(37) | 149(11) | 115(9)  | 78(7)   | 1(6)    | 4(7)    | -19(8)  |
| C(38) | 214(16) | 115(9)  | 100(8)  | 15(8)   | 12(10)  | -20(10) |
| C(39) | 144(10) | 114(9)  | 95(7)   | -9(7)   | 38(7)   | 20(8)   |
| C(40) | 214(16) | 136(11) | 154(13) | -3(10)  | 67(12)  | 84(11)  |
| C(43) | 121(9)  | 139(10) | 91(7)   | -8(7)   | -6(7)   | -19(8)  |
| C(44) | 95(8)   | 176(13) | 134(11) | -13(9)  | 6(8)    | 13(9)   |

Table 4. Positional ( $\times 10^4$ ) and isotropic thermal parameters ( $\times 10^4$ ) of the refined hydrogen atoms. E.s.d.'s in parentheses

|       | x         | y         | z        | U(eq)  |
|-------|-----------|-----------|----------|--------|
| H(7)  | -3634(61) | -1230(22) | 2192(29) | 29(14) |
| H(8)  | -4936(82) | -957(28)  | 1280(35) | 65(20) |
| H(9)  | -3549(93) | -603(33)  | 294(43)  | 89(27) |
| H(10) | -1107(68) | -559(25)  | 417(31)  | 37(18) |
| H(13) | 2398(65)  | -274(26)  | 152(29)  | 43(16) |
| H(14) | 3439(81)  | -700(31)  | -775(38) | 65(22) |
| H(15) | 3863(88)  | -1639(32) | -832(41) | 75(25) |
| H(16) | 3310(72)  | -2194(29) | 29(33)   | 50(19) |
| H(17) | 2152(72)  | -1856(28) | 1058(34) | 57(19) |
| H(19) | -118(70)  | 285(26)   | 1105(32) | 42(19) |
| H(20) | 510(97)   | 1265(34)  | 1066(45) | 86(29) |
| H(21) | 2546(74)  | 1553(30)  | 1471(33) | 59(20) |
| H(22) | 4218(88)  | 844(32)   | 1905(38) | 66(24) |
| H(23) | 3838(83)  | -109(30)  | 1914(37) | 72(23) |

Table 5. Bond lengths [Å] and angles [deg]

---

|             |           |
|-------------|-----------|
| Re(1)-O(1)  | 1.692(5)  |
| Re(1)-N(1)  | 2.007(5)  |
| Re(1)-O(2)  | 2.076(4)  |
| Re(1)-O(3)  | 2.126(5)  |
| Re(1)-S(1)  | 2.294(2)  |
| Re(1)-P     | 2.435(2)  |
| O(2)-C(6)   | 1.313(7)  |
| O(3)-C(1)   | 1.292(8)  |
| P-C(11)     | 1.778(6)  |
| P-C(12)     | 1.830(6)  |
| P-C(18)     | 1.834(6)  |
| S(1)-C(4)   | 1.839(8)  |
| N(1)-C(3)   | 1.345(10) |
| N(1)-C(2)   | 1.450(11) |
| C(1)-O(4)   | 1.227(9)  |
| C(1)-C(2)   | 1.489(12) |
| C(3)-O(5)   | 1.226(9)  |
| C(3)-C(4)   | 1.525(13) |
| C(4)-C(5)   | 1.37(2)   |
| C(4)-C(5A)  | 1.38(2)   |
| C(5)-C(5A)  | 1.88(4)   |
| C(6)-C(11)  | 1.400(8)  |
| C(6)-C(7)   | 1.411(9)  |
| C(7)-C(8)   | 1.351(10) |
| C(8)-C(9)   | 1.370(11) |
| C(9)-C(10)  | 1.372(10) |
| C(10)-C(11) | 1.394(9)  |
| C(12)-C(17) | 1.381(10) |
| C(12)-C(13) | 1.393(9)  |
| C(13)-C(14) | 1.369(10) |
| C(14)-C(15) | 1.372(12) |
| C(15)-C(16) | 1.346(12) |
| C(16)-C(17) | 1.386(11) |
| C(18)-C(19) | 1.386(9)  |
| C(18)-C(23) | 1.392(9)  |
| C(19)-C(20) | 1.369(11) |
| C(20)-C(21) | 1.363(14) |
| C(21)-C(22) | 1.366(14) |
| C(22)-C(23) | 1.377(12) |
| N(2)-C(31)  | 1.482(11) |
| N(2)-C(43)  | 1.497(12) |
| N(2)-C(35)  | 1.525(12) |
| N(2)-C(39)  | 1.545(12) |
| C(31)-C(32) | 1.473(14) |
| C(32)-C(33) | 1.53(2)   |
| C(33)-C(34) | 1.48(2)   |
| C(35)-C(36) | 1.475(14) |
| C(36)-C(37) | 1.495(13) |
| C(37)-C(38) | 1.44(2)   |
| C(39)-C(40) | 1.38(2)   |
| C(40)-C(41) | 1.54(2)   |
| C(41)-C(42) | 1.01(3)   |
| C(43)-C(44) | 1.46(2)   |
| C(44)-C(45) | 1.59(2)   |
| C(45)-C(46) | 1.28(3)   |

|                   |           |
|-------------------|-----------|
| O(1)-Re(1)-N(1)   | 109.2(2)  |
| O(1)-Re(1)-O(2)   | 161.4(2)  |
| N(1)-Re(1)-O(2)   | 85.6(2)   |
| O(1)-Re(1)-O(3)   | 93.2(2)   |
| N(1)-Re(1)-O(3)   | 78.9(2)   |
| O(2)-Re(1)-O(3)   | 78.3(2)   |
| O(1)-Re(1)-S(1)   | 103.3(2)  |
| N(1)-Re(1)-S(1)   | 84.3(2)   |
| O(2)-Re(1)-S(1)   | 89.0(2)   |
| O(3)-Re(1)-S(1)   | 159.6(2)  |
| O(1)-Re(1)-P      | 89.0(2)   |
| N(1)-Re(1)-P      | 161.7(2)  |
| O(2)-Re(1)-P      | 76.08(12) |
| O(3)-Re(1)-P      | 98.51(13) |
| S(1)-Re(1)-P      | 93.74(7)  |
| C(6)-O(2)-Re(1)   | 126.6(4)  |
| C(1)-O(3)-Re(1)   | 115.6(5)  |
| C(11)-P-C(12)     | 107.3(3)  |
| C(11)-P-C(18)     | 107.7(3)  |
| C(12)-P-C(18)     | 104.2(3)  |
| C(11)-P-Re(1)     | 102.5(2)  |
| C(12)-P-Re(1)     | 117.9(2)  |
| C(18)-P-Re(1)     | 116.7(2)  |
| C(4)-S(1)-Re(1)   | 99.5(3)   |
| C(3)-N(1)-C(2)    | 120.0(7)  |
| C(3)-N(1)-Re(1)   | 123.2(5)  |
| C(2)-N(1)-Re(1)   | 116.3(5)  |
| O(4)-C(1)-O(3)    | 123.2(8)  |
| O(4)-C(1)-C(2)    | 119.7(7)  |
| O(3)-C(1)-C(2)    | 117.1(7)  |
| N(1)-C(2)-C(1)    | 111.9(6)  |
| O(5)-C(3)-N(1)    | 122.4(9)  |
| O(5)-C(3)-C(4)    | 120.2(8)  |
| N(1)-C(3)-C(4)    | 117.4(7)  |
| C(5)-C(4)-C(5A)   | 86(2)     |
| C(5)-C(4)-C(3)    | 110.8(13) |
| C(5A)-C(4)-C(3)   | 118.6(14) |
| C(5)-C(4)-S(1)    | 115.2(12) |
| C(5A)-C(4)-S(1)   | 113.4(12) |
| C(3)-C(4)-S(1)    | 110.9(6)  |
| C(4)-C(5)-C(5A)   | 47.2(12)  |
| C(4)-C(5A)-C(5)   | 46.8(13)  |
| O(2)-C(6)-C(11)   | 120.9(5)  |
| O(2)-C(6)-C(7)    | 121.9(6)  |
| C(11)-C(6)-C(7)   | 117.2(6)  |
| C(8)-C(7)-C(6)    | 121.0(7)  |
| C(7)-C(8)-C(9)    | 121.6(7)  |
| C(8)-C(9)-C(10)   | 119.4(7)  |
| C(9)-C(10)-C(11)  | 120.4(7)  |
| C(10)-C(11)-C(6)  | 120.4(6)  |
| C(10)-C(11)-P     | 127.4(5)  |
| C(6)-C(11)-P      | 112.2(4)  |
| C(17)-C(12)-C(13) | 119.0(7)  |
| C(17)-C(12)-P     | 119.4(5)  |
| C(13)-C(12)-P     | 121.5(5)  |
| C(14)-C(13)-C(12) | 120.2(7)  |
| C(13)-C(14)-C(15) | 120.1(8)  |
| C(16)-C(15)-C(14) | 120.1(8)  |
| C(15)-C(16)-C(17) | 121.2(8)  |
| C(12)-C(17)-C(16) | 119.3(8)  |

|                   |           |
|-------------------|-----------|
| C(19)-C(18)-C(23) | 119.4(7)  |
| C(19)-C(18)-P     | 122.2(5)  |
| C(23)-C(18)-P     | 118.4(5)  |
| C(20)-C(19)-C(18) | 120.4(8)  |
| C(21)-C(20)-C(19) | 120.5(9)  |
| C(20)-C(21)-C(22) | 119.2(8)  |
| C(21)-C(22)-C(23) | 122.1(9)  |
| C(22)-C(23)-C(18) | 118.3(8)  |
| C(31)-N(2)-C(43)  | 114.2(8)  |
| C(31)-N(2)-C(35)  | 110.8(7)  |
| C(43)-N(2)-C(35)  | 107.6(8)  |
| C(31)-N(2)-C(39)  | 106.7(8)  |
| C(43)-N(2)-C(39)  | 106.2(8)  |
| C(35)-N(2)-C(39)  | 111.3(8)  |
| C(32)-C(31)-N(2)  | 117.6(9)  |
| C(31)-C(32)-C(33) | 112.9(10) |
| C(34)-C(33)-C(32) | 111.3(12) |
| C(36)-C(35)-N(2)  | 118.2(9)  |
| C(35)-C(36)-C(37) | 111.3(10) |
| C(38)-C(37)-C(36) | 118.3(12) |
| C(40)-C(39)-N(2)  | 120.2(12) |
| C(39)-C(40)-C(41) | 115(2)    |
| C(42)-C(41)-C(40) | 129(3)    |
| C(44)-C(43)-N(2)  | 116.3(10) |
| C(43)-C(44)-C(45) | 108.5(12) |
| C(46)-C(45)-C(44) | 114(2)    |

---