Electronic Supplementary Information for Dalton Transactions


***Electronic Supporting Information for***

# Mono-alkylated bisphosphines as dopants for ESI-MS analysis of catalytic reactions

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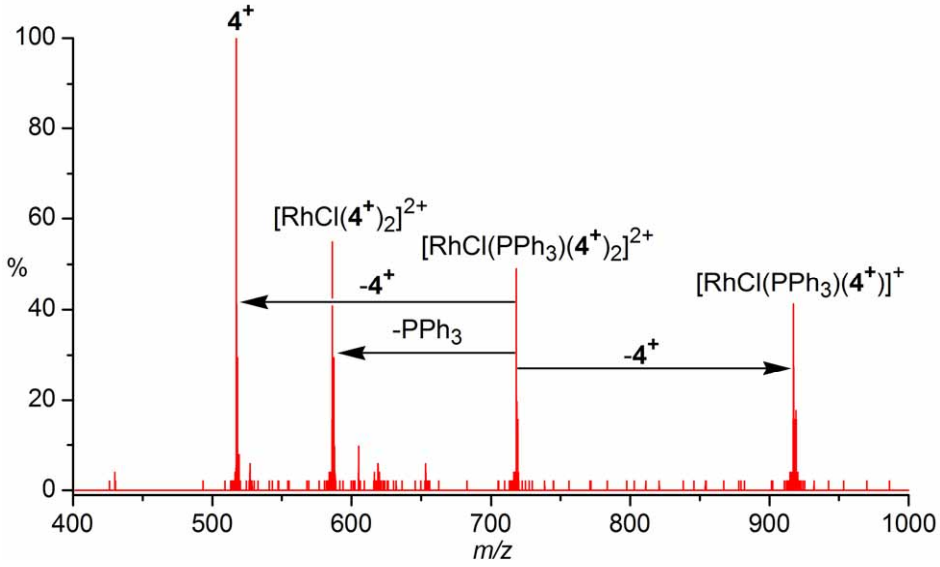


**Figure SI1.** MS/MS of $[RhCl(PPh_3)(\mathbf{4}^+)_2]^+$. Loss of $PPh_3$ (586 m/z) and loss of $\mathbf{4}^+$ (generating two charged fragments, $\mathbf{4}^+$ at 517 *m/z* and $[RhCl(PPh_3)(\mathbf{4}^+)]^+$ at 917 *m/z*) are competitive; while $\mathbf{4}^+$ is a better ligand, it experiences Coloumbic repulsion from the other charged ligand and is therefore eliminated more easily).

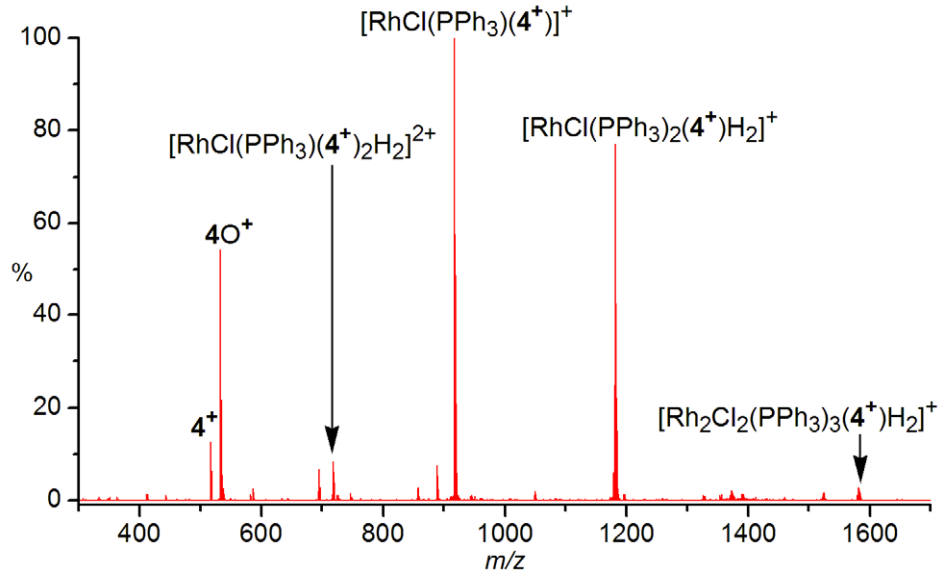


**Figure SI2.** Positive-ion ESI-MS of $RhCl(PPh_3)_3$ + $\mathbf{4}^+$ $BF_4^-$ in $H_2$-saturated chlorobenzene at a cone voltage of 10 V.

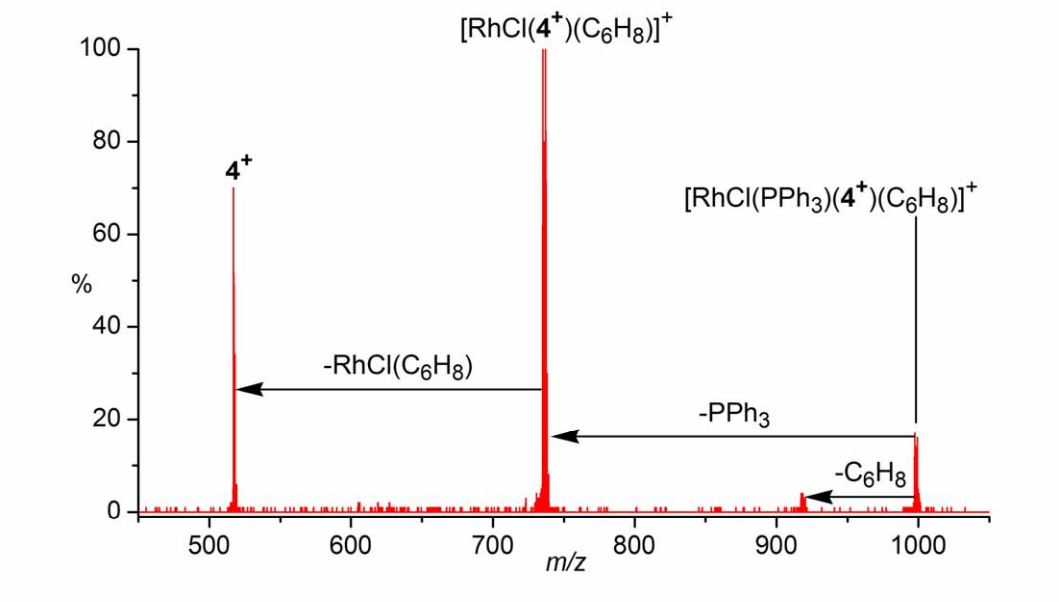


**Figure SI3.** MS/MS of $[RhCl(PPh_3)(\mathbf{4}^+)(C_4H_8)]^+$. Only a small proportion of the product ions involve loss of $C_4H_8$; the predominant fragmentation pathway is loss of $PPh_3$ then $\mathbf{4}^+$.

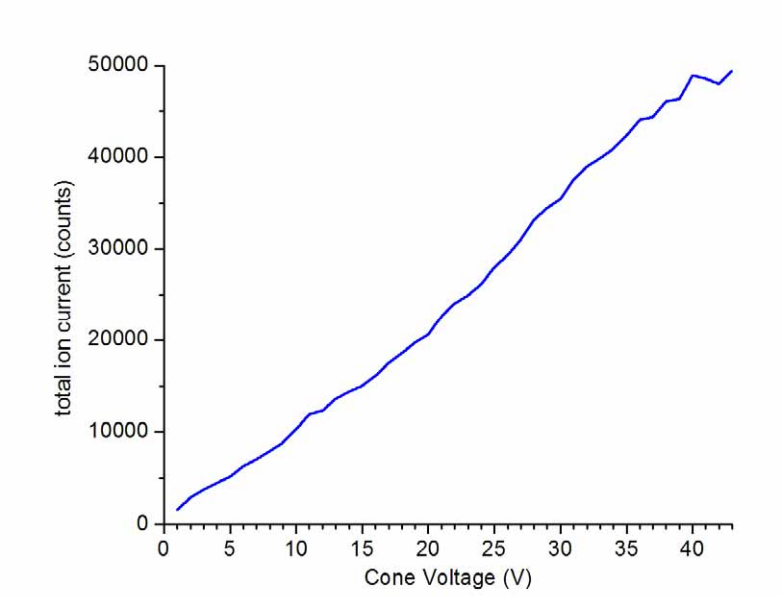


**Figure SI4.** Variation in total ion current (TIC) with cone voltage for the positive-ion ESI-MS of $RhCl(PPh_3)_3$ + $\mathbf{4}^+$ $BF_4^-$ in chlorobenzene. Note the linear response of TIC with voltage. Data were collected for 20 s at each value of cone voltage up to 43 V.

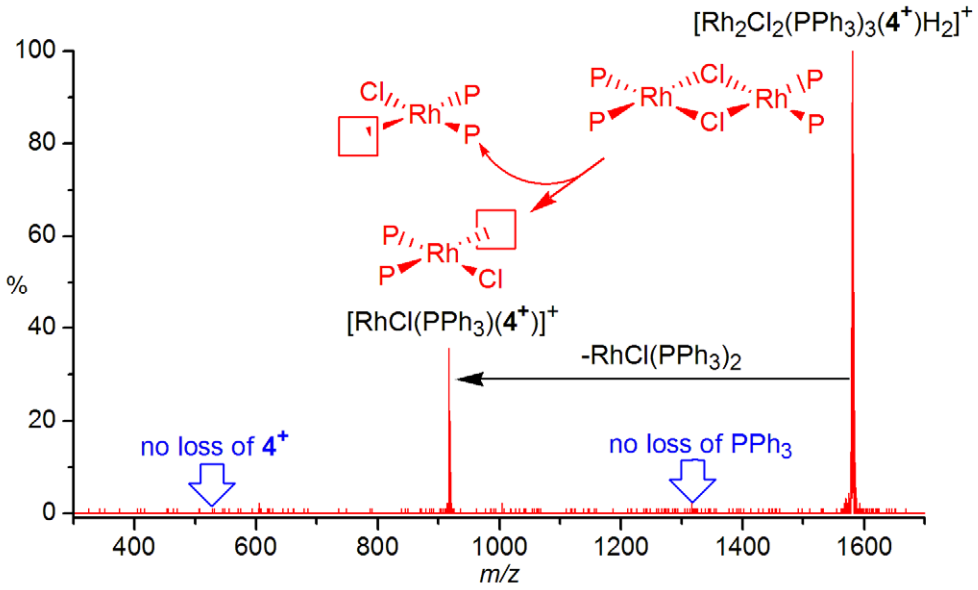


**Figure SI5.** MS/MS of $[Rh_2Cl_2(PPh_3)_3(\mathbf{4}^+)]^+$. Neither loss of $PPh_3$ or loss of $\mathbf{4}^+$ is observed; instead, the dimer cleaves in two symmetrically.

**X-ray Structure Report for $[Ph_2P(CH_2)_4PPh_2CH_2Ph]^+ [PF_6]^-$ ($4^+ PF_6^-$)**

The compound crystallizes as colourless block-like crystals. There are four molecules of the phosphine cation and associated $PF_6$ anion in the unit cell of the primitive, acentric, orthorhombic space group $P2_12_12_1$. This is a chiral space group. The correct enantiomorph of the space group was determined by comparison of intesities of Friedel pairs of reflections. This resulted in an absolute structure parameter of 0.2 (Flack parameter). This vale is indicative of racemic twinning. A racemic twin law was applied to the refined structure.

CRYSTAL SUMMARY

Crystal data for $C_{35}H_{35}F_6P_3$; $M_r$ = 662.54; orthorhombic; space group $P2_12_12_1$; *a* = 10.1738(4) Å; *b* = 15.8803(7) Å; *c* = 19.7059(8) Å; α = 90°; β = 90°; γ = 90°; V = 3183.7(2) $Å^3$; Z = 4; T = 100(2) K; λ(Mo-Kα) = 0.71073 Å; μ(Mo-Kα) = 0.247 $mm^{-1}$; $d_{calc}$ = 1.382g.$cm^{-3}$; 24326 reflections collected; 6483 unique ($R_{int}$ = 0.0470); giving $R_1$ = 0.0426, $wR_2$ = 0.0937 for 5506 data with [I>2σ(I)] and $R_1$ = 0.0546, $wR_2$ = 0.1004 for all 6483 data. Residual electron density ($e^-.Å^{-3}$) max/min: 0.407/-0.303.

An arbitrary sphere of data were collected on a colourless block-like crystal, having approximate dimensions of 0.20 × 0.18 × 0.15 mm, on a Bruker APEX-II diffractometer using a combination of ω- and φ-scans of 0.3°. Data were corrected for absorption and polarization effects and analyzed for space group determination. The structure was solved by direct methods and expanded routinely. The model was refined by full-matrix least-squares analysis of $F^2$ against all reflections. All non-hydrogen atoms were refined with anisotropic thermal displacement parameters. Unless otherwise noted, hydrogen atoms were included in calculated positions. Thermal parameters for the hydrogens were tied to the isotropic thermal parameter of the atom to which they are bonded (1.2 ×).

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

| | |
|---|---|
| Empirical formula | C35 H35 F6 P3 |
| Formula weight | 662.54 |
| Temperature | 100(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Orthorhombic |
| Space group | P212121 |
| Unit cell dimensions | $a$ = 10.1738(4) Å α = 90° |
| | $b$ = 15.8803(7) Å β = 90° |
| | $c$ = 19.7059(8) Å γ = 90° |
| Volume | 3183.7(2) $Å^3$ |
| Z | 4 |
| Density (calculated) | 1.382 $g.cm^{-3}$ |
| Absorption coefficient (μ) | 0.247 $mm^{-1}$ |
| F(000) | 1376 |
| Crystal size | 0.20 × 0.18 × 0.15 $mm^3$ |
| ω range for data collection | 1.65 to 26.46° |
| Index ranges | $-12 \le h \le 12$, $-17 \le k \le 19$, $-24 \le l \le 24$ |
| Reflections collected | 24326 |
| Independent reflections | 6483 [$R_{int}$ = 0.0470] |
| Completeness to θ = 26.46° | 99.4 % |
| Absorption correction | numerical |
| Max. and min. transmission | 1.0000 and 0.8915 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Data / restraints / parameters | 6483 / 0 / 398 |
| Goodness-of-fit on $F^2$ | 1.032 |
| Final R indices [I>2σ(I)] | $R_1$ = 0.0426, $wR_2$ = 0.0937 |
| R indices (all data) | $R_1$ = 0.0546, $wR_2$ = 0.1004 |
| Absolute structure parameter | 0.20(8) |
| Largest diff. peak and hole | 0.407 and -0.303 $e^-.Å^{-3}$ |

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

| | x | y | z | U(eq) |
|---|---|---|---|---|
| P(1) | 0.43911(6) | -0.07444(4) | 0.39599(4) | 0.020(1) |
| P(2) | 0.66646(7) | 0.20173(5) | 0.63754(4) | 0.026(1) |
| C(1) | 0.4454(3) | 0.02584(17) | 0.43879(13) | 0.022(1) |
| C(2) | 0.5707(3) | 0.03763(19) | 0.48077(14) | 0.026(1) |
| C(3) | 0.5564(3) | 0.10887(18) | 0.53208(14) | 0.025(1) |
| C(4) | 0.6895(3) | 0.13399(19) | 0.56284(14) | 0.028(1) |
| C(11) | 0.3929(3) | -0.15446(18) | 0.45708(14) | 0.025(1) |
| C(12) | 0.2707(3) | -0.13429(17) | 0.49698(13) | 0.023(1) |
| C(13) | 0.2820(3) | -0.09898(19) | 0.56205(14) | 0.027(1) |
| C(14) | 0.1726(3) | -0.0832(2) | 0.60072(15) | 0.032(1) |
| C(15) | 0.0482(3) | -0.1022(2) | 0.57601(16) | 0.034(1) |
| C(16) | 0.0354(3) | -0.13650(18) | 0.51111(16) | 0.030(1) |
| C(17) | 0.1460(3) | -0.15234(18) | 0.47206(14) | 0.027(1) |
| C(21) | 0.5979(2) | -0.10213(17) | 0.36331(14) | 0.022(1) |
| C(22) | 0.6514(3) | -0.05444(17) | 0.31082(14) | 0.025(1) |
| C(23) | 0.7763(3) | -0.07162(19) | 0.28731(15) | 0.029(1) |
| C(24) | 0.8481(3) | -0.13674(18) | 0.31585(14) | 0.028(1) |
| C(25) | 0.7961(3) | -0.18406(19) | 0.36758(15) | 0.029(1) |
| C(26) | 0.6703(3) | -0.16720(18) | 0.39145(15) | 0.027(1) |
| C(31) | 0.3230(3) | -0.06791(17) | 0.32780(13) | 0.022(1) |
| C(32) | 0.2263(3) | -0.00573(19) | 0.32792(15) | 0.028(1) |
| C(33) | 0.1358(3) | -0.00202(19) | 0.27488(15) | 0.029(1) |
| C(34) | 0.1414(3) | -0.05962(19) | 0.22335(14) | 0.029(1) |
| C(35) | 0.2363(3) | -0.1208(2) | 0.22326(16) | 0.035(1) |
| C(36) | 0.3289(3) | -0.12475(18) | 0.27529(14) | 0.030(1) |
| C(41) | 0.5902(3) | 0.29445(18) | 0.59840(15) | 0.026(1) |
| C(42) | 0.4751(3) | 0.3245(2) | 0.62749(16) | 0.033(1) |
| C(43) | 0.4120(3) | 0.3950(2) | 0.60161(17) | 0.038(1) |
| C(44) | 0.4617(3) | 0.4359(2) | 0.54664(19) | 0.041(1) |
| C(45) | 0.5777(3) | 0.4062(2) | 0.51604(16) | 0.036(1) |
| C(46) | 0.6406(3) | 0.33610(19) | 0.54203(14) | 0.028(1) |
| C(51) | 0.8318(3) | 0.24425(17) | 0.65345(14) | 0.024(1) |

| | | | | |
|---|---|---|---|---|
| C(52) | 0.9404(3) | 0.23186(18) | 0.61195(15) | 0.028(1) |
| C(53) | 1.0627(3) | 0.26680(18) | 0.62861(15) | 0.029(1) |
| C(54) | 1.0765(3) | 0.31335(18) | 0.68701(15) | 0.029(1) |
| C(55) | 0.9689(3) | 0.32657(19) | 0.72902(14) | 0.028(1) |
| C(56) | 0.8482(3) | 0.29167(18) | 0.71258(14) | 0.029(1) |
| P(3) | 0.82858(7) | 0.12914(5) | 0.14822(4) | 0.025(1) |
| F(1) | 0.92418(17) | 0.04997(11) | 0.14040(9) | 0.041(1) |
| F(2) | 0.73054(18) | 0.20635(12) | 0.15520(10) | 0.047(1) |
| F(3) | 0.8681(2) | 0.13496(15) | 0.22559(10) | 0.066(1) |
| F(4) | 0.7859(2) | 0.12172(15) | 0.07099(9) | 0.060(1) |
| F(5) | 0.71247(18) | 0.06754(13) | 0.16917(11) | 0.056(1) |
| F(6) | 0.94375(19) | 0.18987(13) | 0.12776(15) | 0.078(1) |
| H(1A) | 0.4399 | 0.0714 | 0.4046 | 0.027 |
| H(1B) | 0.3682 | 0.0309 | 0.4691 | 0.027 |
| H(2A) | 0.5909 | -0.0154 | 0.5051 | 0.031 |
| H(2B) | 0.6451 | 0.0501 | 0.4500 | 0.031 |
| H(3A) | 0.4963 | 0.0909 | 0.5689 | 0.030 |
| H(3B) | 0.5168 | 0.1584 | 0.5095 | 0.030 |
| H(4A) | 0.7422 | 0.1643 | 0.5284 | 0.033 |
| H(4B) | 0.7383 | 0.0827 | 0.5762 | 0.033 |
| H(11A) | 0.3792 | -0.2083 | 0.4327 | 0.030 |
| H(11B) | 0.4666 | -0.1627 | 0.4892 | 0.030 |
| H(13A) | 0.3666 | -0.0859 | 0.5795 | 0.033 |
| H(14A) | 0.1820 | -0.0591 | 0.6446 | 0.039 |
| H(15A) | -0.0275 | -0.0921 | 0.6031 | 0.041 |
| H(16A) | -0.0494 | -0.1490 | 0.4936 | 0.036 |
| H(17A) | 0.1366 | -0.1757 | 0.4279 | 0.032 |
| H(22A) | 0.6018 | -0.0100 | 0.2912 | 0.030 |
| H(23A) | 0.8129 | -0.0389 | 0.2517 | 0.035 |
| H(24A) | 0.9340 | -0.1487 | 0.2995 | 0.033 |
| H(25A) | 0.8461 | -0.2284 | 0.3871 | 0.035 |
| H(26A) | 0.6340 | -0.2003 | 0.4270 | 0.033 |
| H(32A) | 0.2221 | 0.0339 | 0.3640 | 0.033 |
| H(33A) | 0.0703 | 0.0406 | 0.2745 | 0.035 |
| H(34A) | 0.0792 | -0.0572 | 0.1875 | 0.034 |
| H(35A) | 0.2391 | -0.1609 | 0.1875 | 0.042 |
| H(36A) | 0.3957 | -0.1665 | 0.2745 | 0.035 |

| | | | | |
|---|---|---|---|---|
| H(42A) | 0.4390 | 0.2964 | 0.6657 | 0.040 |
| H(43A) | 0.3336 | 0.4148 | 0.6224 | 0.046 |
| H(44A) | 0.4183 | 0.4842 | 0.5291 | 0.049 |
| H(45A) | 0.6129 | 0.4343 | 0.4775 | 0.043 |
| H(46A) | 0.7189 | 0.3162 | 0.5212 | 0.034 |
| H(52A) | 0.9318 | 0.1993 | 0.5718 | 0.034 |
| H(53A) | 1.1360 | 0.2583 | 0.5996 | 0.034 |
| H(54A) | 1.1597 | 0.3365 | 0.6986 | 0.035 |
| H(55A) | 0.9780 | 0.3595 | 0.7690 | 0.034 |
| H(56A) | 0.7754 | 0.3001 | 0.7420 | 0.035 |

**Table 3. Anisotropic displacement parameters $(Å)^2$ for agojsm3. The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12} ]$**

| | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| P(1) | 0.0185(3) | 0.0203(4) | 0.0213(3) | 0.0000(3) | -0.0007(3) | 0.0002(3) |
| P(2) | 0.0271(4) | 0.0289(4) | 0.0230(4) | -0.0012(3) | -0.0013(3) | -0.0043(3) |
| C(1) | 0.0225(13) | 0.0210(15) | 0.0227(14) | 0.0010(12) | -0.0001(11) | -0.0023(11) |
| C(2) | 0.0259(14) | 0.0235(15) | 0.0275(15) | -0.0021(12) | -0.0040(12) | -0.0023(12) |
| C(3) | 0.0275(14) | 0.0226(16) | 0.0248(14) | -0.0028(12) | -0.0021(12) | -0.0017(12) |
| C(4) | 0.0324(16) | 0.0226(16) | 0.0286(15) | -0.0021(13) | -0.0052(12) | 0.0004(12) |
| C(11) | 0.0266(15) | 0.0217(16) | 0.0260(15) | 0.0022(13) | -0.0007(12) | 0.0006(11) |
| C(12) | 0.0222(13) | 0.0220(16) | 0.0234(15) | 0.0058(13) | 0.0001(11) | -0.0017(11) |
| C(13) | 0.0189(13) | 0.0343(18) | 0.0292(16) | 0.0025(13) | -0.0037(11) | -0.0045(12) |
| C(14) | 0.0320(15) | 0.0414(19) | 0.0235(15) | 0.0009(14) | 0.0050(13) | -0.0022(14) |
| C(15) | 0.0250(15) | 0.042(2) | 0.0358(17) | 0.0067(15) | 0.0078(13) | 0.0004(13) |
| C(16) | 0.0211(14) | 0.0273(17) | 0.0413(17) | 0.0072(15) | -0.0032(12) | -0.0044(12) |
| C(17) | 0.0268(15) | 0.0249(16) | 0.0290(15) | 0.0008(12) | -0.0022(12) | -0.0042(12) |
| C(21) | 0.0184(12) | 0.0243(15) | 0.0234(14) | -0.0052(12) | -0.0008(11) | 0.0003(10) |
| C(22) | 0.0278(14) | 0.0196(15) | 0.0279(14) | 0.0019(12) | -0.0024(12) | 0.0039(12) |
| C(23) | 0.0292(14) | 0.0282(17) | 0.0302(16) | 0.0031(14) | 0.0065(12) | -0.0025(13) |
| C(24) | 0.0224(14) | 0.0285(17) | 0.0319(15) | -0.0049(13) | 0.0013(12) | 0.0008(12) |
| C(25) | 0.0234(14) | 0.0298(17) | 0.0349(17) | 0.0016(14) | -0.0040(12) | 0.0064(11) |
| C(26) | 0.0252(14) | 0.0273(16) | 0.0295(15) | 0.0054(13) | -0.0002(13) | 0.0010(12) |
| C(31) | 0.0219(13) | 0.0222(15) | 0.0206(13) | 0.0011(12) | -0.0016(11) | -0.0026(11) |
| C(32) | 0.0254(14) | 0.0267(17) | 0.0311(16) | -0.0090(13) | -0.0028(12) | 0.0061(12) |
| C(33) | 0.0221(14) | 0.0309(16) | 0.0345(17) | -0.0017(14) | -0.0058(12) | 0.0080(12) |
| C(34) | 0.0216(14) | 0.0376(19) | 0.0268(15) | -0.0001(13) | -0.0031(11) | -0.0008(12) |
| C(35) | 0.0358(17) | 0.0336(19) | 0.0364(17) | -0.0119(15) | -0.0086(14) | 0.0065(14) |
| C(36) | 0.0276(14) | 0.0278(17) | 0.0333(16) | -0.0041(14) | -0.0061(13) | 0.0090(13) |
| C(41) | 0.0288(15) | 0.0211(15) | 0.0290(16) | -0.0048(13) | -0.0056(12) | -0.0053(12) |
| C(42) | 0.0289(15) | 0.039(2) | 0.0324(17) | -0.0053(14) | 0.0003(12) | -0.0054(13) |
| C(43) | 0.0261(15) | 0.040(2) | 0.049(2) | -0.0116(17) | 0.0017(15) | 0.0028(13) |
| C(44) | 0.0306(17) | 0.0260(18) | 0.066(2) | -0.0025(17) | -0.0163(16) | 0.0013(13) |
| C(45) | 0.0376(17) | 0.0295(18) | 0.0404(18) | 0.0034(15) | -0.0087(14) | -0.0090(14) |
| C(46) | 0.0276(15) | 0.0285(17) | 0.0292(15) | -0.0063(13) | -0.0021(12) | -0.0024(13) |
| C(51) | 0.0251(14) | 0.0213(15) | 0.0269(15) | 0.0019(12) | -0.0065(12) | -0.0047(12) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(52) | 0.0279(14) | 0.0313(17) | 0.0259(15) | 0.0000(13) | -0.0038(13) | 0.0025(12) |
| C(53) | 0.0257(14) | 0.0289(17) | 0.0315(16) | 0.0032(13) | -0.0002(12) | 0.0021(12) |
| C(54) | 0.0296(15) | 0.0273(17) | 0.0309(16) | 0.0051(14) | -0.0082(13) | -0.0030(13) |
| C(55) | 0.0358(17) | 0.0287(17) | 0.0199(14) | -0.0028(13) | -0.0051(12) | -0.0009(13) |
| C(56) | 0.0328(16) | 0.0290(17) | 0.0256(15) | 0.0011(13) | 0.0005(12) | -0.0021(13) |
| P(3) | 0.0211(3) | 0.0281(4) | 0.0273(4) | 0.0017(3) | 0.0012(3) | 0.0047(3) |
| F(1) | 0.0370(9) | 0.0386(11) | 0.0470(11) | 0.0071(9) | 0.0047(9) | 0.0155(8) |
| F(2) | 0.0410(10) | 0.0407(12) | 0.0604(13) | 0.0013(10) | 0.0081(10) | 0.0214(9) |
| F(3) | 0.0737(15) | 0.0814(17) | 0.0416(12) | -0.0196(12) | -0.0205(11) | 0.0295(13) |
| F(4) | 0.0697(14) | 0.0835(17) | 0.0267(10) | -0.0036(11) | -0.0049(9) | 0.0382(12) |
| F(5) | 0.0286(10) | 0.0552(13) | 0.0847(16) | 0.0323(12) | -0.0052(10) | -0.0042(9) |
| F(6) | 0.0300(11) | 0.0471(13) | 0.156(3) | 0.0421(15) | 0.0092(13) | -0.0025(9) |

**Table 4. Bond lengths [Å].**

| atom-atom | distance | atom-atom | distance | |
|---|---|---|---|---|
| P(1)-C(31) | 1.792(3) | P(1)-C(21) | 1.794(3) | P(1)- |
| C(1) | 1.803(3) | P(1)-C(11) | 1.813(3) | P(2)- |
| C(41) | 1.834(3) | P(2)-C(4) | 1.838(3) | P(2)- |
| C(51) | 1.840(3) | C(1)-C(2) | 1.531(4) | C(2)- |
| C(3) | 1.524(4) | C(3)-C(4) | 1.536(4) | C(11)- |
| C(12) | 1.506(4) | C(12)-C(17) | 1.390(4) | C(12)- |
| C(13) | 1.404(4) | C(13)-C(14) | 1.372(4) | C(14)- |
| C(15) | 1.389(4) | C(15)-C(16) | 1.396(4) | C(16)- |
| C(17) | 1.387(4) | C(21)-C(26) | 1.384(4) | C(21)- |
| C(22) | 1.393(4) | C(22)-C(23) | 1.380(4) | C(23)- |
| C(24) | 1.386(4) | C(24)-C(25) | 1.373(4) | C(25)- |
| C(26) | 1.390(4) | C(31)-C(36) | 1.374(4) | C(31)- |
| C(32) | 1.394(4) | C(32)-C(33) | 1.394(4) | C(33)- |
| C(34) | 1.368(4) | C(34)-C(35) | 1.370(4) | C(35)- |
| C(36) | 1.393(4) | C(41)-C(42) | 1.389(4) | C(41)- |
| C(46) | 1.391(4) | C(42)-C(43) | 1.387(4) | C(43)- |
| C(44) | 1.361(5) | C(44)-C(45) | 1.408(5) | C(45)- |
| C(46) | 1.382(4) | C(51)-C(52) | 1.389(4) | C(51)- |
| C(56) | 1.397(4) | C(52)-C(53) | 1.401(4) | C(53)- |
| C(54) | 1.375(4) | C(54)-C(55) | 1.389(4) | C(55)- |
| C(56) | 1.386(4) | P(3)-F(6) | 1.570(2) | P(3)- |
| F(3) | 1.579(2) | P(3)-F(2) | 1.5866(18) | P(3)- |
| F(4) | 1.5871(19) | P(3)-F(5) | 1.5883(19) | P(3)- |
| F(1) | 1.5969(18) | C(1)-H(1A) | 0.9900 | C(1)- |
| H(1B) | 0.9900 | C(2)-H(2A) | 0.9900 | C(2)- |
| H(2B) | 0.9900 | C(3)-H(3A) | 0.9900 | C(3)- |
| H(3B) | 0.9900 | C(4)-H(4A) | 0.9900 | C(4)- |
| H(4B) | 0.9900 | C(11)-H(11A) | 0.9900 | C(11)- |
| H(11B) | 0.9900 | C(13)-H(13A) | 0.9500 | C(14)- |
| H(14A) | 0.9500 | C(15)-H(15A) | 0.9500 | C(16)- |
| H(16A) | 0.9500 | C(17)-H(17A) | 0.9500 | C(22)- |
| H(22A) | 0.9500 | C(23)-H(23A) | 0.9500 | C(24)- |
| H(24A) | 0.9500 | C(25)-H(25A) | 0.9500 | C(26)- |
| H(26A) | 0.9500 | C(32)-H(32A) | 0.9500 | C(33)- |
| H(33A) | 0.9500 | C(34)-H(34A) | 0.9500 | C(35)- |
| H(35A) | 0.9500 | C(36)-H(36A) | 0.9500 | C(42)- |
| H(42A) | 0.9500 | C(43)-H(43A) | 0.9500 | C(44)- |
| H(44A) | 0.9500 | C(45)-H(45A) | 0.9500 | C(46)- |
| H(46A) | 0.9500 | C(52)-H(52A) | 0.9500 | C(53)- |
| H(53A) | 0.9500 | C(54)-H(54A) | 0.9500 | C(55)- |
| H(55A) | 0.9500 | C(56)-H(56A) | 0.9500 | |

**Table 5. Bond angles [°].**

| atom-atom-atom | angle | atom-atom-atom | angle | |
|---|---|---|---|---|
| C(31)-P(1)-C(21) | 109.80(12) | C(31)-P(1)-C(1) | 108.86(13) | C(21)- |
| P(1)-C(1) | 110.62(12) | C(31)-P(1)-C(11) | 111.57(13) | C(21)- |
| P(1)-C(11) | 107.47(13) | C(1)-P(1)-C(11) | 108.53(13) | C(41)- |
| P(2)-C(4) | 100.77(13) | C(41)-P(2)-C(51) | 99.43(13) | C(4)- |
| P(2)-C(51) | 103.58(13) | C(2)-C(1)-P(1) | 112.99(19) | C(3)- |
| C(2)-C(1) | 111.7(2) | C(2)-C(3)-C(4) | 111.7(2) | C(3)- |
| C(4)-P(2) | 110.84(19) | C(12)-C(11)-P(1) | 114.3(2) | C(17)- |
| C(12)-C(13) | 118.7(2) | C(17)-C(12)-C(11) | 121.7(2) | C(13)- |
| C(12)-C(11) | 119.6(2) | C(14)-C(13)-C(12) | 120.9(3) | C(13)- |
| C(14)-C(15) | 120.3(3) | C(14)-C(15)-C(16) | 119.4(3) | C(17)- |
| C(16)-C(15) | 120.2(3) | C(16)-C(17)-C(12) | 120.5(3) | C(26)- |
| C(21)-C(22) | 119.7(2) | C(26)-C(21)-P(1) | 121.2(2) | C(22)- |
| C(21)-P(1) | 119.0(2) | C(23)-C(22)-C(21) | 120.1(3) | C(22)- |
| C(23)-C(24) | 119.8(3) | C(25)-C(24)-C(23) | 120.4(3) | C(24)- |
| C(25)-C(26) | 120.1(3) | C(21)-C(26)-C(25) | 119.9(3) | C(36)- |
| C(31)-C(32) | 119.8(2) | C(36)-C(31)-P(1) | 119.9(2) | C(32)- |
| C(31)-P(1) | 120.3(2) | C(33)-C(32)-C(31) | 119.6(3) | C(34)- |
| C(33)-C(32) | 120.1(3) | C(33)-C(34)-C(35) | 120.3(3) | C(34)- |
| C(35)-C(36) | 120.5(3) | C(31)-C(36)-C(35) | 119.7(3) | C(42)- |
| C(41)-C(46) | 118.5(3) | C(42)-C(41)-P(2) | 117.3(2) | C(46)- |
| C(41)-P(2) | 124.2(2) | C(43)-C(42)-C(41) | 121.1(3) | C(44)- |
| C(43)-C(42) | 120.4(3) | C(43)-C(44)-C(45) | 119.5(3) | C(46)- |
| C(45)-C(44) | 120.0(3) | C(45)-C(46)-C(41) | 120.5(3) | C(52)- |
| C(51)-C(56) | 118.2(3) | C(52)-C(51)-P(2) | 125.1(2) | C(56)- |
| C(51)-P(2) | 116.7(2) | C(51)-C(52)-C(53) | 120.8(3) | C(54)- |
| C(53)-C(52) | 120.0(3) | C(53)-C(54)-C(55) | 120.0(3) | C(56)- |
| C(55)-C(54) | 119.9(3) | C(55)-C(56)-C(51) | 121.1(3) | F(6)- |
| P(3)-F(3) | 91.25(15) | F(6)-P(3)-F(2) | 90.96(11) | F(3)- |
| P(3)-F(2) | 91.79(11) | F(6)-P(3)-F(4) | 90.20(14) | F(3)- |
| P(3)-F(4) | 178.55(15) | F(2)-P(3)-F(4) | 88.20(11) | F(6)- |
| P(3)-F(5) | 179.75(14) | F(3)-P(3)-F(5) | 88.54(13) | F(2)- |
| P(3)-F(5) | 89.19(11) | F(4)-P(3)-F(5) | 90.01(13) | F(6)- |
| P(3)-F(1) | 90.24(11) | F(3)-P(3)-F(1) | 89.09(11) | F(2)- |
| P(3)-F(1) | 178.49(11) | F(4)-P(3)-F(1) | 90.89(11) | F(5)- |
| P(3)-F(1) | 89.61(10) | C(2)-C(1)-H(1A) | 109.0 | P(1)- |
| C(1)-H(1A) | 109.0 | C(2)-C(1)-H(1B) | 109.0 | P(1)- |
| C(1)-H(1B) | 109.0 | H(1A)-C(1)-H(1B) | 107.8 | C(3)- |
| C(2)-H(2A) | 109.3 | C(1)-C(2)-H(2A) | 109.3 | C(3)- |
| C(2)-H(2B) | 109.3 | C(1)-C(2)-H(2B) | 109.3 | H(2A)- |
| C(2)-H(2B) | 107.9 | C(2)-C(3)-H(3A) | 109.3 | C(4)- |
| C(3)-H(3A) | 109.3 | C(2)-C(3)-H(3B) | 109.3 | C(4)- |
| C(3)-H(3B) | 109.3 | H(3A)-C(3)-H(3B) | 107.9 | C(3)- |
| C(4)-H(4A) | 109.5 | P(2)-C(4)-H(4A) | 109.5 | C(3)- |

| | | | | |
|---|---|---|---|---|
| C(4)-H(4B) | 109.5 | P(2)-C(4)-H(4B) | 109.5 | H(4A)- |
| C(4)-H(4B) | 108.1 | C(12)-C(11)-H(11A) | 108.7 | P(1)- |
| C(11)-H(11A) | 108.7 | C(12)-C(11)-H(11B) | 108.7 | P(1)- |
| C(11)-H(11B) | 108.7 | H(11A)-C(11)-H(11B) | 107.6 | C(14)- |
| C(13)-H(13A) | 119.6 | C(12)-C(13)-H(13A) | 119.6 | C(13)- |
| C(14)-H(14A) | 119.8 | C(15)-C(14)-H(14A) | 119.8 | C(14)- |
| C(15)-H(15A) | 120.3 | C(16)-C(15)-H(15A) | 120.3 | C(17)- |
| C(16)-H(16A) | 119.9 | C(15)-C(16)-H(16A) | 119.9 | C(16)- |
| C(17)-H(17A) | 119.8 | C(12)-C(17)-H(17A) | 119.8 | C(23)- |
| C(22)-H(22A) | 119.9 | C(21)-C(22)-H(22A) | 119.9 | C(22)- |
| C(23)-H(23A) | 120.1 | C(24)-C(23)-H(23A) | 120.1 | C(25)- |
| C(24)-H(24A) | 119.8 | C(23)-C(24)-H(24A) | 119.8 | C(24)- |
| C(25)-H(25A) | 120.0 | C(26)-C(25)-H(25A) | 120.0 | C(21)- |
| C(26)-H(26A) | 120.1 | C(25)-C(26)-H(26A) | 120.1 | C(33)- |
| C(32)-H(32A) | 120.2 | C(31)-C(32)-H(32A) | 120.2 | C(34)- |
| C(33)-H(33A) | 120.0 | C(32)-C(33)-H(33A) | 120.0 | C(33)- |
| C(34)-H(34A) | 119.8 | C(35)-C(34)-H(34A) | 119.8 | C(34)- |
| C(35)-H(35A) | 119.8 | C(36)-C(35)-H(35A) | 119.8 | C(31)- |
| C(36)-H(36A) | 120.1 | C(35)-C(36)-H(36A) | 120.1 | C(43)- |
| C(42)-H(42A) | 119.5 | C(41)-C(42)-H(42A) | 119.5 | C(44)- |
| C(43)-H(43A) | 119.8 | C(42)-C(43)-H(43A) | 119.8 | C(43)- |
| C(44)-H(44A) | 120.3 | C(45)-C(44)-H(44A) | 120.3 | C(46)- |
| C(45)-H(45A) | 120.0 | C(44)-C(45)-H(45A) | 120.0 | C(45)- |
| C(46)-H(46A) | 119.7 | C(41)-C(46)-H(46A) | 119.7 | C(51)- |
| C(52)-H(52A) | 119.6 | C(53)-C(52)-H(52A) | 119.6 | C(54)- |
| C(53)-H(53A) | 120.0 | C(52)-C(53)-H(53A) | 120.0 | C(53)- |
| C(54)-H(54A) | 120.0 | C(55)-C(54)-H(54A) | 120.0 | C(56)- |
| C(55)-H(55A) | 120.0 | C(54)-C(55)-H(55A) | 120.0 | C(55)- |
| C(56)-H(56A) | 119.5 | C(51)-C(56)-H(56A) | 119.5 | |

**Table 6. Torsion angles [°].**

| atom-atom-atom-atom | angle | atom-atom-atom-atom | angle | |
|---|---|---|---|---|
| C(31)-P(1)-C(1)-C(2) | -160.38(19) | C(21)-P(1)-C(1)-C(2) | -39.7(2) | C(11)- |
| P(1)-C(1)-C(2) | 78.0(2) | P(1)-C(1)-C(2)-C(3) | -163.0(2) | C(1)- |
| C(2)-C(3)-C(4) | -167.2(2) | C(2)-C(3)-C(4)-P(2) | -167.0(2) | C(41)- |
| P(2)-C(4)-C(3) | -64.4(2) | C(51)-P(2)-C(4)-C(3) | -167.0(2) | C(31)- |
| P(1)-C(11)-C(12) | -68.1(2) | C(21)-P(1)-C(11)-C(12) | 171.52(19) | C(1)- |
| P(1)-C(11)-C(12) | 51.9(2) | P(1)-C(11)-C(12)-C(17) | 84.8(3) | P(1)- |
| C(11)-C(12)-C(13) | -97.4(3) | C(17)-C(12)-C(13)-C(14) | 0.6(4) | C(11)- |
| C(12)-C(13)-C(14) | -177.3(3) | C(12)-C(13)-C(14)-C(15) | 0.2(5) | C(13)- |
| C(14)-C(15)-C(16) | -0.9(5) | C(14)-C(15)-C(16)-C(17) | 0.8(5) | C(15)- |
| C(16)-C(17)-C(12) | -0.1(4) | C(13)-C(12)-C(17)-C(16) | -0.6(4) | C(11)- |
| C(12)-C(17)-C(16) | 177.2(3) | C(31)-P(1)-C(21)-C(26) | -130.1(2) | C(1)- |
| P(1)-C(21)-C(26) | 109.7(2) | C(11)-P(1)-C(21)-C(26) | -8.6(3) | C(31)- |
| P(1)-C(21)-C(22) | 53.0(3) | C(1)-P(1)-C(21)-C(22) | -67.2(2) | C(11)- |
| P(1)-C(21)-C(22) | 174.5(2) | C(26)-C(21)-C(22)-C(23) | -0.5(4) | P(1)- |
| C(21)-C(22)-C(23) | 176.5(2) | C(21)-C(22)-C(23)-C(24) | 0.3(4) | C(22)- |
| C(23)-C(24)-C(25) | -0.3(4) | C(23)-C(24)-C(25)-C(26) | 0.4(4) | C(22)- |
| C(21)-C(26)-C(25) | 0.6(4) | P(1)-C(21)-C(26)-C(25) | -176.3(2) | C(24)- |
| C(25)-C(26)-C(21) | -0.5(4) | C(21)-P(1)-C(31)-C(36) | 38.5(3) | C(1)- |
| P(1)-C(31)-C(36) | 159.7(2) | C(11)-P(1)-C(31)-C(36) | -80.6(2) | C(21)- |
| P(1)-C(31)-C(32) | -141.9(2) | C(1)-P(1)-C(31)-C(32) | -20.7(3) | C(11)- |
| P(1)-C(31)-C(32) | 99.1(2) | C(36)-C(31)-C(32)-C(33) | 0.3(4) | P(1)- |
| C(31)-C(32)-C(33) | -179.3(2) | C(31)-C(32)-C(33)-C(34) | 0.6(4) | C(32)- |
| C(33)-C(34)-C(35) | -0.5(4) | C(33)-C(34)-C(35)-C(36) | -0.5(5) | C(32)- |
| C(31)-C(36)-C(35) | -1.3(4) | P(1)-C(31)-C(36)-C(35) | 178.3(2) | C(34)- |
| C(35)-C(36)-C(31) | 1.4(5) | C(4)-P(2)-C(41)-C(42) | 129.4(2) | C(51)- |
| P(2)-C(41)-C(42) | -124.7(2) | C(4)-P(2)-C(41)-C(46) | -50.8(3) | C(51)- |
| P(2)-C(41)-C(46) | 55.1(3) | C(46)-C(41)-C(42)-C(43) | -0.6(4) | P(2)- |
| C(41)-C(42)-C(43) | 179.2(2) | C(41)-C(42)-C(43)-C(44) | 0.3(5) | C(42)- |
| C(43)-C(44)-C(45) | 0.1(5) | C(43)-C(44)-C(45)-C(46) | -0.3(5) | C(44)- |
| C(45)-C(46)-C(41) | 0.0(4) | C(42)-C(41)-C(46)-C(45) | 0.5(4) | P(2)- |
| C(41)-C(46)-C(45) | -179.3(2) | C(41)-P(2)-C(51)-C(52) | -97.7(3) | C(4)- |
| P(2)-C(51)-C(52) | 5.9(3) | C(41)-P(2)-C(51)-C(56) | 83.2(2) | C(4)- |
| P(2)-C(51)-C(56) | -173.3(2) | C(56)-C(51)-C(52)-C(53) | -0.7(4) | P(2)- |
| C(51)-C(52)-C(53) | -179.8(2) | C(51)-C(52)-C(53)-C(54) | 0.6(4) | C(52)- |
| C(53)-C(54)-C(55) | -0.8(4) | C(53)-C(54)-C(55)-C(56) | 1.0(4) | C(54)- |
| C(55)-C(56)-C(51) | -1.1(4) | C(52)-C(51)-C(56)-C(55) | 1.0(4) | P(2)- |
| C(51)-C(56)-C(55) | -179.8(2) | | | |