

Table 1. Crystal data and structure refinement for Ph₄P[8].

| | |
|-----------------------------------|--|
| Identification code | p33gpe2 |
| Empirical formula | C ₃₂ H ₂₉ PREs ₅ |
| Formula weight | 791.02 |
| Temperature | 173(2) K |
| Wavelength | 0.71073 Å |
| Crystal system, space group | Triclinic, P-1 |
| Unit cell dimensions | a = 10.917(2) Å alpha = 85.742(3) deg. b = 11.588(2) Å beta = 89.972(3) deg. c = 13.859(3) Å gamma = 63.378(3) deg. |
| Volume | 1562.1(5) Å ³ |
| Z, Calculated density | 2, 1.682 Mg/m ³ |
| Absorption coefficient | 4.296 mm ⁻¹ |
| F(000) | 782 |
| Crystal size | 0.03 x 0.05 x 0.16 mm |
| Theta range for data collection | 1.47 to 28.26 deg. |
| Limiting indices | -14<=h<=14, -15<=k<=15, -18<=l<=15 |
| Reflections collected / unique | 9932 / 7060 [R(int) = 0.0778] |
| Completeness to theta = 28.26 | 91.0 % |
| Absorption correction | Empirical |
| Max. and min. transmission | 0.9756 and 0.3638 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 7060 / 0 / 362 |
| Goodness-of-fit on F ² | 0.944 |
| Final R indices [I>2sigma(I)] | R ₁ = 0.0735, wR ₂ = 0.1768 |
| R indices (all data) | R ₁ = 0.1196, wR ₂ = 0.1992 |
| Largest diff. peak and hole | 3.690 and -4.841 e.Å ⁻³ |

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{Ph}_4\text{P}[\mathbf{8}]$.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

| | x | y | z | U(eq) |
|-------|------------|----------|-----------|--------|
| Re(1) | -3316(1) | 1044(1) | -2486(1) | 31(1) |
| S(2) | -4876(3) | 633(3) | -3311(2) | 42(1) |
| S(3) | -4514(3) | 960(4) | -1158(2) | 46(1) |
| S(4) | -3010(3) | 2676(3) | -1787(2) | 37(1) |
| S(5) | -3514(3) | 2351(3) | -3916(2) | 40(1) |
| S(1) | -1411(3) | -622(3) | -2437(2) | 39(1) |
| P(1) | -9629(3) | 3976(3) | -2854(2) | 28(1) |
| C(1) | -2816(12) | 2449(12) | -498(9) | 38(3) |
| C(2) | -3327(15) | 3526(14) | 17(11) | 53(3) |
| C(3) | -3130(16) | 3431(16) | 1032(11) | 58(4) |
| C(4) | -2389(16) | 2242(19) | 1500(10) | 63(5) |
| C(5) | -1854(14) | 1142(15) | 981(9) | 48(3) |
| C(6) | -2061(13) | 1247(13) | 2(10) | 45(3) |
| C(7) | -5180(80) | -290(90) | -1360(40) | 42(19) |
| C(7B) | -5820(50) | 470(50) | -1530(20) | 60(10) |
| C(8) | -5597(19) | -92(17) | -2407(12) | 69(5) |
| C(9) | -8798(11) | 4946(10) | -3309(8) | 28(2) |
| C(10) | -9600(12) | 6244(10) | -3623(8) | 32(2) |
| C(11) | -8965(12) | 7015(11) | -3931(8) | 33(2) |
| C(12) | -7566(12) | 6473(11) | -3917(8) | 34(3) |
| C(13) | -6735(11) | 5174(11) | -3591(9) | 37(3) |
| C(14) | -7369(11) | 4415(10) | -3311(8) | 30(2) |
| C(15) | -11450(11) | 4920(10) | -2977(8) | 28(2) |
| C(16) | -12199(12) | 5507(11) | -2184(9) | 38(3) |
| C(17) | -13626(13) | 6314(12) | -2295(10) | 45(3) |
| C(18) | -14252(13) | 6549(13) | -3212(10) | 48(3) |
| C(19) | -13493(12) | 5967(12) | -3988(10) | 42(3) |
| C(20) | -12119(12) | 5147(12) | -3881(9) | 37(3) |
| C(21) | -9156(11) | 3490(10) | -1593(8) | 28(2) |
| C(22) | -9744(13) | 2810(12) | -1046(9) | 39(3) |
| C(23) | -9360(13) | 2412(12) | -104(9) | 43(3) |
| C(24) | -8338(14) | 2654(13) | 325(9) | 46(3) |
| C(25) | -7753(16) | 3293(17) | -187(10) | 61(4) |
| C(26) | -8182(14) | 3748(13) | -1155(9) | 46(3) |
| C(27) | -9137(11) | 2596(9) | -3547(8) | 27(2) |
| C(28) | -8158(11) | 2303(11) | -4253(8) | 32(2) |
| C(29) | -7792(12) | 1205(11) | -4750(9) | 37(3) |
| C(30) | -8401(13) | 386(11) | -4553(9) | 42(3) |
| C(31) | -9388(12) | 688(11) | -3855(9) | 38(3) |
| C(32) | -9763(12) | 1781(11) | -3341(8) | 35(3) |

Table 3. Bond lengths [Å] and angles [deg] for Ph₄P[8].

| | |
|-----------------|------------|
| Re(1)-S(1) | 2.107(3) |
| Re(1)-S(3) | 2.279(3) |
| Re(1)-S(2) | 2.290(3) |
| Re(1)-S(4) | 2.338(3) |
| Re(1)-S(5) | 2.352(3) |
| S(2)-C(8) | 1.824(15) |
| S(3)-C(7B) | 1.85(3) |
| S(3)-C(7) | 1.93(5) |
| S(4)-C(1) | 1.785(12) |
| P(1)-C(15) | 1.789(11) |
| P(1)-C(27) | 1.796(10) |
| P(1)-C(21) | 1.797(11) |
| P(1)-C(9) | 1.815(11) |
| C(1)-C(2) | 1.374(18) |
| C(1)-C(6) | 1.389(18) |
| C(2)-C(3) | 1.41(2) |
| C(3)-C(4) | 1.36(2) |
| C(4)-C(5) | 1.40(2) |
| C(5)-C(6) | 1.364(17) |
| C(7)-C(8) | 1.49(6) |
| C(7B)-C(8) | 1.39(3) |
| C(9)-C(10) | 1.393(14) |
| C(9)-C(14) | 1.397(15) |
| C(10)-C(11) | 1.400(16) |
| C(11)-C(12) | 1.366(16) |
| C(12)-C(13) | 1.402(16) |
| C(13)-C(14) | 1.378(15) |
| C(15)-C(20) | 1.396(16) |
| C(15)-C(16) | 1.397(16) |
| C(16)-C(17) | 1.414(16) |
| C(17)-C(18) | 1.393(18) |
| C(18)-C(19) | 1.380(18) |
| C(19)-C(20) | 1.370(16) |
| C(21)-C(26) | 1.379(16) |
| C(21)-C(22) | 1.404(15) |
| C(22)-C(23) | 1.354(17) |
| C(23)-C(24) | 1.408(18) |
| C(24)-C(25) | 1.343(18) |
| C(25)-C(26) | 1.404(18) |
| C(27)-C(28) | 1.392(15) |
| C(27)-C(32) | 1.407(15) |
| C(28)-C(29) | 1.388(16) |
| C(29)-C(30) | 1.394(18) |
| C(30)-C(31) | 1.390(18) |
| C(31)-C(32) | 1.395(16) |
| S(1)-Re(1)-S(3) | 109.85(13) |
| S(1)-Re(1)-S(2) | 108.25(12) |
| S(3)-Re(1)-S(2) | 84.89(12) |
| S(1)-Re(1)-S(4) | 108.29(12) |
| S(3)-Re(1)-S(4) | 86.88(11) |
| S(2)-Re(1)-S(4) | 143.20(11) |
| S(1)-Re(1)-S(5) | 109.06(11) |

| | |
|-------------------|------------|
| S(3)-Re(1)-S(5) | 141.07(13) |
| S(2)-Re(1)-S(5) | 82.03(12) |
| S(4)-Re(1)-S(5) | 82.19(11) |
| C(8)-S(2)-Re(1) | 105.9(5) |
| C(7B)-S(3)-C(7) | 26.3(17) |
| C(7B)-S(3)-Re(1) | 107.8(9) |
| C(7)-S(3)-Re(1) | 106.7(13) |
| C(1)-S(4)-Re(1) | 113.7(4) |
| C(15)-P(1)-C(27) | 108.5(5) |
| C(15)-P(1)-C(21) | 108.9(5) |
| C(27)-P(1)-C(21) | 111.3(5) |
| C(15)-P(1)-C(9) | 109.6(5) |
| C(27)-P(1)-C(9) | 109.5(5) |
| C(21)-P(1)-C(9) | 109.0(5) |
| C(2)-C(1)-C(6) | 118.3(12) |
| C(2)-C(1)-S(4) | 118.4(11) |
| C(6)-C(1)-S(4) | 123.1(10) |
| C(1)-C(2)-C(3) | 121.7(14) |
| C(4)-C(3)-C(2) | 118.6(14) |
| C(3)-C(4)-C(5) | 120.2(14) |
| C(6)-C(5)-C(4) | 120.7(14) |
| C(5)-C(6)-C(1) | 120.6(13) |
| C(8)-C(7)-S(3) | 106(4) |
| C(8)-C(7B)-S(3) | 115(2) |
| C(7B)-C(8)-C(7) | 35(2) |
| C(7B)-C(8)-S(2) | 113.5(16) |
| C(7)-C(8)-S(2) | 122(2) |
| C(10)-C(9)-C(14) | 120.1(10) |
| C(10)-C(9)-P(1) | 119.2(8) |
| C(14)-C(9)-P(1) | 120.6(8) |
| C(9)-C(10)-C(11) | 119.6(10) |
| C(12)-C(11)-C(10) | 119.0(10) |
| C(11)-C(12)-C(13) | 122.6(11) |
| C(14)-C(13)-C(12) | 118.0(10) |
| C(13)-C(14)-C(9) | 120.7(10) |
| C(20)-C(15)-C(16) | 119.6(10) |
| C(20)-C(15)-P(1) | 120.2(8) |
| C(16)-C(15)-P(1) | 120.1(9) |
| C(15)-C(16)-C(17) | 120.1(11) |
| C(18)-C(17)-C(16) | 118.8(12) |
| C(19)-C(18)-C(17) | 120.2(12) |
| C(20)-C(19)-C(18) | 121.4(12) |
| C(19)-C(20)-C(15) | 119.9(11) |
| C(26)-C(21)-C(22) | 119.0(11) |
| C(26)-C(21)-P(1) | 120.5(9) |
| C(22)-C(21)-P(1) | 120.5(9) |
| C(23)-C(22)-C(21) | 120.6(11) |
| C(22)-C(23)-C(24) | 119.7(12) |
| C(25)-C(24)-C(23) | 120.7(12) |
| C(24)-C(25)-C(26) | 119.8(12) |
| C(21)-C(26)-C(25) | 120.2(12) |
| C(28)-C(27)-C(32) | 119.8(10) |
| C(28)-C(27)-P(1) | 122.7(8) |
| C(32)-C(27)-P(1) | 117.5(8) |
| C(29)-C(28)-C(27) | 120.3(11) |
| C(28)-C(29)-C(30) | 120.6(11) |
| C(31)-C(30)-C(29) | 119.1(10) |

| | |
|-------------------|-----------|
| C(30)-C(31)-C(32) | 121.3(11) |
| C(31)-C(32)-C(27) | 119.0(11) |

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{Ph}_4\text{P}[\mathbf{8}]$.
 The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

| | U11 | U22 | U33 | U23 | U13 | U12 |
|--------------|--------|---------|--------|---------|--------|---------|
| Re(1) | 25(1) | 25(1) | 37(1) | -1(1) | 1(1) | -7(1) |
| S(2) | 37(2) | 38(2) | 48(2) | -8(1) | -2(1) | -15(1) |
| S(3) | 45(2) | 63(2) | 45(2) | -14(2) | 14(1) | -36(2) |
| S(4) | 37(2) | 28(1) | 44(2) | -4(1) | 6(1) | -15(1) |
| S(5) | 36(2) | 35(2) | 38(2) | 6(1) | 1(1) | -9(1) |
| S(1) | 32(2) | 30(2) | 44(2) | 4(1) | 5(1) | -5(1) |
| P(1) | 29(1) | 21(1) | 32(2) | 0(1) | 0(1) | -10(1) |
| C(1) | 38(7) | 42(7) | 36(6) | -9(5) | 3(5) | -19(6) |
| <u>C(2)</u> | 50(8) | 39(7) | 65(10) | -11(7) | 7(7) | -15(7) |
| <u>C(3)</u> | 58(9) | 59(10) | 56(9) | -23(8) | 10(7) | -21(8) |
| <u>C(4)</u> | 65(10) | 109(15) | 37(8) | -16(9) | 10(7) | -56(11) |
| <u>C(5)</u> | 50(8) | 67(10) | 33(7) | 0(6) | -6(6) | -31(7) |
| C(6) | 39(7) | 43(7) | 52(8) | -8(6) | -3(6) | -18(6) |
| <u>C(7)</u> | 40(30) | 70(40) | 40(30) | -40(30) | 10(20) | -40(30) |
| <u>C(7B)</u> | 60(20) | 60(20) | 75(18) | -27(17) | 26(15) | -41(19) |
| <u>C(8)</u> | 93(13) | 71(11) | 68(11) | 19(9) | -17(9) | -62(11) |
| C(9) | 26(5) | 21(5) | 36(6) | -5(4) | -1(4) | -10(4) |
| C(10) | 30(6) | 24(5) | 34(6) | -2(5) | 0(5) | -6(5) |
| C(11) | 44(7) | 22(5) | 36(6) | -3(5) | -3(5) | -18(5) |
| C(12) | 41(7) | 28(6) | 36(6) | 4(5) | 2(5) | -19(5) |
| C(13) | 23(6) | 33(6) | 55(8) | -8(5) | 1(5) | -11(5) |
| C(14) | 36(6) | 23(5) | 32(6) | 2(4) | 0(5) | -16(5) |
| C(15) | 34(6) | 16(5) | 33(6) | 2(4) | 3(4) | -11(4) |
| C(16) | 39(7) | 23(6) | 43(7) | 5(5) | -8(5) | -8(5) |
| C(17) | 36(7) | 40(7) | 53(8) | -23(6) | 7(6) | -9(6) |
| C(18) | 30(7) | 44(8) | 62(9) | -11(6) | -11(6) | -9(6) |
| C(19) | 33(6) | 39(7) | 47(7) | -4(6) | -5(5) | -10(6) |
| C(20) | 37(7) | 35(6) | 36(6) | -8(5) | 5(5) | -12(5) |
| C(21) | 31(6) | 18(5) | 34(6) | 0(4) | 1(4) | -10(4) |
| C(22) | 53(8) | 34(6) | 40(7) | 2(5) | -2(6) | -27(6) |
| C(23) | 38(7) | 38(7) | 50(8) | 7(6) | -3(6) | -17(6) |
| C(24) | 60(8) | 45(8) | 35(7) | 7(6) | -7(6) | -27(7) |
| C(25) | 75(11) | 98(13) | 38(7) | 5(8) | -14(7) | -67(10) |
| C(26) | 55(8) | 53(8) | 42(7) | 1(6) | 3(6) | -35(7) |
| C(27) | 33(6) | 15(5) | 31(6) | 1(4) | -6(4) | -9(4) |
| C(28) | 23(5) | 36(6) | 37(6) | -2(5) | -4(5) | -13(5) |
| C(29) | 35(6) | 30(6) | 38(7) | -4(5) | 0(5) | -7(5) |
| C(30) | 42(7) | 29(6) | 44(7) | -19(5) | -15(6) | -3(5) |
| C(31) | 41(7) | 24(6) | 51(7) | -2(5) | 0(6) | -15(5) |
| C(32) | 39(7) | 29(6) | 36(6) | -4(5) | 6(5) | -12(5) |

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{Ph}_4\text{P}[\mathbf{8}]$.

| | x | y | z | U(eq) |
|--------|--------|-------|-------|-------|
| H(2A) | -3825 | 4357 | -318 | 64 |
| H(3A) | -3509 | 4183 | 1381 | 70 |
| H(4A) | -2233 | 2158 | 2182 | 76 |
| H(5A) | -1340 | 313 | 1314 | 58 |
| H(6A) | -1686 | 490 | -341 | 54 |
| H(7A) | -4452 | -1183 | -1199 | 51 |
| H(7B) | -5974 | -147 | -953 | 51 |
| H(7B1) | -5868 | -151 | -1020 | 71 |
| H(7B2) | -6727 | 1244 | -1580 | 71 |
| H(8A) | -6604 | 444 | -2452 | 83 |
| H(8B) | -5416 | -951 | -2616 | 83 |
| H(10A) | -10571 | 6604 | -3627 | 38 |
| H(11A) | -9498 | 7902 | -4147 | 40 |
| H(12A) | -7139 | 6995 | -4136 | 41 |
| H(13A) | -5764 | 4828 | -3564 | 45 |
| H(14A) | -6830 | 3522 | -3117 | 36 |
| H(16A) | -11748 | 5364 | -1569 | 45 |
| H(17A) | -14148 | 6688 | -1753 | 54 |
| H(18A) | -15204 | 7113 | -3304 | 57 |
| H(19A) | -13932 | 6138 | -4610 | 50 |
| H(20A) | -11622 | 4732 | -4420 | 45 |
| H(22A) | -10417 | 2627 | -1340 | 47 |
| H(23A) | -9777 | 1971 | 267 | 51 |
| H(24A) | -8058 | 2363 | 983 | 55 |
| H(25A) | -7051 | 3437 | 104 | 73 |
| H(26A) | -7801 | 4235 | -1510 | 56 |
| H(28A) | -7739 | 2855 | -4395 | 39 |
| H(29A) | -7121 | 1010 | -5229 | 45 |
| H(30A) | -8145 | -367 | -4892 | 51 |
| H(31A) | -9815 | 139 | -3725 | 46 |
| H(32A) | -10431 | 1971 | -2860 | 43 |

Table 7. Crystal data and structure refinement for Ph₄P[10].

| | |
|-----------------------------------|--|
| Identification code | p72g |
| Empirical formula | (C ₃₄ H ₄₂ PreS ₅ Si ₂)(CH ₃ CN) _{0.5} |
| Formula weight | 904.85 |
| Temperature | 173(2) K |
| Wavelength | 0.71073 Å |
| Crystal system, space group | Monoclinic, P2 ₁ /c |
| Unit cell dimensions | a = 10.8920(6) Å alpha = 90 deg. b = 15.0195(8) Å beta = 99.334(1) deg. c = 24.8296(14) Å gamma = 90 deg. |
| Volume | 4008.2(4) Å ³ |
| Z, Calculated density | 4, 1.499 Mg/m ³ |
| Absorption coefficient | 3.416 mm ⁻¹ |
| F(000) | 1820 |
| Crystal size | 0.03 x 0.05 x 0.15 mm |
| Theta range for data collection | 1.59 to 28.34 deg. |
| Limiting indices | -13<=h<=14, -20<=k<=19, -28<=l<=32 |
| Reflections collected / unique | 24714 / 9487 [R(int) = 0.0506] |
| Completeness to theta = 28.34 | 94.8 % |
| Absorption correction | Empirical |
| Max. and min. transmission | 0.9909 and 0.3858 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 9487 / 3 / 419 |
| Goodness-of-fit on F ² | 1.052 |
| Final R indices [I>2sigma(I)] | R ₁ = 0.0338, wR ₂ = 0.0773 |
| R indices (all data) | R ₁ = 0.0605, wR ₂ = 0.0849 |
| Largest diff. peak and hole | 1.210 and -1.215 e.Å ⁻³ |

Table 8. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{Ph}_4\text{P}[10]$. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

| | x | y | z | U(eq) |
|-------|-----------|----------|---------|--------|
| Re(1) | 866(1) | 4026(1) | 1861(1) | 30(1) |
| S(1) | 1006(1) | 5426(1) | 1881(1) | 47(1) |
| S(2) | -478(1) | 3534(1) | 2431(1) | 42(1) |
| S(3) | 2410(1) | 3429(1) | 2514(1) | 38(1) |
| S(4) | 2028(1) | 3469(1) | 1247(1) | 33(1) |
| S(5) | -759(1) | 3639(1) | 1190(1) | 33(1) |
| Si(1) | 1966(1) | 2941(1) | 56(1) | 43(1) |
| Si(2) | -1493(1) | 3222(1) | -10(1) | 40(1) |
| P(1) | -6143(1) | 101(1) | 2021(1) | 32(1) |
| C(1) | 444(4) | 3134(4) | 3062(2) | 62(2) |
| C(2) | 1722(4) | 3449(4) | 3146(2) | 68(2) |
| C(3) | 1056(3) | 3281(2) | 605(2) | 31(1) |
| C(4) | -184(3) | 3360(2) | 584(2) | 30(1) |
| C(5) | 2051(5) | 1704(3) | 36(3) | 83(2) |
| C(6) | 1339(5) | 3404(4) | -635(2) | 68(2) |
| C(7) | 3587(4) | 3406(4) | 206(2) | 69(2) |
| C(8) | -1357(4) | 2183(3) | -410(2) | 59(1) |
| C(9) | -1617(6) | 4227(4) | -442(2) | 77(2) |
| C(10) | -2990(4) | 3102(4) | 251(2) | 74(2) |
| C(11) | -5025(3) | 895(2) | 1853(2) | 31(1) |
| C(12) | -5404(4) | 1740(3) | 1663(2) | 40(1) |
| C(13) | -4517(4) | 2372(3) | 1592(2) | 43(1) |
| C(14) | -3272(4) | 2176(3) | 1698(2) | 46(1) |
| C(15) | -2888(4) | 1338(3) | 1883(2) | 43(1) |
| C(16) | -3760(3) | 690(3) | 1959(2) | 36(1) |
| C(17) | -5791(3) | -980(2) | 1783(2) | 31(1) |
| C(18) | -5179(4) | -1081(3) | 1339(2) | 41(1) |
| C(19) | -4966(4) | -1925(3) | 1151(2) | 49(1) |
| C(20) | -5359(4) | -2666(3) | 1402(2) | 53(1) |
| C(21) | -5989(4) | -2568(3) | 1839(2) | 53(1) |
| C(22) | -6212(4) | -1726(3) | 2031(2) | 43(1) |
| C(23) | -6032(4) | 61(2) | 2752(2) | 35(1) |
| C(24) | -4867(4) | -64(3) | 3066(2) | 42(1) |
| C(25) | -4745(4) | -94(3) | 3629(2) | 45(1) |
| C(26) | -5780(5) | 5(3) | 3878(2) | 56(1) |
| C(27) | -6935(5) | 119(3) | 3566(2) | 57(1) |
| C(28) | -7082(4) | 137(3) | 3009(2) | 51(1) |
| C(29) | -7682(3) | 403(3) | 1707(2) | 37(1) |
| C(30) | -8309(4) | -99(3) | 1282(2) | 44(1) |
| C(31) | -9477(4) | 135(4) | 1028(2) | 62(1) |
| C(32) | -10045(4) | 888(3) | 1205(3) | 66(2) |
| C(33) | -9449(4) | 1394(3) | 1632(3) | 64(2) |
| C(34) | -8263(4) | 1161(3) | 1884(2) | 51(1) |
| N(1) | -3211(10) | 600(8) | 451(5) | 132(5) |
| C(35) | -4155(9) | 423(5) | 197(4) | 132(7) |
| C(36) | -5353(9) | 199(9) | -125(5) | 90(5) |

Table 9. Bond lengths [Å] and angles [deg] for Ph₄P[10].

| | |
|-----------------|------------|
| Re(1)-S(1) | 2.1084(11) |
| Re(1)-S(4) | 2.2922(10) |
| Re(1)-S(5) | 2.2994(10) |
| Re(1)-S(2) | 2.3157(10) |
| Re(1)-S(3) | 2.3202(10) |
| S(2)-C(1) | 1.821(5) |
| S(3)-C(2) | 1.846(5) |
| S(4)-C(3) | 1.787(4) |
| S(5)-C(4) | 1.771(4) |
| Si(1)-C(5) | 1.861(5) |
| Si(1)-C(6) | 1.874(5) |
| Si(1)-C(7) | 1.879(5) |
| Si(1)-C(3) | 1.883(4) |
| Si(2)-C(9) | 1.844(5) |
| Si(2)-C(10) | 1.858(4) |
| Si(2)-C(8) | 1.869(4) |
| Si(2)-C(4) | 1.889(4) |
| P(1)-C(29) | 1.790(4) |
| P(1)-C(17) | 1.790(4) |
| P(1)-C(23) | 1.799(4) |
| P(1)-C(11) | 1.802(4) |
| C(1)-C(2) | 1.452(6) |
| C(3)-C(4) | 1.348(5) |
| C(11)-C(12) | 1.394(5) |
| C(11)-C(16) | 1.394(5) |
| C(12)-C(13) | 1.386(5) |
| C(13)-C(14) | 1.372(6) |
| C(14)-C(15) | 1.381(6) |
| C(15)-C(16) | 1.393(5) |
| C(17)-C(18) | 1.387(6) |
| C(17)-C(22) | 1.391(5) |
| C(18)-C(19) | 1.384(5) |
| C(19)-C(20) | 1.378(6) |
| C(20)-C(21) | 1.382(6) |
| C(21)-C(22) | 1.388(6) |
| C(23)-C(24) | 1.390(6) |
| C(23)-C(28) | 1.401(5) |
| C(24)-C(25) | 1.382(6) |
| C(25)-C(26) | 1.379(6) |
| C(26)-C(27) | 1.377(7) |
| C(27)-C(28) | 1.368(7) |
| C(29)-C(30) | 1.383(6) |
| C(29)-C(34) | 1.407(5) |
| C(30)-C(31) | 1.371(6) |
| C(31)-C(32) | 1.394(7) |
| C(32)-C(33) | 1.379(8) |
| C(33)-C(34) | 1.386(7) |
| N(1)-C(35) | 1.1462 |
| C(35)-C(36) | 1.4559 |
| S(1)-Re(1)-S(4) | 109.54(4) |
| S(1)-Re(1)-S(5) | 108.31(4) |
| S(4)-Re(1)-S(5) | 82.49(3) |

| | |
|-------------------|------------|
| S(1)-Re(1)-S(2) | 110.72(4) |
| S(4)-Re(1)-S(2) | 139.66(4) |
| S(5)-Re(1)-S(2) | 82.72(4) |
| S(1)-Re(1)-S(3) | 109.09(4) |
| S(4)-Re(1)-S(3) | 84.73(4) |
| S(5)-Re(1)-S(3) | 142.59(4) |
| S(2)-Re(1)-S(3) | 84.70(4) |
| C(1)-S(2)-Re(1) | 108.43(15) |
| C(2)-S(3)-Re(1) | 103.90(17) |
| C(3)-S(4)-Re(1) | 109.82(12) |
| C(4)-S(5)-Re(1) | 109.76(12) |
| C(5)-Si(1)-C(6) | 111.1(3) |
| C(5)-Si(1)-C(7) | 109.1(3) |
| C(6)-Si(1)-C(7) | 103.6(2) |
| C(5)-Si(1)-C(3) | 108.9(2) |
| C(6)-Si(1)-C(3) | 114.0(2) |
| C(7)-Si(1)-C(3) | 110.09(19) |
| C(9)-Si(2)-C(10) | 107.3(3) |
| C(9)-Si(2)-C(8) | 112.3(3) |
| C(10)-Si(2)-C(8) | 104.7(2) |
| C(9)-Si(2)-C(4) | 109.8(2) |
| C(10)-Si(2)-C(4) | 109.44(19) |
| C(8)-Si(2)-C(4) | 113.03(19) |
| C(29)-P(1)-C(17) | 108.74(18) |
| C(29)-P(1)-C(23) | 110.71(19) |
| C(17)-P(1)-C(23) | 108.67(18) |
| C(29)-P(1)-C(11) | 110.57(18) |
| C(17)-P(1)-C(11) | 109.54(17) |
| C(23)-P(1)-C(11) | 108.57(18) |
| C(2)-C(1)-S(2) | 113.5(4) |
| C(1)-C(2)-S(3) | 112.6(4) |
| C(4)-C(3)-S(4) | 117.8(3) |
| C(4)-C(3)-Si(1) | 129.6(3) |
| S(4)-C(3)-Si(1) | 112.53(19) |
| C(3)-C(4)-S(5) | 118.6(3) |
| C(3)-C(4)-Si(2) | 130.2(3) |
| S(5)-C(4)-Si(2) | 111.26(18) |
| C(12)-C(11)-C(16) | 119.6(3) |
| C(12)-C(11)-P(1) | 120.5(3) |
| C(16)-C(11)-P(1) | 119.7(3) |
| C(13)-C(12)-C(11) | 119.5(4) |
| C(14)-C(13)-C(12) | 121.1(4) |
| C(13)-C(14)-C(15) | 119.8(4) |
| C(14)-C(15)-C(16) | 120.3(4) |
| C(15)-C(16)-C(11) | 119.7(4) |
| C(18)-C(17)-C(22) | 120.0(4) |
| C(18)-C(17)-P(1) | 121.2(3) |
| C(22)-C(17)-P(1) | 118.7(3) |
| C(19)-C(18)-C(17) | 119.8(4) |
| C(20)-C(19)-C(18) | 120.4(4) |
| C(19)-C(20)-C(21) | 120.0(4) |
| C(20)-C(21)-C(22) | 120.2(4) |
| C(21)-C(22)-C(17) | 119.6(4) |
| C(24)-C(23)-C(28) | 119.6(4) |
| C(24)-C(23)-P(1) | 118.5(3) |
| C(28)-C(23)-P(1) | 122.0(3) |
| C(25)-C(24)-C(23) | 120.1(4) |

| | |
|-------------------|----------|
| C(26)-C(25)-C(24) | 119.9(5) |
| C(27)-C(26)-C(25) | 120.0(5) |
| C(28)-C(27)-C(26) | 121.2(4) |
| C(27)-C(28)-C(23) | 119.3(4) |
| C(30)-C(29)-C(34) | 119.2(4) |
| C(30)-C(29)-P(1) | 120.4(3) |
| C(34)-C(29)-P(1) | 120.3(3) |
| C(31)-C(30)-C(29) | 121.2(4) |
| C(30)-C(31)-C(32) | 119.2(5) |
| C(33)-C(32)-C(31) | 120.8(5) |
| C(32)-C(33)-C(34) | 119.8(4) |
| C(33)-C(34)-C(29) | 119.7(5) |
| N(1)-C(35)-C(36) | 180.0 |

Symmetry transformations used to generate equivalent atoms:

Table 10. Anisotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{Ph}_4\text{P}[10]$.
 The anisotropic displacement factor exponent takes the form:
 $-\frac{1}{2} \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

| | U11 | U22 | U33 | U23 | U13 | U12 |
|-------|---------|---------|---------|--------|--------|---------|
| Re(1) | 32(1) | 30(1) | 27(1) | -1(1) | 3(1) | 0(1) |
| S(1) | 69(1) | 31(1) | 39(1) | -2(1) | 1(1) | 0(1) |
| S(2) | 36(1) | 57(1) | 32(1) | 2(1) | 8(1) | -1(1) |
| S(3) | 35(1) | 41(1) | 34(1) | 5(1) | -3(1) | -3(1) |
| S(4) | 29(1) | 39(1) | 32(1) | -1(1) | 4(1) | 1(1) |
| S(5) | 29(1) | 40(1) | 29(1) | -4(1) | 5(1) | 3(1) |
| Si(1) | 39(1) | 55(1) | 39(1) | -11(1) | 15(1) | 1(1) |
| Si(2) | 35(1) | 51(1) | 32(1) | -10(1) | 0(1) | 6(1) |
| P(1) | 33(1) | 23(1) | 40(1) | -3(1) | 10(1) | -1(1) |
| C(1) | 58(3) | 92(4) | 38(3) | 19(3) | 11(2) | 12(3) |
| C(2) | 60(3) | 107(5) | 36(3) | 20(3) | 3(2) | -13(3) |
| C(3) | 34(2) | 32(2) | 27(2) | -1(2) | 4(2) | 1(2) |
| C(4) | 33(2) | 30(2) | 26(2) | -4(2) | 5(2) | 3(2) |
| C(5) | 102(4) | 62(4) | 97(5) | -13(3) | 50(4) | 22(3) |
| C(6) | 67(3) | 105(5) | 34(3) | -7(3) | 19(2) | -16(3) |
| C(7) | 41(3) | 121(5) | 49(3) | -27(3) | 19(2) | -4(3) |
| C(8) | 45(2) | 64(3) | 66(3) | -30(3) | 7(2) | -3(2) |
| C(9) | 92(4) | 72(4) | 58(4) | 10(3) | -15(3) | 14(3) |
| C(10) | 35(2) | 132(5) | 53(3) | -38(3) | -1(2) | 1(3) |
| C(11) | 35(2) | 24(2) | 36(2) | -2(2) | 8(2) | 0(2) |
| C(12) | 40(2) | 34(2) | 46(3) | 3(2) | 9(2) | 1(2) |
| C(13) | 56(3) | 28(2) | 46(3) | 5(2) | 9(2) | -8(2) |
| C(14) | 49(2) | 48(3) | 40(3) | 3(2) | 9(2) | -19(2) |
| C(15) | 33(2) | 57(3) | 38(3) | 6(2) | 7(2) | -4(2) |
| C(16) | 36(2) | 36(2) | 37(2) | 7(2) | 8(2) | 2(2) |
| C(17) | 28(2) | 26(2) | 37(2) | -3(2) | 1(2) | 1(2) |
| C(18) | 44(2) | 34(2) | 44(3) | -6(2) | 11(2) | -4(2) |
| C(19) | 50(2) | 46(3) | 52(3) | -16(2) | 10(2) | 5(2) |
| C(20) | 63(3) | 34(2) | 58(3) | -13(2) | 1(2) | 13(2) |
| C(21) | 70(3) | 25(2) | 63(3) | 5(2) | 2(3) | 4(2) |
| C(22) | 56(3) | 32(2) | 41(3) | 0(2) | 11(2) | -2(2) |
| C(23) | 45(2) | 24(2) | 40(2) | -5(2) | 14(2) | -6(2) |
| C(24) | 53(2) | 33(2) | 43(3) | -2(2) | 17(2) | -2(2) |
| C(25) | 68(3) | 31(2) | 38(3) | -2(2) | 11(2) | -1(2) |
| C(26) | 92(4) | 35(2) | 44(3) | -5(2) | 27(3) | -18(3) |
| C(27) | 66(3) | 56(3) | 60(3) | -14(2) | 39(3) | -22(2) |
| C(28) | 47(2) | 52(3) | 59(3) | -15(2) | 22(2) | -19(2) |
| C(29) | 31(2) | 27(2) | 56(3) | 3(2) | 13(2) | 0(2) |
| C(30) | 37(2) | 39(2) | 56(3) | 1(2) | 9(2) | 4(2) |
| C(31) | 45(3) | 69(4) | 68(4) | 2(3) | 0(2) | 0(2) |
| C(32) | 38(2) | 60(3) | 100(5) | 16(3) | 9(3) | 12(2) |
| C(33) | 43(3) | 33(3) | 122(5) | 5(3) | 32(3) | 8(2) |
| C(34) | 39(2) | 33(2) | 85(4) | -9(2) | 20(2) | -5(2) |
| N(1) | 165(11) | 122(10) | 105(12) | -13(9) | 8(8) | -61(10) |
| C(35) | 174(14) | 118(13) | 92(12) | 61(11) | -19(9) | -70(12) |
| C(36) | 95(9) | 119(13) | 63(10) | -26(9) | 29(6) | 16(9) |

Table 11. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $\text{Ph}_4\text{P}[\mathbf{10}]$.

| | x | y | z | U(eq) |
|--------|--------|-------|------|-------|
| H(1A) | 45 | 3329 | 3373 | 74 |
| H(1B) | 446 | 2475 | 3058 | 74 |
| H(2A) | 2229 | 3070 | 3425 | 82 |
| H(2B) | 1749 | 4065 | 3289 | 82 |
| H(5A) | 2530 | 1486 | 379 | 100 |
| H(5B) | 2460 | 1519 | -269 | 100 |
| H(5C) | 1209 | 1456 | -11 | 100 |
| H(6A) | 503 | 3167 | -759 | 81 |
| H(6B) | 1887 | 3233 | -895 | 81 |
| H(6C) | 1301 | 4055 | -614 | 81 |
| H(7A) | 3548 | 4058 | 218 | 83 |
| H(7B) | 4055 | 3223 | -81 | 83 |
| H(7C) | 4004 | 3181 | 559 | 83 |
| H(8A) | -2106 | 2109 | -684 | 70 |
| H(8B) | -1266 | 1669 | -163 | 70 |
| H(8C) | -626 | 2224 | -593 | 70 |
| H(9A) | -2154 | 4102 | -790 | 92 |
| H(9B) | -787 | 4398 | -510 | 92 |
| H(9C) | -1974 | 4714 | -255 | 92 |
| H(10A) | -3171 | 3653 | 434 | 89 |
| H(10B) | -2928 | 2607 | 511 | 89 |
| H(10C) | -3660 | 2982 | -55 | 89 |
| H(12) | -6264 | 1882 | 1583 | 48 |
| H(13) | -4777 | 2951 | 1469 | 52 |
| H(14) | -2675 | 2615 | 1645 | 55 |
| H(15) | -2025 | 1202 | 1958 | 51 |
| H(16) | -3493 | 112 | 2081 | 44 |
| H(18) | -4906 | -572 | 1164 | 49 |
| H(19) | -4547 | -1995 | 846 | 59 |
| H(20) | -5196 | -3244 | 1276 | 63 |
| H(21) | -6271 | -3080 | 2008 | 64 |
| H(22) | -6649 | -1658 | 2330 | 52 |
| H(24) | -4154 | -128 | 2894 | 50 |
| H(25) | -3949 | -183 | 3843 | 54 |
| H(26) | -5695 | -5 | 4265 | 67 |
| H(27) | -7642 | 186 | 3741 | 69 |
| H(28) | -7886 | 199 | 2798 | 61 |
| H(30) | -7923 | -614 | 1164 | 52 |
| H(31) | -9895 | -213 | 734 | 74 |
| H(32) | -10852 | 1055 | 1029 | 79 |
| H(33) | -9849 | 1900 | 1753 | 77 |
| H(34) | -7843 | 1511 | 2176 | 61 |
| H(36A) | -5230 | -224 | -413 | 109 |
| H(36B) | -5749 | 740 | -292 | 109 |
| H(36C) | -5888 | -71 | 111 | 109 |