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Electronic Supplementary Information

Novel Halogen-Free Chelated Orthoborate-Phosphonium Ionic Liquids: Synthesis and Tribophysical Properties

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Material Contained in this Section:

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Synthesis and Characterisation details of hf-BILs

Tributyltetradecylphosphonium bis(mandelato)borate $[P_{4,4,4,14}][BMB]$

The procedure is similar to that used in the synthesis of $[P_{4,4,4,8}[[BMB]$. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), mandelic acid (3.043 g, 20 mmol) and tributyltetradecylphosphonium chloride (4.349 g, 10 mmol). A viscous colorless ionic liquid was obtained in 81 % yield (5.75 g).

MS (ESI) calcd for $[C_{26}H_{56}P]^+$ *m/z* 399.5; found *m/z* 399.2; calcd for $[C_{16}H_{12}O_6B]^-$ *m/z* 311.0; found *m/z* 310.9.

^{1}H NMR (400.17 MHz, $CDCl_3$): 7.653-7.606 (m, 4H, C_6H_5), 7.320-7.274 (m, 4H, C_6H_5), 7.235-7.229 (m, 2H, C_6H_5), 5.337 (d, 1H, $^4J_{HH}$ = 5.3 Hz, C_6H_5-C<u>H</u>), 5.250 (d, 1H, $^4J_{HH}$ = 2.8 Hz, C_6H_5-C<u>H</u>), 1.958-1.886 (m, 8H, P-CH_2), 1.362-1.235 (m, 36H, -CH_2-), 0.893-0.859 (m, 12H, CH_3) ppm.

^{13}C NMR (100.62 MHz, $CDCl_3$): 177.98, 177.86, 140.17, 139.98, 128.01, 127.89, 127.14, 126.36, 126.15, 126.04, 31.82, 30.55, 29.59, 29.53, 29.25, 28.77, 23.71, 23.56, 23.28, 22.58, 22.40, 21.36, 18.71, 18.94, 14.02, 13.23 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.563 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 10.866 ppm.

Trihexyltetradecylphosphonium bis(mandelato)borate $[P_{6,6,6,14}][BMB]$

The procedure is similar to that used in the synthesis of $[P_{4,4,4,8}[[BMB]$. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), mandelic acid (3.043 g, 20 mmol) and trihexyltetradecylphosphonium chloride (5.189 g, 10 mmol). A viscous colorless ionic liquid was obtained in 91 % yield (7.25 g).

MS (ESI) calcd for $[C_{32}H_{68}P]^+$ *m/z* 483.5; found *m/z* 483.3; calcd for $[C_{16}H_{12}O_6B]^-$ *m/z* 311.0; found *m/z* 311.0.

^{1}H NMR (400.17 MHz, $CDCl_3$): 7.649-7.594 (m, 4H, C_6H_5), 7.311-7.259 (m, 4H, C_6H_5), 7.238-7.208 (m, 2H, C_6H_5), 5.315 (d, 1H, $^4J_{HH}$ = 3.2 Hz, C_6H_5-C<u>H</u>), 5.236 (d, 1H, $^4J_{HH}$ = 4.4 Hz, C_6H_5-C<u>H</u>), 1.924-1.858 (m, 8H, P-CH_2), 1.316-1.222 (m, 48H, -CH_2-), 0.898-0.853 (m, 12H, CH_3) ppm.

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^{13}C NMR (100.62 MHz, $CDCl_3$): 177.92, 177.81, 140.23, 140.13, 128.04, 127.27, 127.16, 26.46, 126.37, 126.13, 31.89, 30.89, 30.42, 30.08, 29.63, 29.50, 29.23, 28.83, 22.65, 22.24, 21.41, 18.73, 14.08, 13.88 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.567 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 10.874 ppm.

Tributyloctylphosphonium bis(salicylato)borate [$P_{4,4,4,8}$][BScB]

The procedure is similar to that used in the synthesis of [$P_{4,4,4,8}$[[BMB]. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), salicylic acid (2.762 g, 20 mmol) and tributyloctylphosphonium chloride (3.509 g, 10 mmol). A viscous colorless ionic liquid was obtained in 88 % yield (5.28 g).

MS (ESI) calcd for $[C_{20}H_{44}P]^+$ *m/z* 315.4; found *m/z* 315.3; calcd for $[C_{14}H_8O_6B]^-$ *m/z* 283.0; found *m/z* 283.1.

^{1}H NMR (400.17 MHz, $CDCl_3$): 7.870 (dd, 2H, $^3J_{HH}$ = 6.0 Hz, $^4J_{HH}$ = 1.6 Hz, C_6H_4), 7.376-7.294 (m, 2H, C_6H_4), 6.881-6.822 (m, 4H, C_6H_4), 2.090-2.018 (m, 8H, P-CH_2), 1.421-1.186 (m, 24H, -CH_2-), 0.935-0.839 (m, 12H, CH_3) ppm.

^{13}C NMR (100.62 MHz, $CDCl_3$): 165.24, 159.40, 134.38, 130.66, 118.68, 118.31, 116.57, 115.46, 31.50, 30.51, 30.36, 28.75, 27.45, 24.13, 2.68, 23.30, 22.42, 21.34, 18.73, 18.26, 13.90, 13.13 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.603 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 3.503 ppm.

Tributyltetradecylphosphonium bis(salicylato)borate [$P_{4,4,4,14}$][BScB]

The procedure is similar to that used in the synthesis of [$P_{4,4,4,8}$[[BMB]. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), salicylic acid (2.762 g, 20 mmol) and tributyltetradecylphosphonium chloride (4.349 g, 10 mmol). A viscous colorless ionic liquid was obtained in 94 % yield (6.44 g).

MS (ESI) calcd for $[C_{26}H_{56}P]^+$ *m/z* 399.5; found *m/z* 399.4; calcd for $[C_{14}H_8O_6B]^-$ *m/z* 283.0; found *m/z* 283.0.

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^{1}H NMR (400.17 MHz, $CDCl_3$): 7.865 (dd, 2H, $^3J_{HH}$ = 6.0 Hz, $^4J_{HH}$ = 1.6 Hz, C_6H_4), 7.374-7.330 (m, 2H, C_6H_4), 6.872-6.770 (m, 4H, C_6H_4), 2.080-1.999 (m, 8H, P-CH_2), 1.404-1.184 (m, 36H, -CH_2-), 0.895-0.832 (m, 12H, CH_3) ppm.

^{13}C NMR (100.62 MHz, $CDCl_3$): 165.20, 159.38, 134.35, 130.65, 118.67, 118.29, 116.54, 115.45, 31.76, 30.50, 29.52, 29.45, 29.19, 28.68, 23.80, 23.51, 23.23, 22.52, 21.31, 18.67, 18.46, 13.96, 13.11 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.60 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 3.501 ppm.

Trihexyltetradecylphosphonium bis(salicylato)borate [$P_{6,6,6,14}$][BScB]

The procedure is similar to that used in the synthesis of [$P_{4,4,4,8}$[[BMB]. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), salicylic acid (2.762 g, 20 mmol) and trihexyltetradecylphosphonium chloride (5.189 g, 10 mmol). A viscous colorless ionic liquid was obtained in 95 % yield (7.30 g).

MS (ESI) calcd for $[C_{32}H_{68}P]^+$ *m/z* 483.5; found *m/z* 483.5; calcd for $[C_{14}H_8O_6B]^-$ *m/z* 283.0; found *m/z* 283.0.

^{1}H NMR (400.17 MHz, $CDCl_3$): 7.862 (dd, 2H, $^3J_{HH}$ = 6.0 Hz, $^4J_{HH}$ = 1.6 Hz, C_6H_4), 7.354-7.333 (m, 2H, C_6H_4), 6.875-6.836 (m, 4H, C_6H_4), 2.090-2.017 (m, 8H, P-CH_2), 1.436-1.187 (m, 48H, -CH_2-), 0.897-0.809 (m, 12H, CH_3) ppm.

^{13}C NMR (100.62 MHz, $CDCl_3$): 165.22, 159.41, 134.33, 130.65, 118.63, 118.29, 116.60, 115.46, 31.77, 30.76, 30.34, 30.01, 29.50, 29.20, 29.11, 28.67, 22.24, 22.11, 21.94, 21.30, 13.96, 13.72 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.601 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 3.504 ppm.

Trihexyltetradecylphosphonium bis(oxalato)borate [$P_{6,6,6,14}$][BOB]

The procedure is similar to that used in the synthesis of [$P_{4,4,4,8}$[[BMB]. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), oxalic acid (1.801 g, 20 mmol) and trihexyltetradecylphosphonium chloride (5.189 g, 10 mmol). A viscous colorless ionic liquid was obtained in 85 % yield (5.70 g).

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MS (ESI) calcd for $[C_{32}H_{68}P]^+$ *m/z* 483.5; found *m/z* 483.7; calcd for $[C_4O_8B]^-$ *m/z* 186.9; found *m/z* 187.0.

^{1}H NMR (400.17 MHz, $CDCl_3$): 2.239-2.167 (m, 8H, P-CH_2), 1.469-1.173 (m, 48H, -CH_2-), 0.775-0.730 (m, 12H, CH_3) ppm.

^{13}C NMR (100.62 MHz, $CDCl_3$): 162.73, 31.58, 30.71, 30.32, 29.98, 29.32, 29.19, 28.97, 27.34, 22.34, 22.08, 21.99, 21.40, 13.77, 13.59 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.643 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 7.321 ppm.

Trihexyltetradecylphosphonium bis(malonato)borate $[P_{6,6,6,14}]$[BMLB]

The procedure is similar to that used in the synthesis of $[P_{4,4,4,8}$[[BMB]. The reaction started with lithium carbonate (0.369 g, 5 mmol), boric acid (0.618 g, 10 mmol), malonic acid (2.081 g, 20 mmol) and trihexyltetradecylphosphonium chloride (5.189 g, 10 mmol). A viscous colorless ionic liquid was obtained in 88 % yield (6.15 g).

MS (ESI) calcd for $[C_{32}H_{68}P]^+$ *m/z* 483.5; found *m/z* 483.6; calcd for $[C_{12}H_4O_8B]^-$ *m/z* 214.9; found *m/z* 215.0.

^{1}H NMR (400.17 MHz, $CDCl_3$): 3.398 (s, 4H, -CO-C$\underline{H}_2$-CO-), 2.258-2.017 (m, 8H, P-CH_2), 1.593-1.261 (m, 48H, -CH_2-), 0.916-0.861 (m, 12H, CH_3) ppm.

^{13}C NMR (100.62 MHz, $CDCl_3$): 166.56, 39.25, 31.90, 30.93, 30.63, 30.28, 29.63, 29.34, 28.94, 28.33, 22.67, 22.28, 22.10, 14.11, 13.89 ppm.

^{31}P NMR (161.99 MHz, $CDCl_3$): 33.694 ppm.

^{11}B NMR (129.39 MHz, $CDCl_3$): 3.436 ppm.

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Table SI-1 Water content of hf-BILs after drying at 60 ºC in a vacuum oven during 48 h using a 831 Karl Fischer coulometer (Metrohm). Duplicate measurements were carried out and the mean values are tabulated together with standard deviations (SD).

hf-BIL	Water content (%) before drying ± SD	Water content (%) after drying at 60 ºC during 48 h ± SD
$[P_{4,4,4,8}]$[BMB]	0.468 ± 0.016	0.250 ± 0.055
$[P_{4,4,4,14}]$[BMB]	0.419 ± 0.062	0.327 ±0.007
$[P_{6,6,6,14}]$[BMB]	0.215 ± 0.027	0.038 ± 0.003
$[P_{4,4,4,8}]$[BScB]	0.286 ± 0.002	0.031 ± 0.003
$[P_{4,4,4,14}]$[BScB]	1.395 ± 0.051	0.034 ± 0.009
$[P_{6,6,6,14}]$[BScB]	0.183 ± 0.016	0.033 ± 0.008
$[P_{6,6,6,14}]$[BOB]	2.323 ± 0.007	2.026 ± 0.006
$[P_{6,6,6,14}]$[BMLB]	0.391 ± 0.051	0.134 ± 0.014

Figure SI-1. ESI-MS of $[P_{4,4,4,8}][BMB]$.

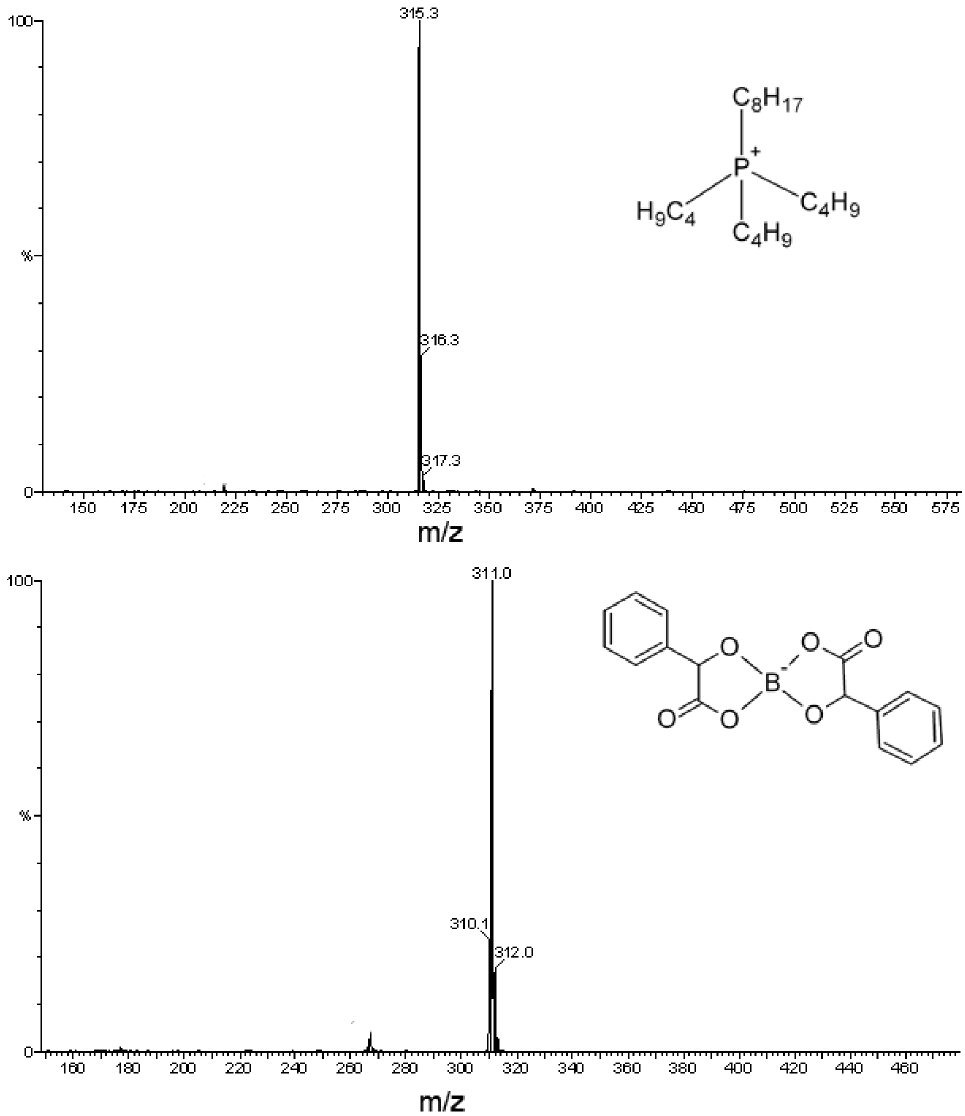

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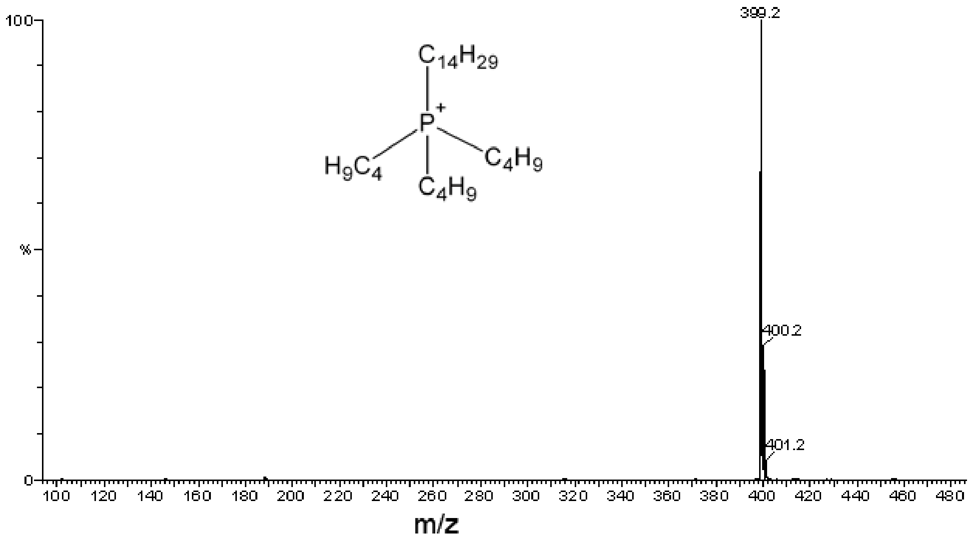
Figure SI-2. ESI-MS of $[P_{4,4,4,14}]$[BMB].



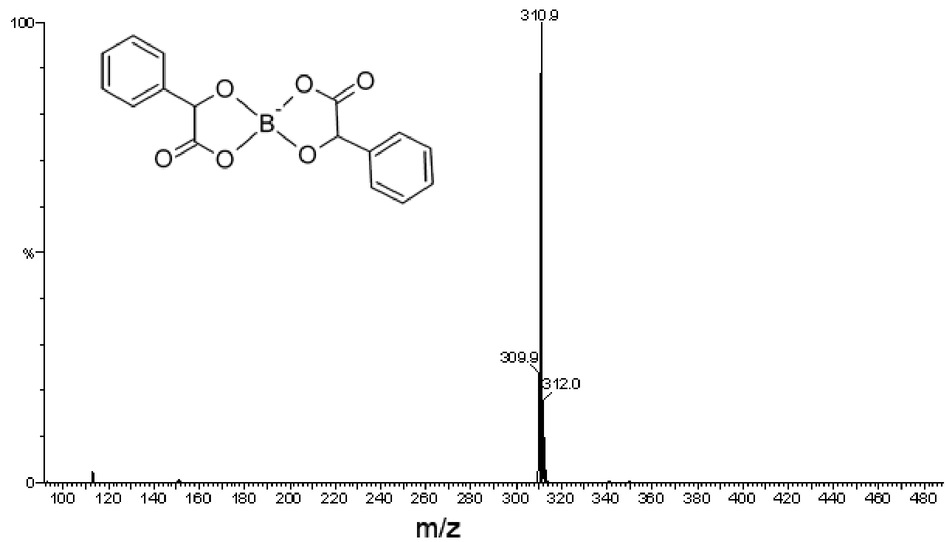


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Figure SI-3. ESI-MS of $[P_{6,6,6,14}]$[BMB].

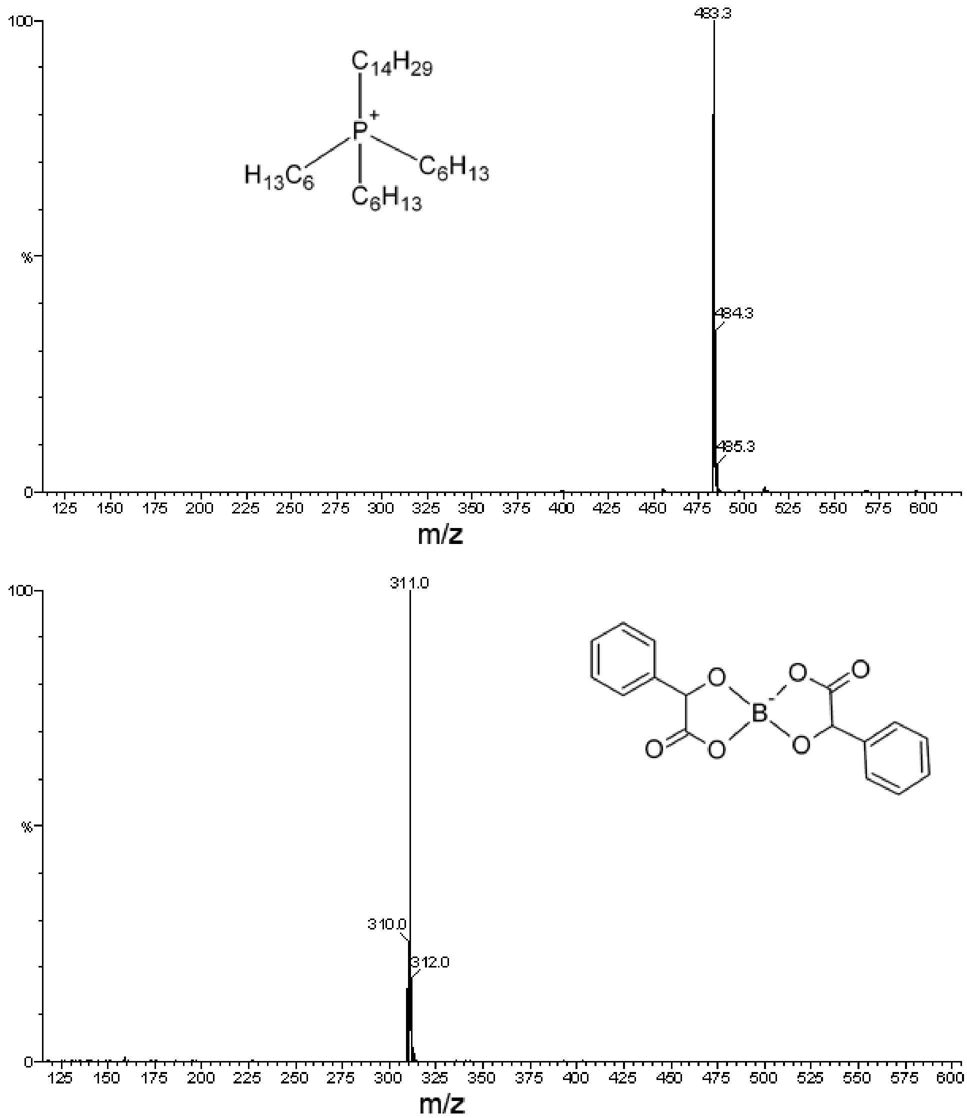

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Figure SI-4. ESI-MS of $[P_{4,4,4,8}][BScB]$.

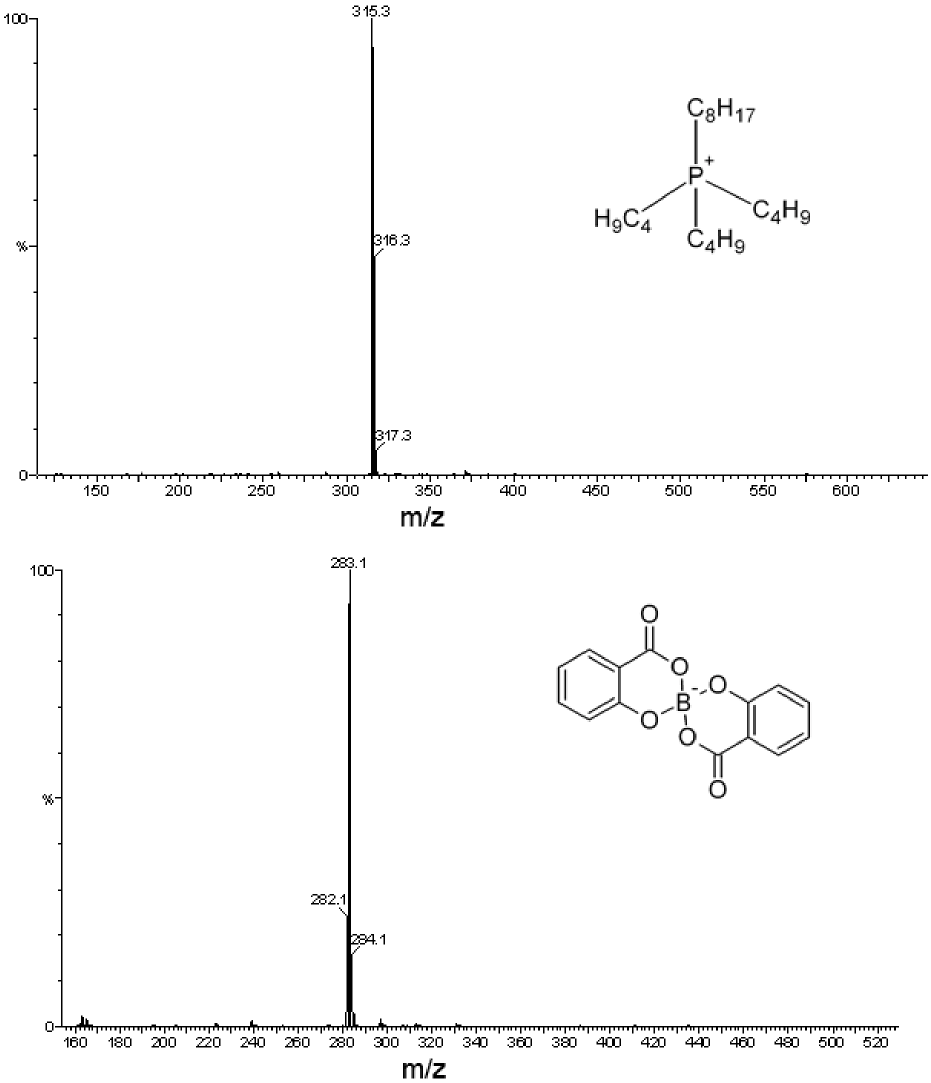

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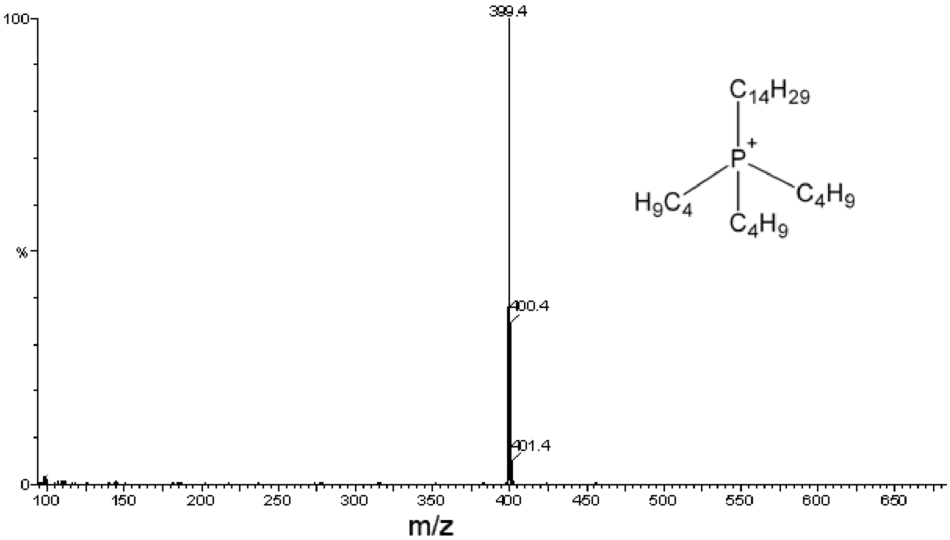
Figure SI-5. ESI-MS of $[P_{4,4,4,14}][BScB]$.



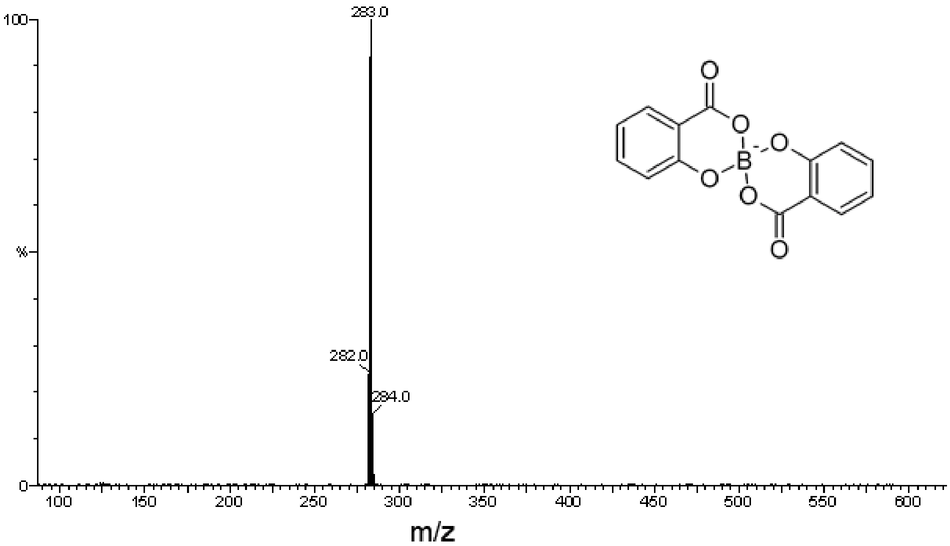


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Figure SI-6. ESI-MS of [$P_{6,6,6,14}$][BScB].

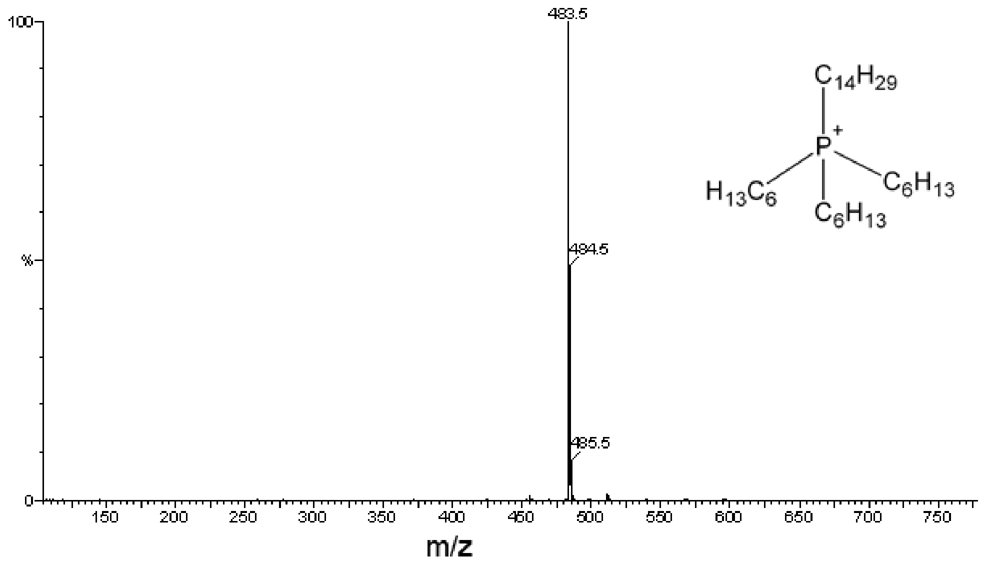

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Figure SI-7. ESI-MS of [$P_{6,6,6,14}$][BScB] after storage in water for 10 days.

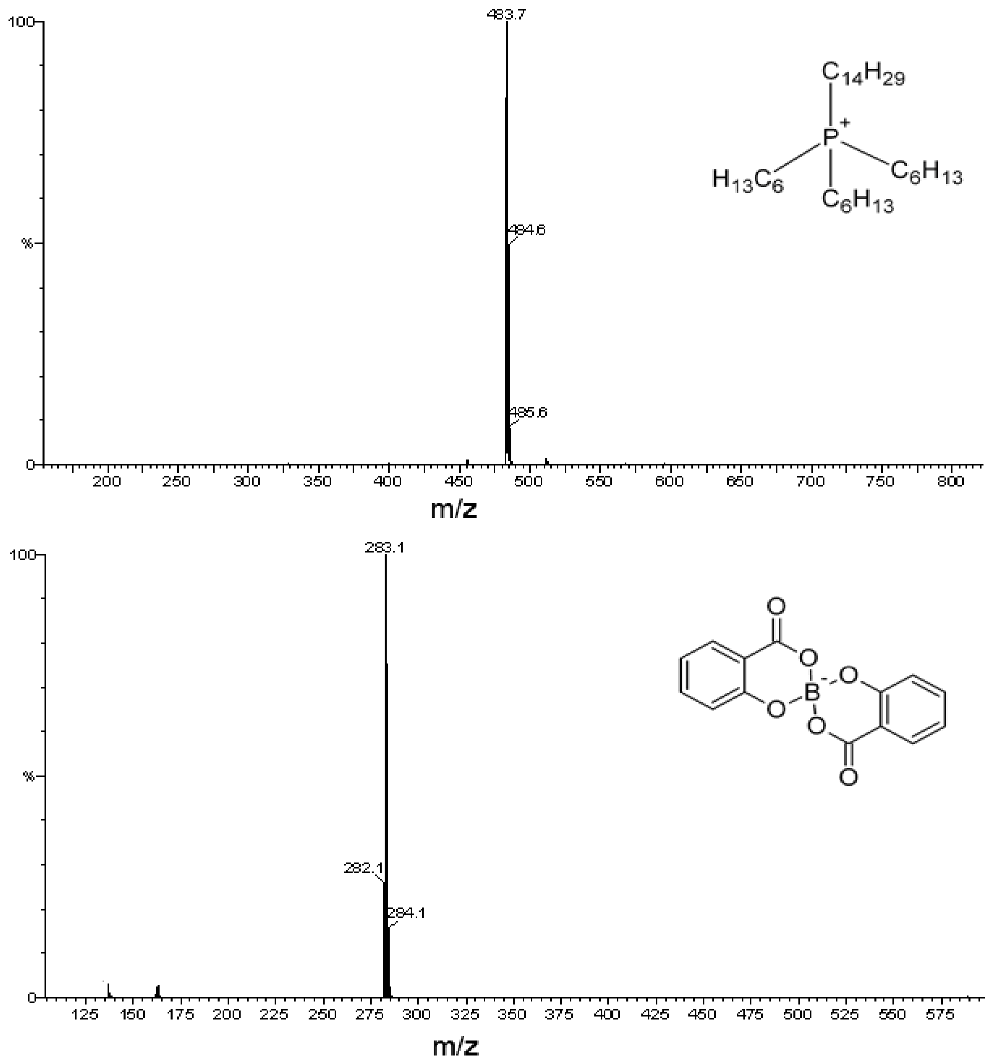

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Figure SI-8. ESI-MS of [$P_{6,6,6,14}$][BOB].

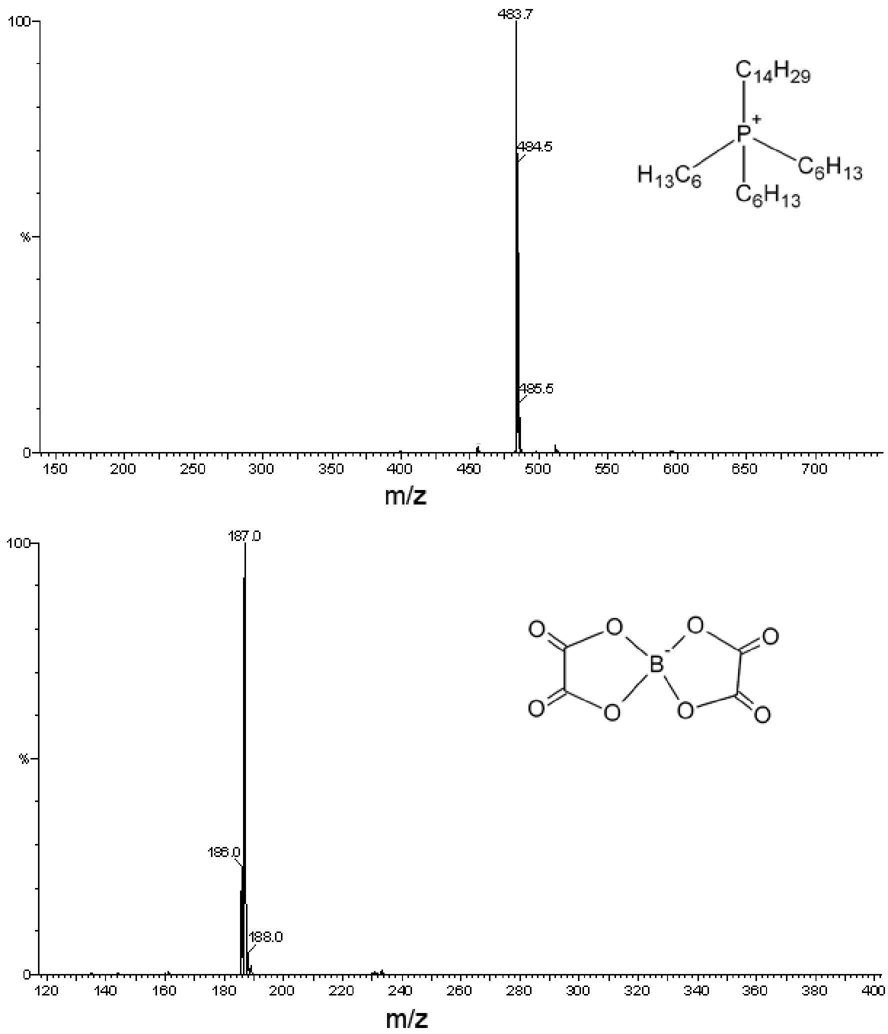

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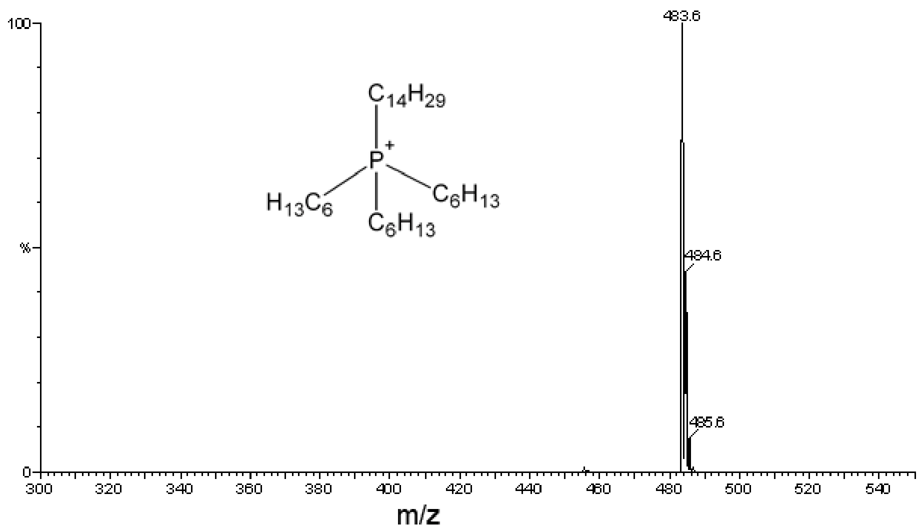
Figure SI-9. ESI-MS of [$P_{6,6,6,14}$][BMLB].



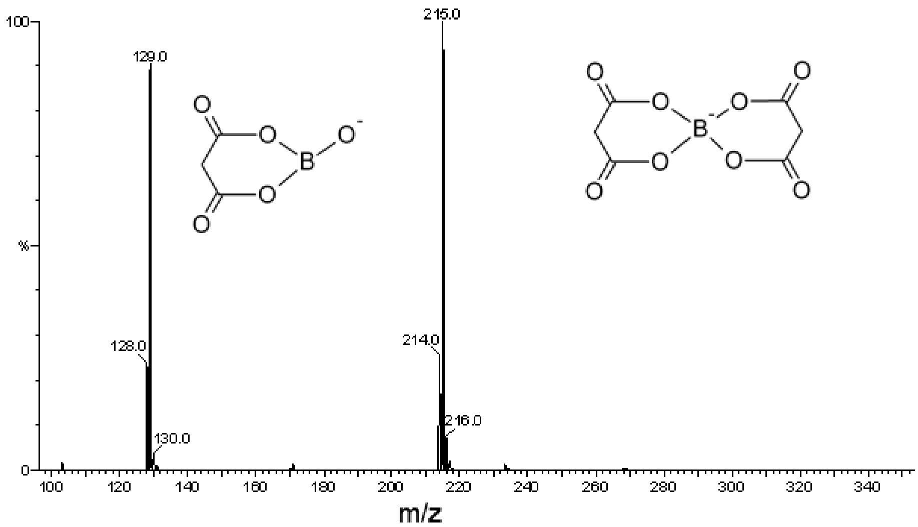


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Figure SI-10. 400.13 MHz ^{1}H NMR of $[P_{4,4,4,8}]$[BMB] in $CDCl_3$.

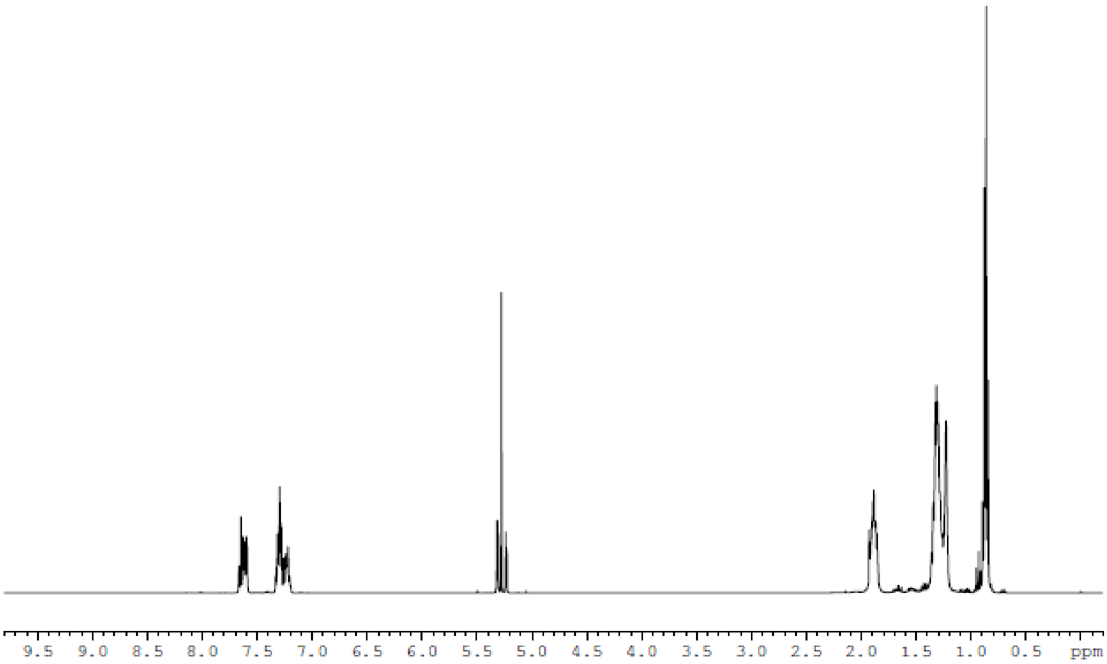

Figure SI-11. 100.62 MHz ^{13}C NMR of $[P_{4,4,4,8}]$[BMB] in $CDCl_3$.

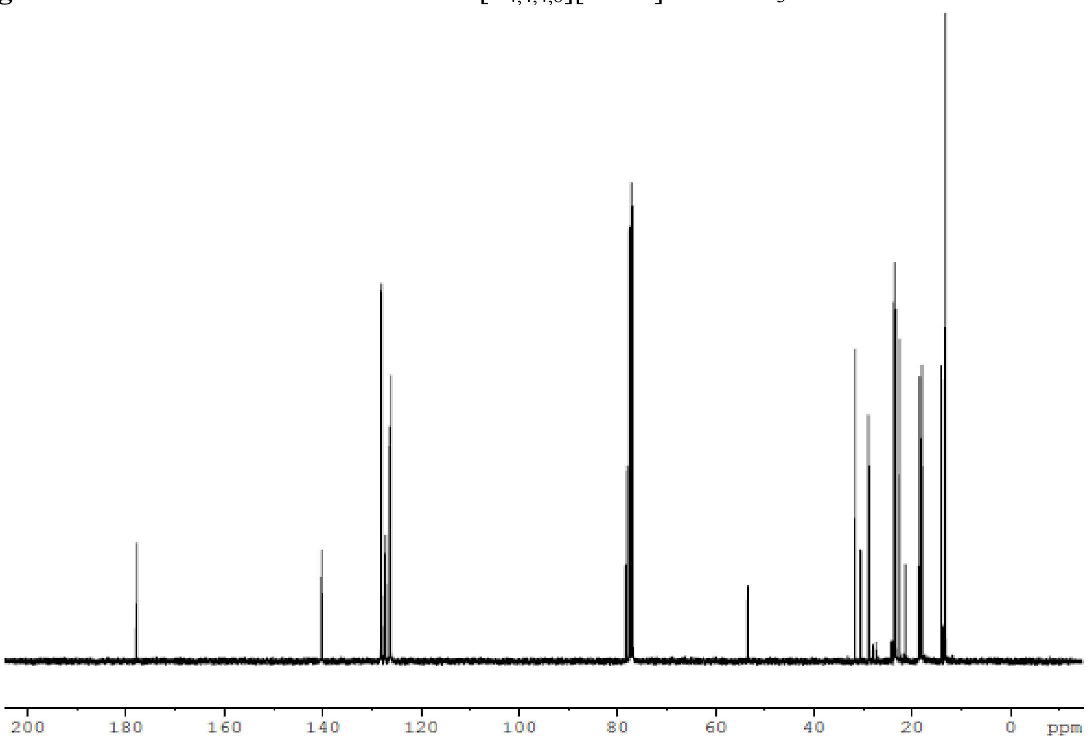

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Figure SI-12. 120.39 MHz ^{11}B NMR of $[P_{4,4,4,8}][BMB]$ in $CDCl_3$ (A broad background signal is from the sample tube).

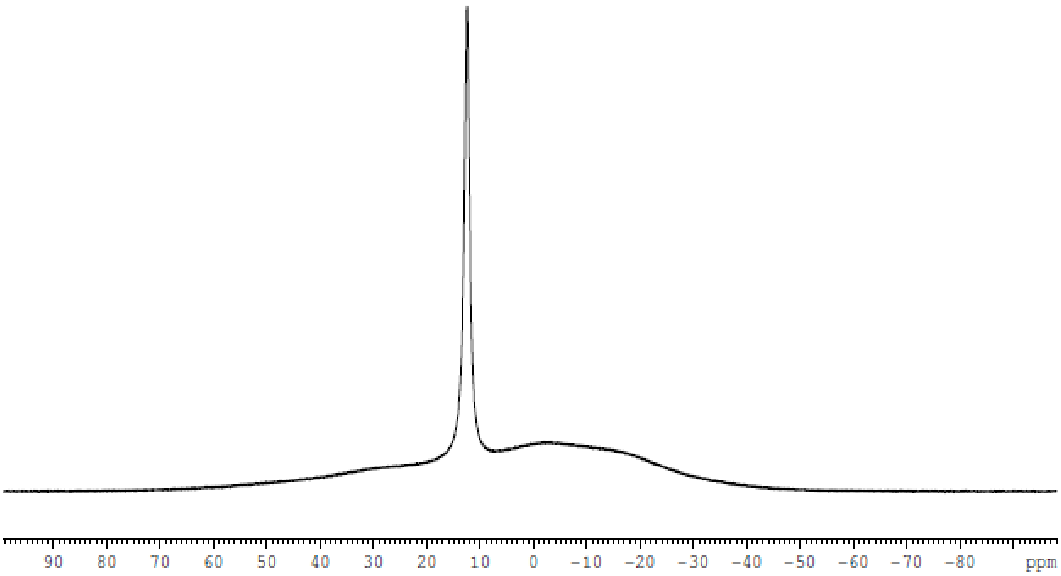

Figure SI-13. 161.99 MHz ^{31}P NMR of $[P_{4,4,4,8}][BMB]$ in $CDCl_3$.

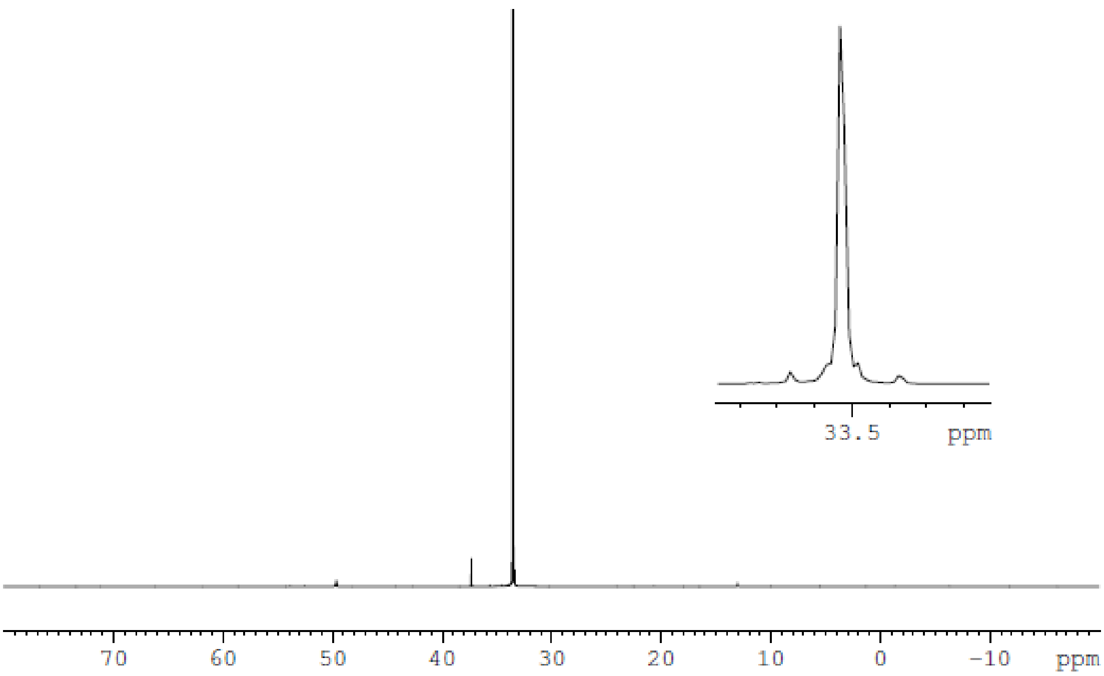

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Figure SI-14. 400.13 MHz [1]H NMR of $[P_{4,4,4,14}]$[BMB] in $CDCl_3$.

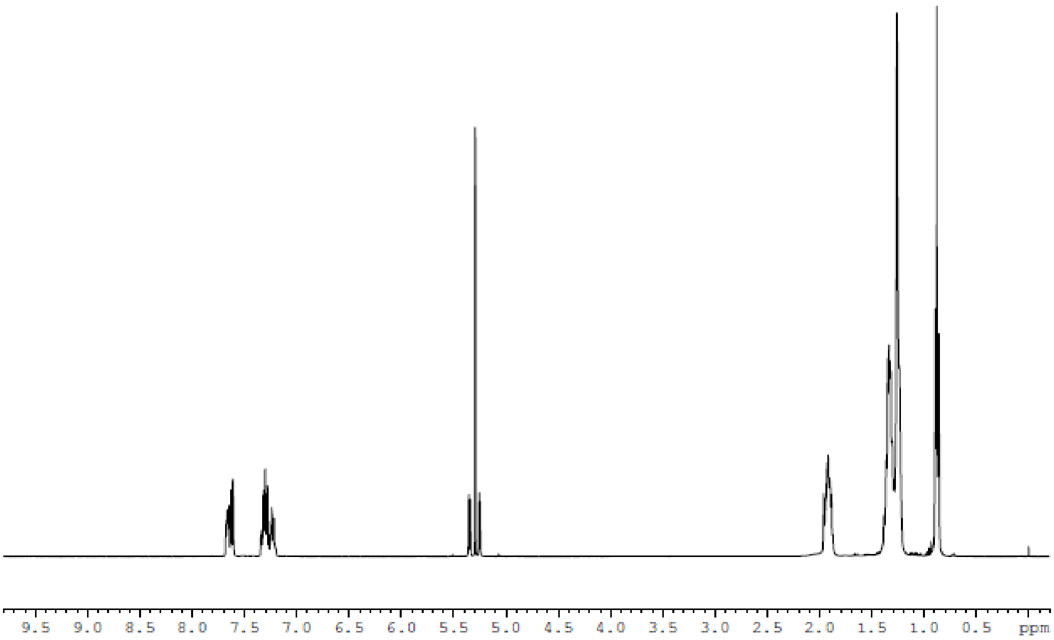

Figure SI-15. 100.62 MHz [13]C NMR of $[P_{4,4,4,14}]$[BMB] in $CDCl_3$.

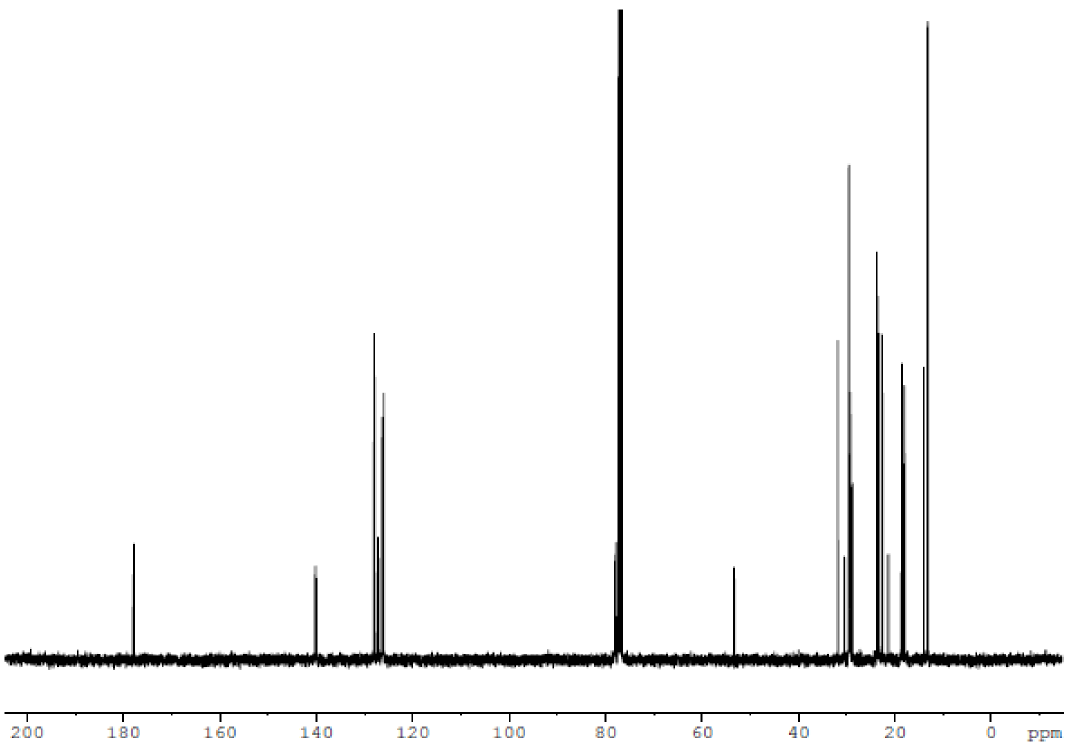

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Figure SI-16. 120.39 MHz ^{11}B NMR of $[P_{4,4,4,14}]$[BMB] in $CDCl_3$ (A broad background signal is from the sample tube).

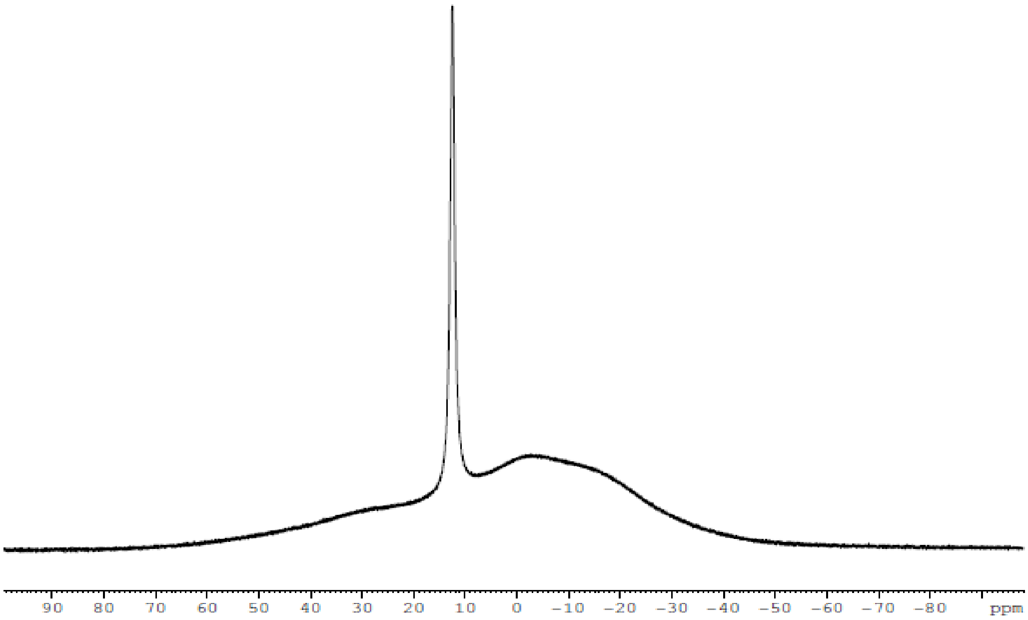

Figure SI-17. 161.99 MHz ^{31}P NMR of $[P_{4,4,4,14}]$[BMB] in $CDCl_3$.

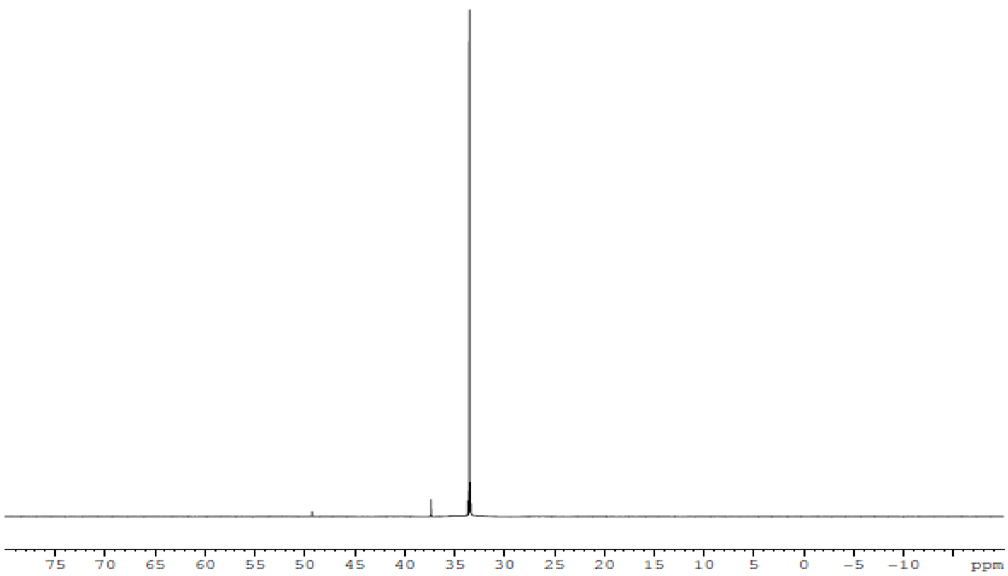

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Figure SI-18. 400.13 MHz ^{1}H NMR of $[P_{6,6,6,14}]$[BMB] in $CDCl_3$.

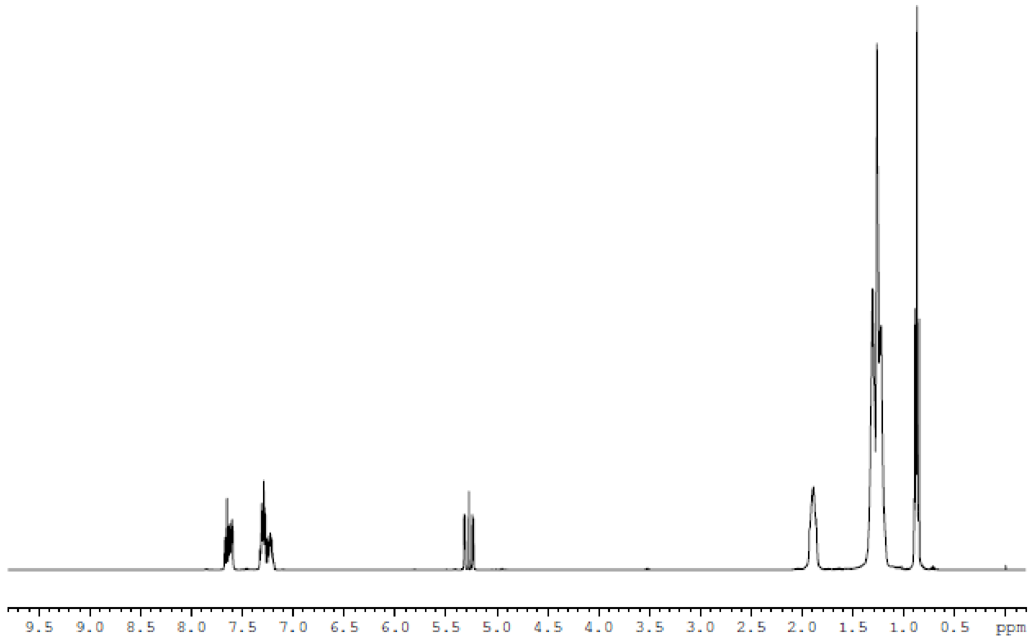

Figure SI-19. 100.62 MHz ^{13}C NMR of $[P_{6,6,6,14}]$[BMB] in $CDCl_3$.

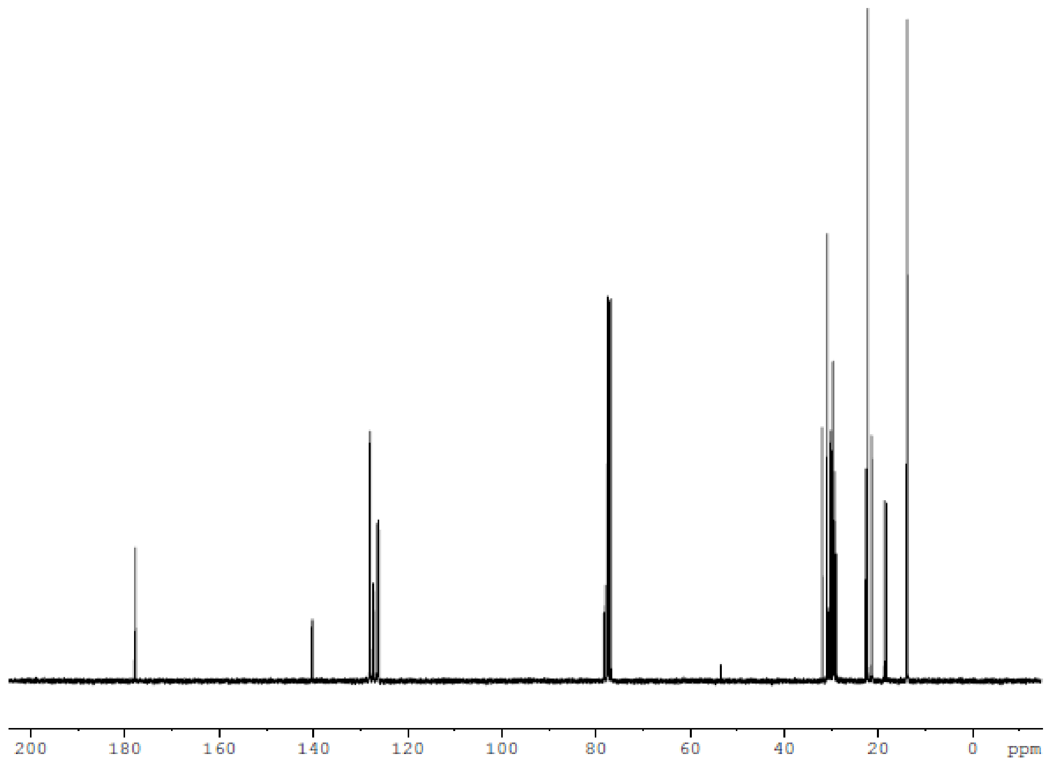

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Figure SI-20. 120.39 MHz ^{11}B NMR of $[P_{6,6,6,14}][BMB]$ in $CDCl_3$ (A broad background signal is from the sample tube).

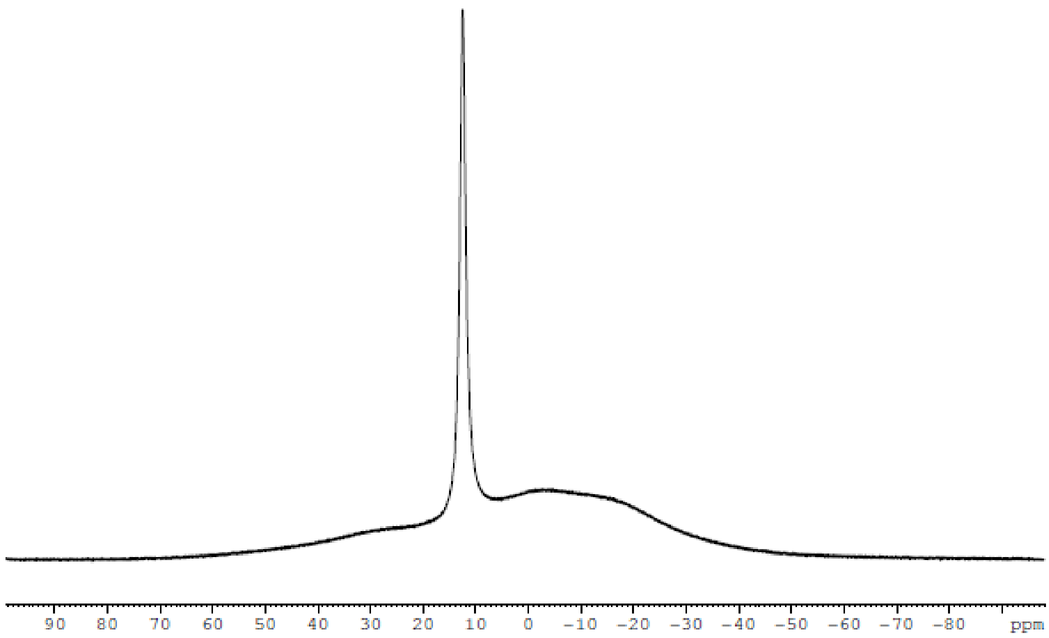

Figure SI-21. 161.99 MHz ^{31}P NMR of $[P_{6,6,6,14}][BMB]$ in $CDCl_3$.

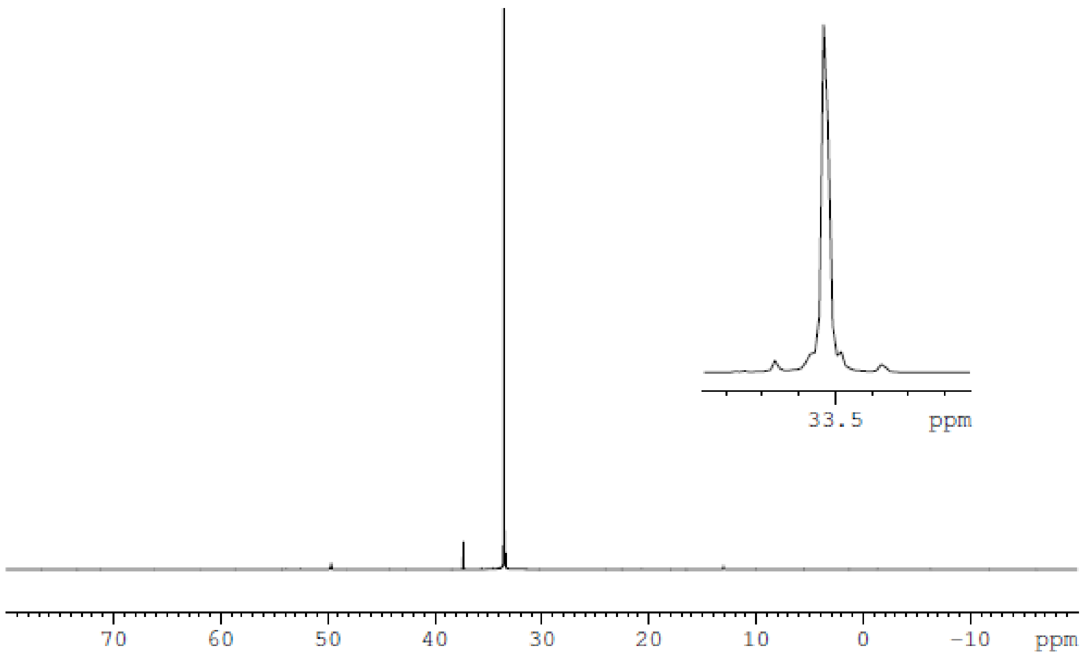

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Figure SI-22. 400.13 MHz ^{1}H NMR of $[P_{4,4,4,8}][BScB]$ in $CDCl_3$.

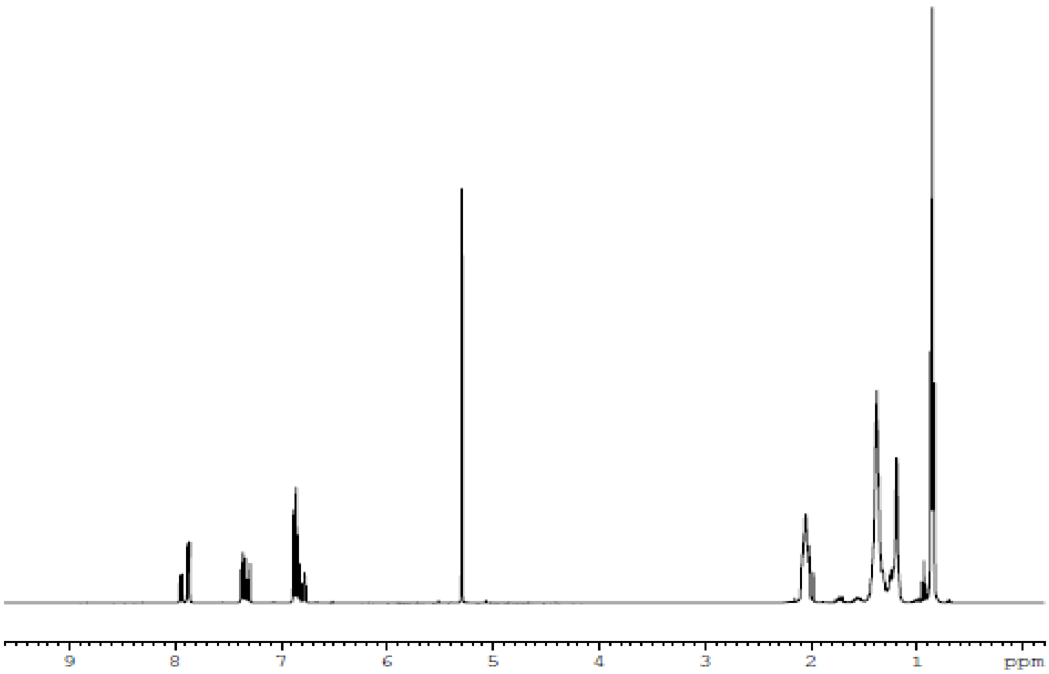

Figure SI-23. 100.62 MHz ^{13}C NMR of $[P_{4,4,4,8}][BScB]$ in $CDCl_3$.

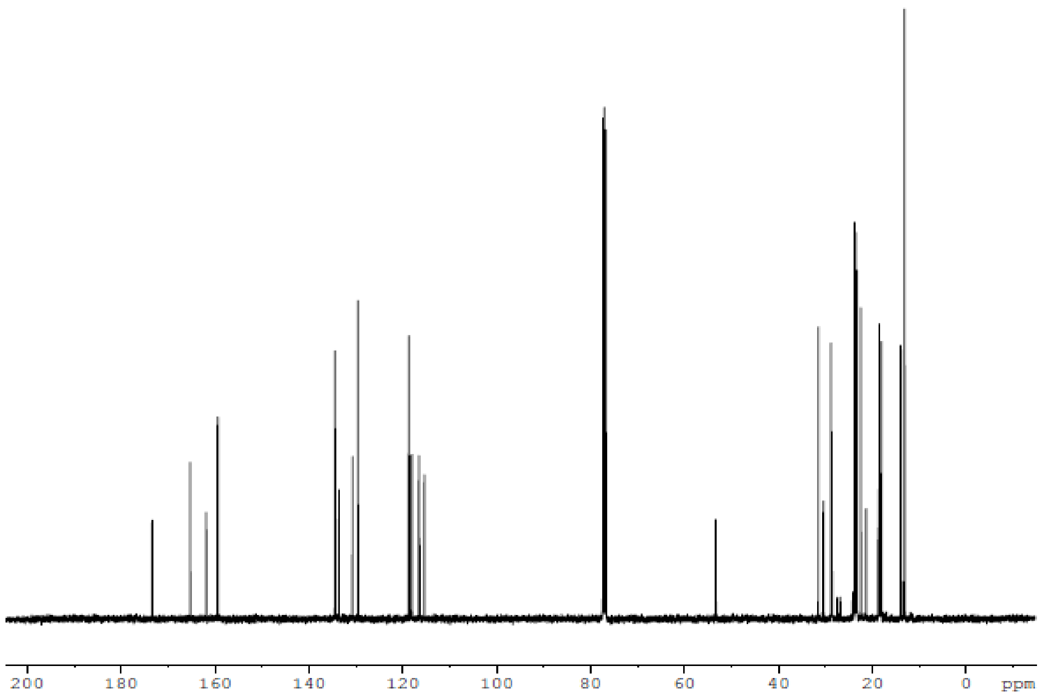

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Figure SI-24. 120.39 MHz ^{11}B NMR of $[P_{4,4,4,8}][BScB]$ in $CDCl_3$ (A broad background signal is from the sample tube).

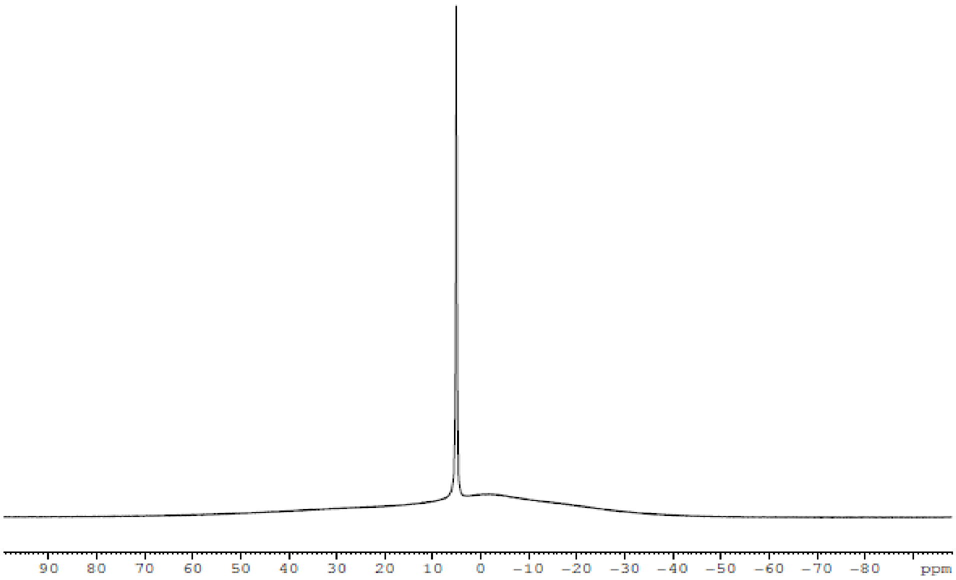

Figure SI-25. 161.99 MHz ^{31}P NMR of $[P_{4,4,4,8}][BScB]$ in $CDCl_3$.

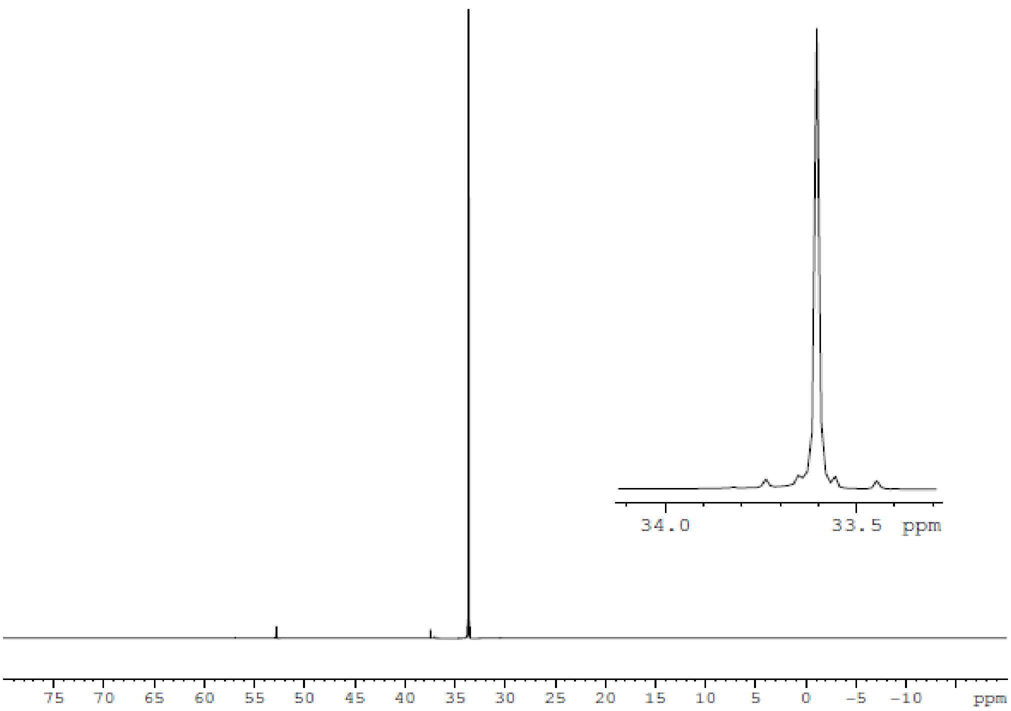

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Figure SI-26. 400.13 MHz ^{1}H NMR of $[P_{4,4,4,14}][BScB]$ in $CDCl_3$.

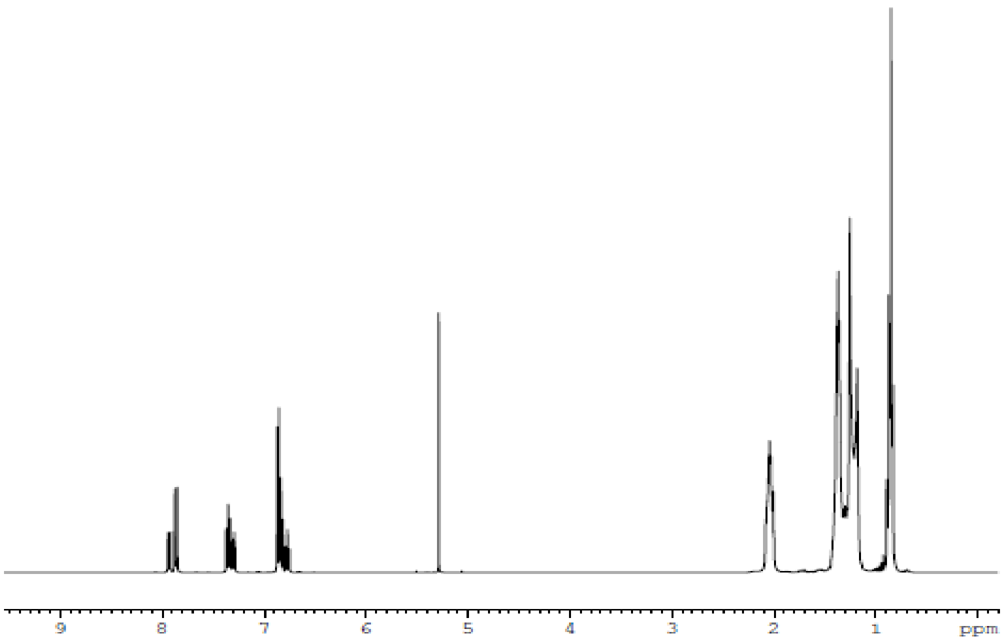

Figure SI-27. 100.62 MHz ^{13}C NMR of $[P_{4,4,4,14}][BScB]$ in $CDCl_3$.

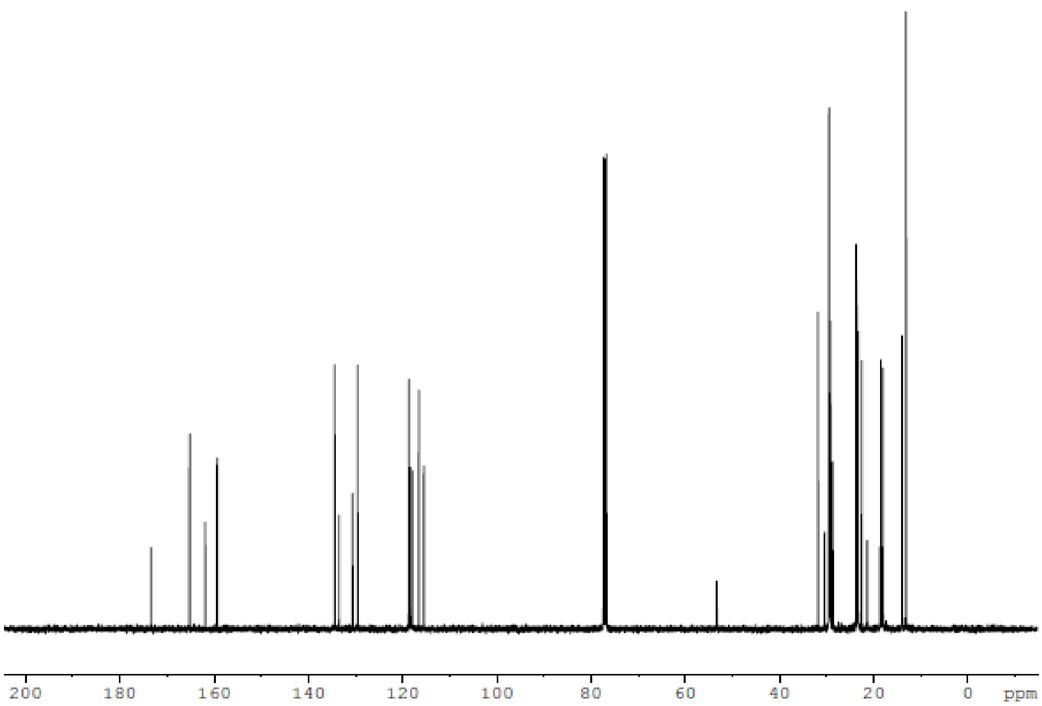

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Figure SI-28. 120.39 MHz ^{11}B NMR of $[P_{4,4,4,14}][BScB]$ in $CDCl_3$ (A broad background signal is from the sample tube).

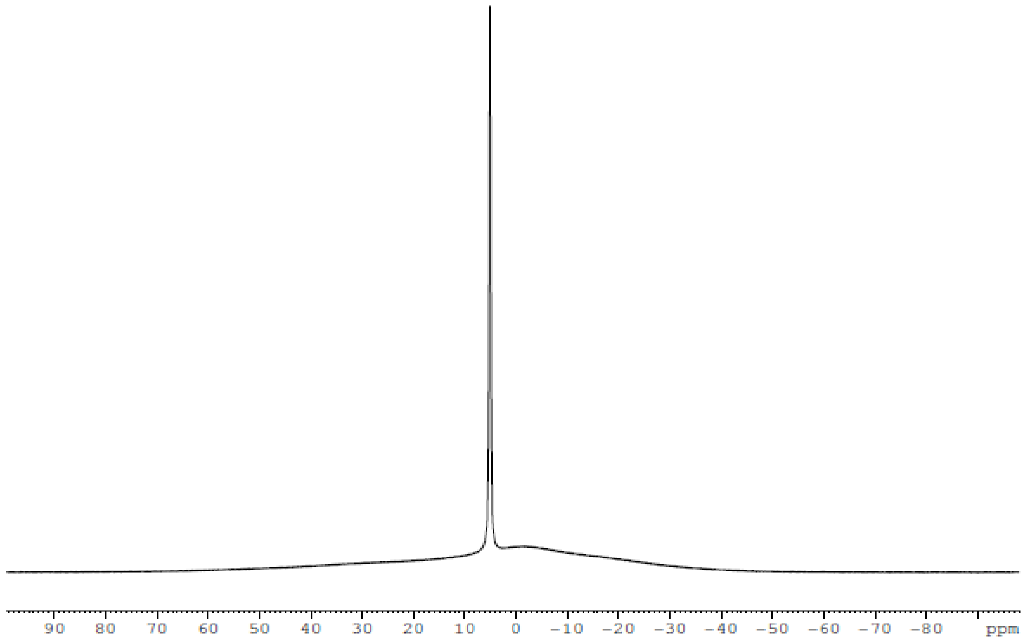

Figure SI-29. 161.99 MHz ^{31}P NMR of $[P_{4,4,4,14}][BScB]$ in $CDCl_3$.

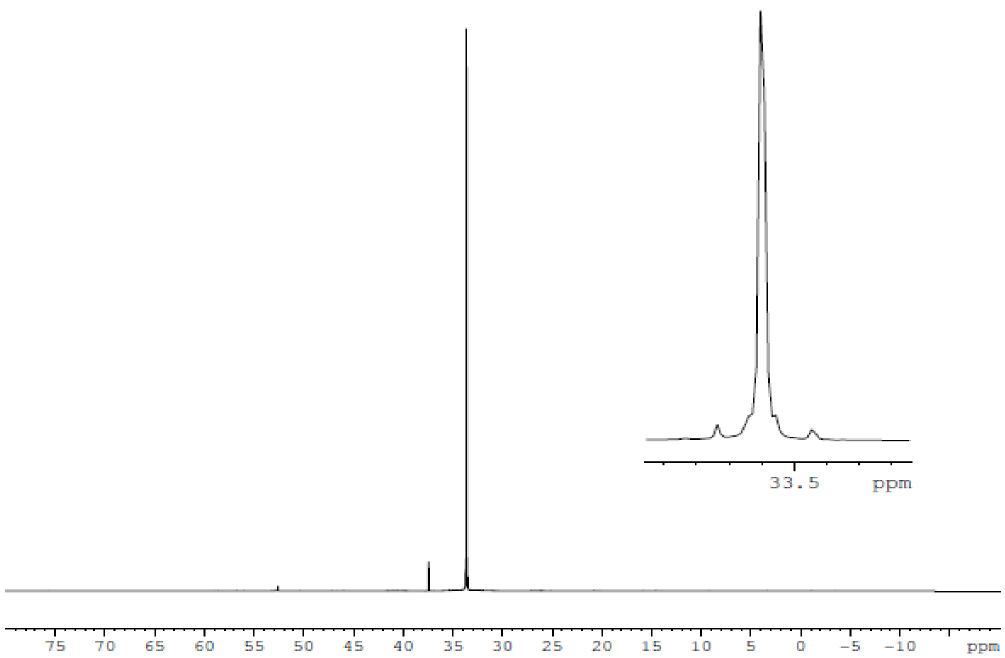

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Figure SI-30. 400.13 MHz ^{1}H NMR of $[P_{6,6,6,14}]$[BScB] in $CDCl_3$.

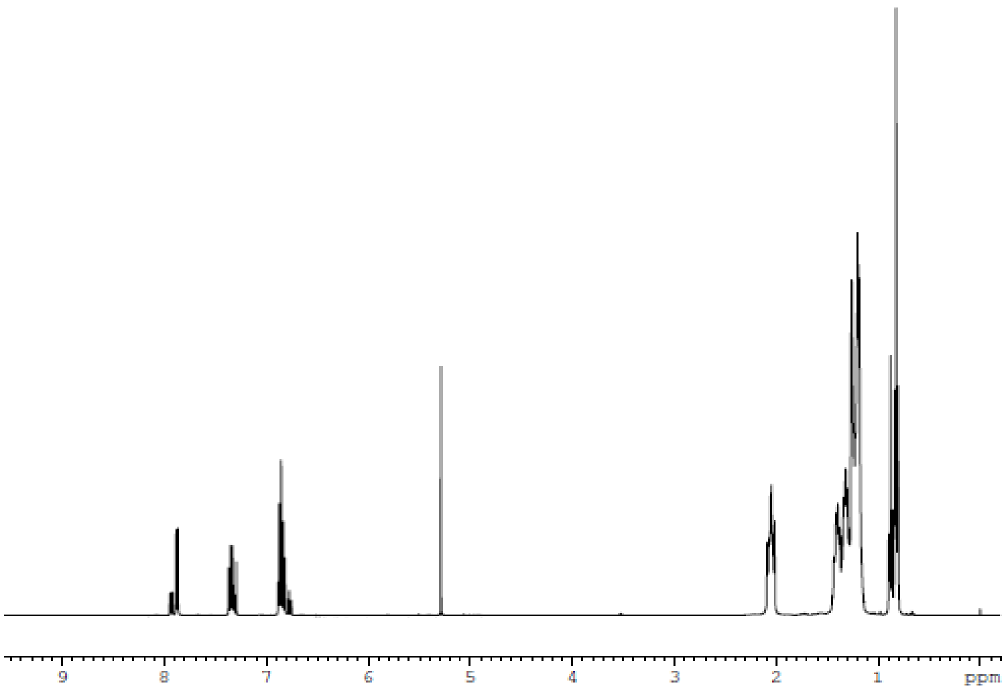

Figure SI-31. 100.62 MHz ^{13}C NMR of $[P_{6,6,6,14}]$[BScB] in $CDCl_3$.

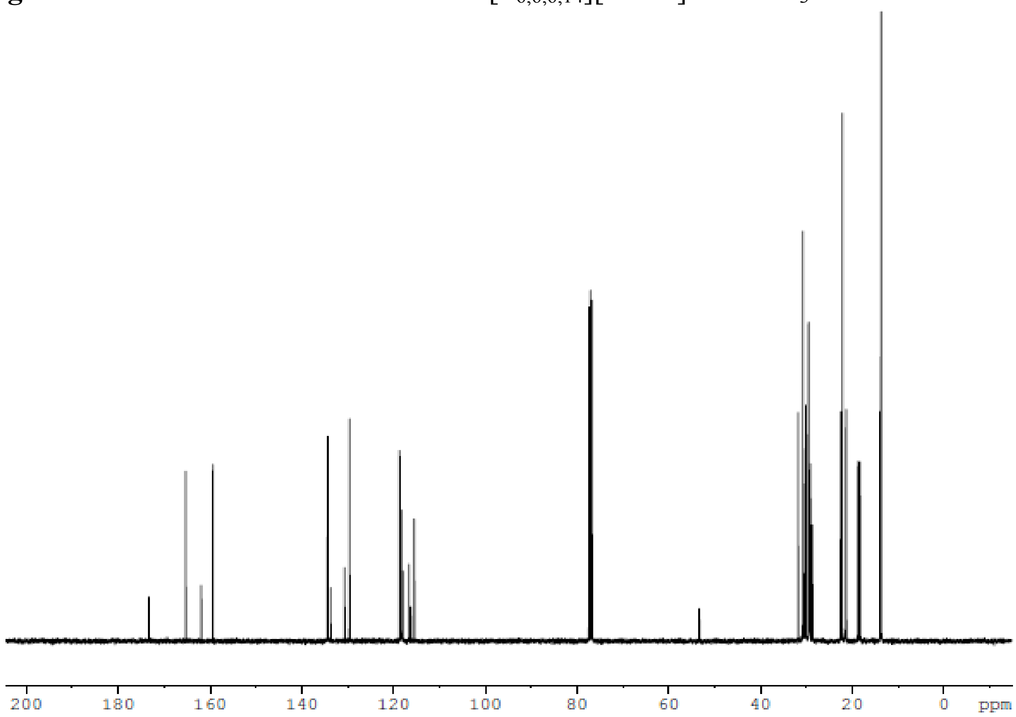

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Figure SI-32: 120.39 MHz ^{11}B NMR of $[P_{6,6,6,14}][BScB]$ in $CDCl_3$ (A broad background signal is from the sample tube).

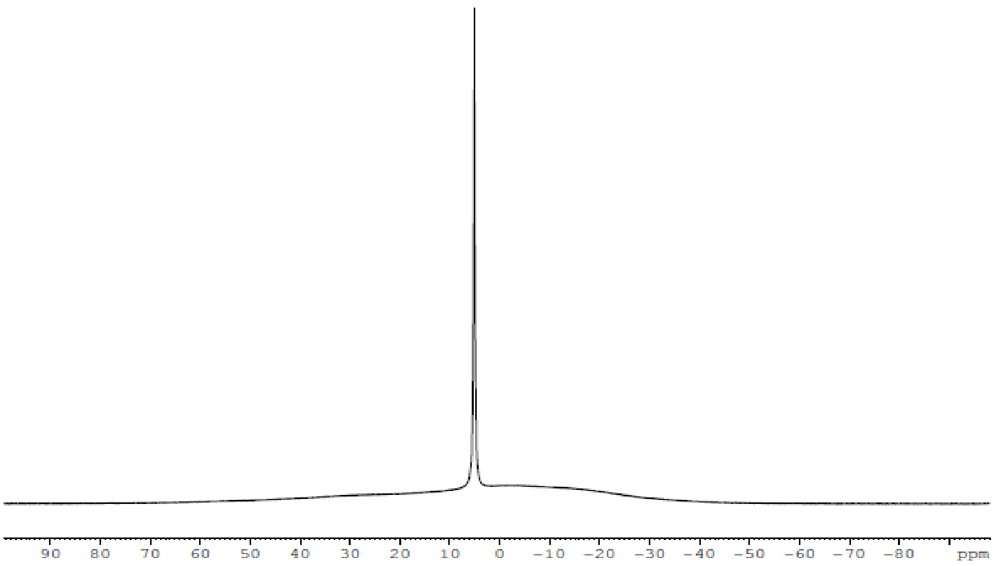

Figure SI-33. 161.99 MHz ^{31}P NMR of $[P_{6,6,6,14}][BScB]$ in $CDCl_3$.

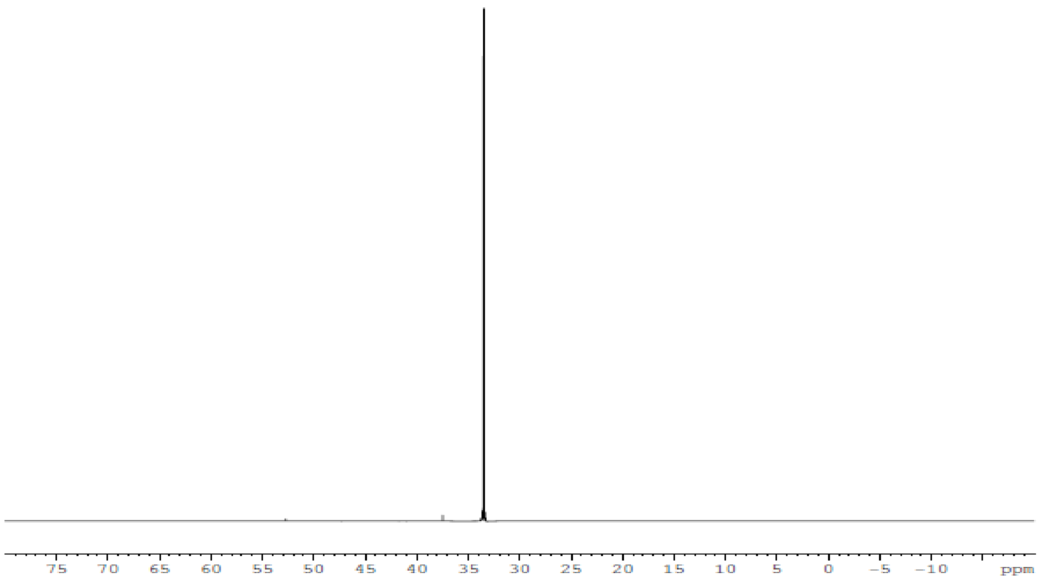

Figure SI-34. 400.13 MHz ^{1}H NMR of $[P_{6,6,6,14}]$[BOB] in $CDCl_3$.

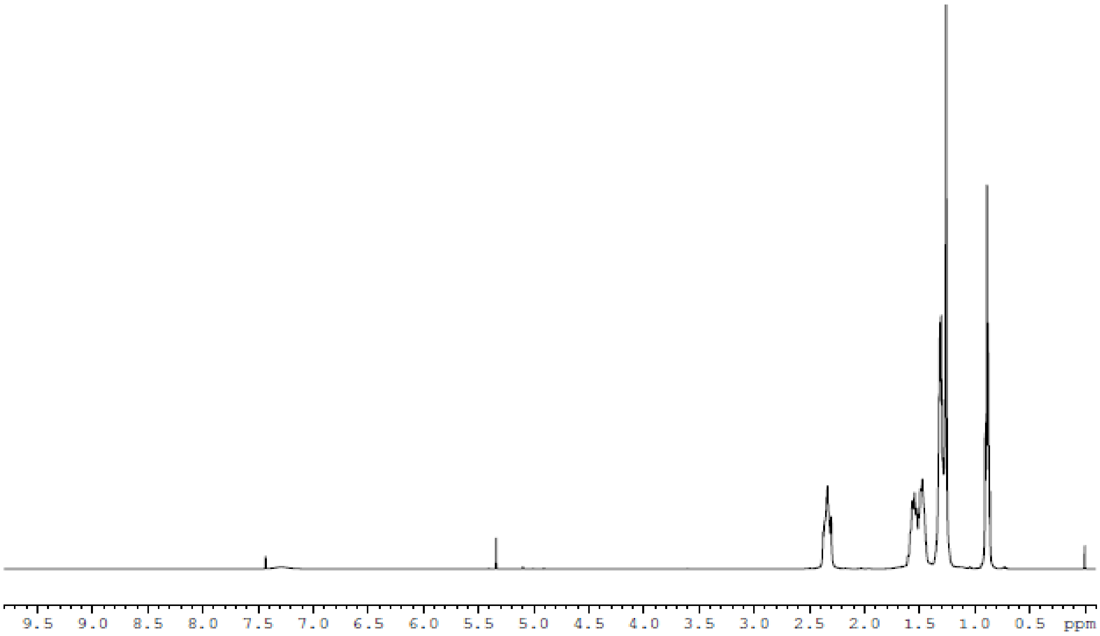

Figure SI-35. 100.62 MHz ^{13}C NMR of $[P_{6,6,6,14}]$[BOB] in $CDCl_3$.

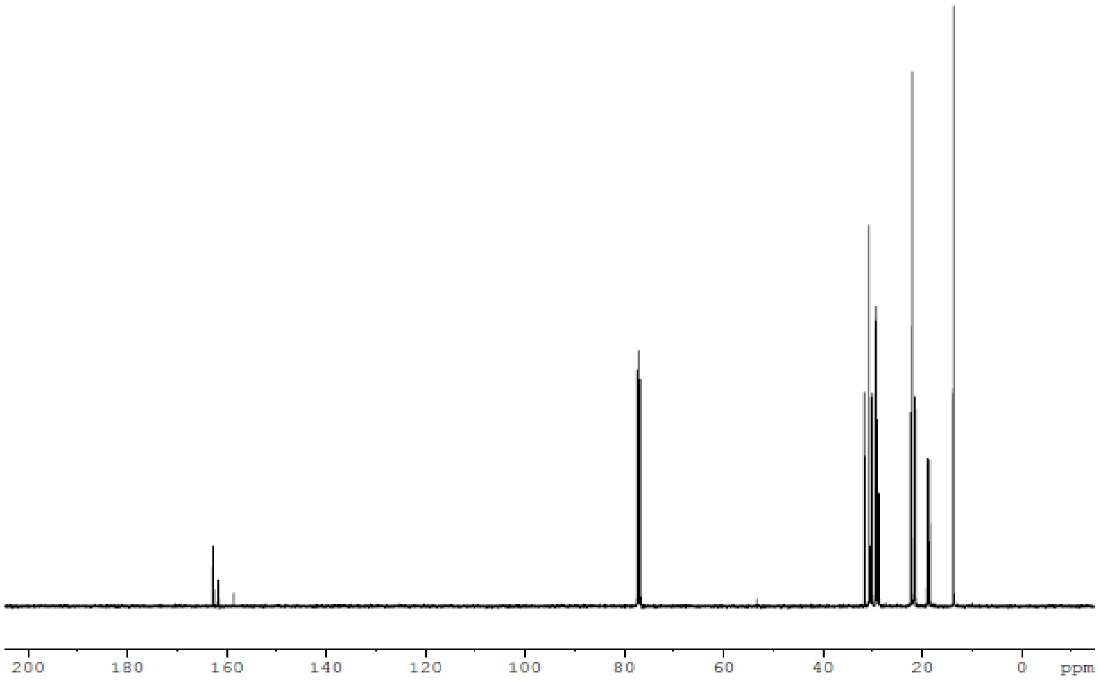

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Figure SI-36. 120.39 MHz ^{11}B NMR of $[P_{6,6,6,14}][BOB]$ in $CDCl_3$ (A broad background signal is from the sample tube).

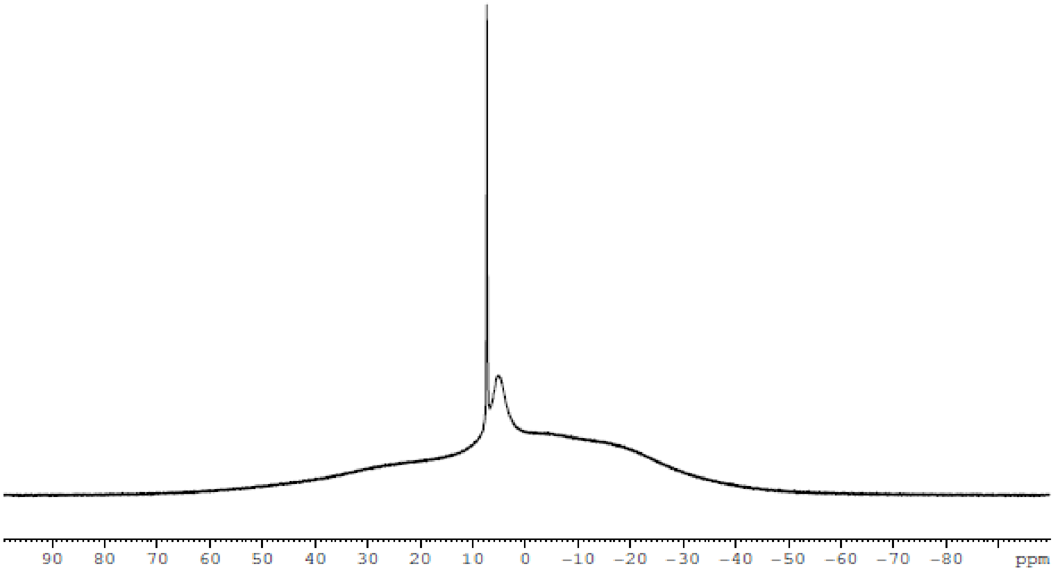

Figure SI-37. 161.99 MHz ^{31}P NMR of $[P_{6,6,6,14}][BOB]$ in $CDCl_3$.

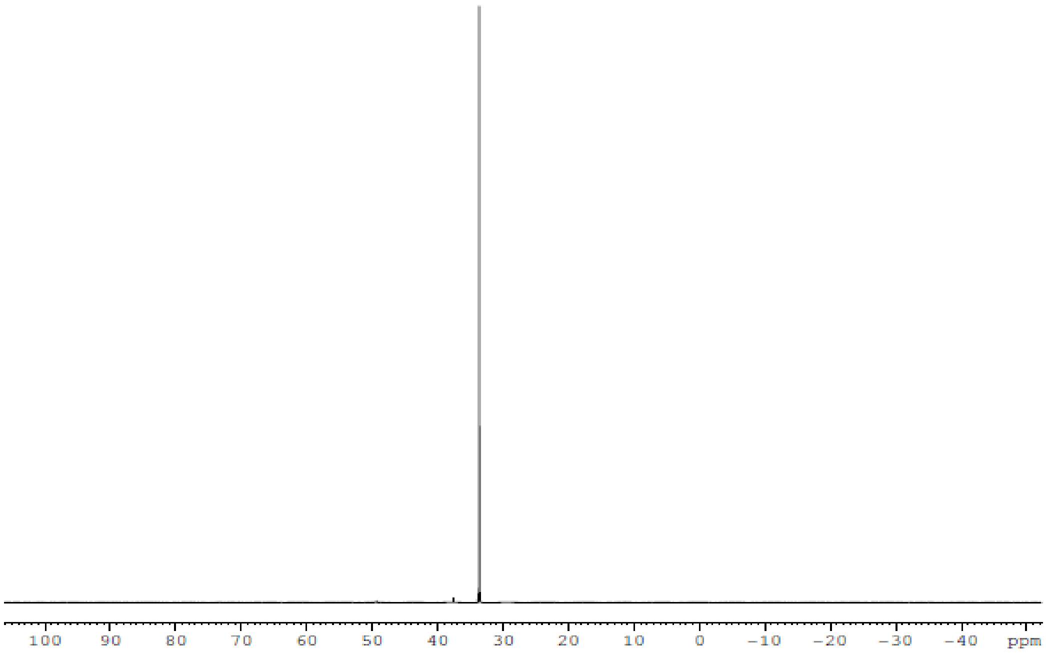

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Figure SI-38. 400.13 MHz ^{1}H NMR of $[P_{6,6,6,14}]$[BMLB] in $CDCl_3$.

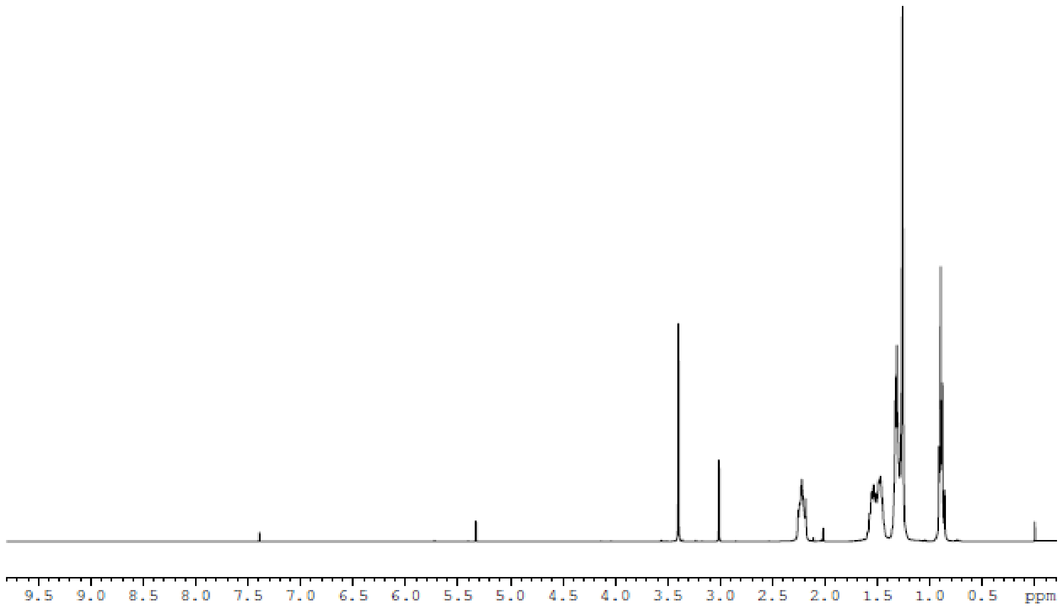

Figure SI-39. 100.62 MHz ^{13}C NMR of $[P_{6,6,6,14}]$[BMLB] in $CDCl_3$.

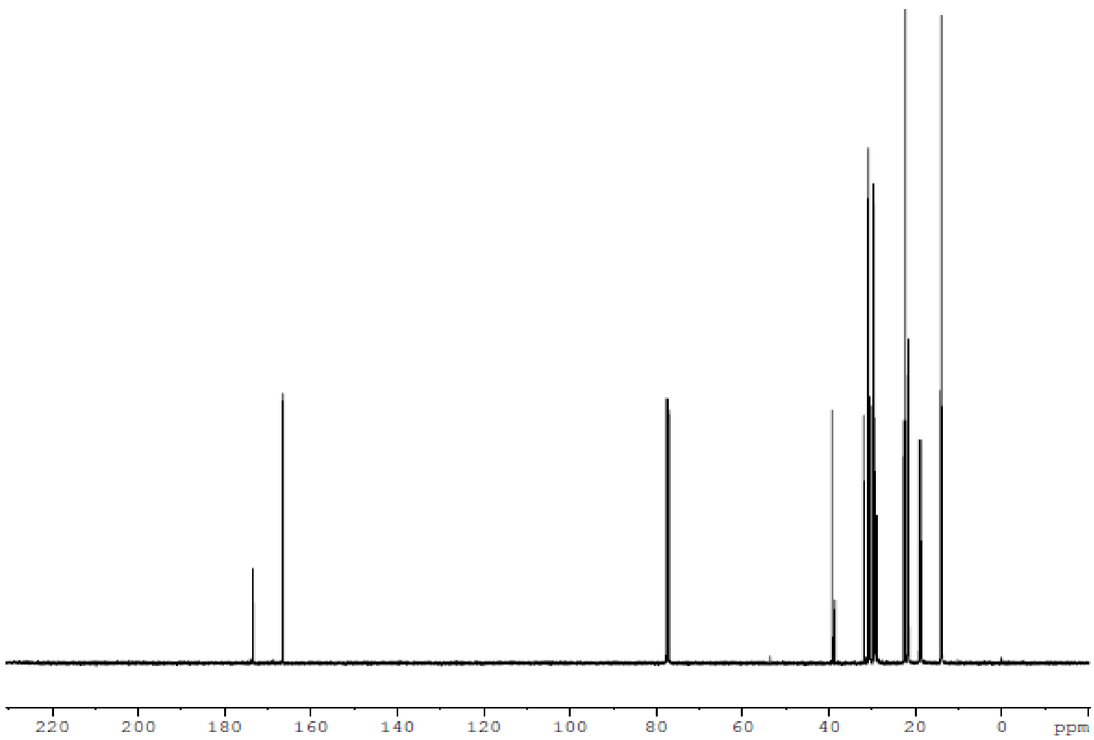

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Figure SI-40. 120.39 MHz ^{11}B NMR of $[P_{6,6,6,14}]$[BMLB] in $CDCl_3$ (A broad background signal is from the sample tube).

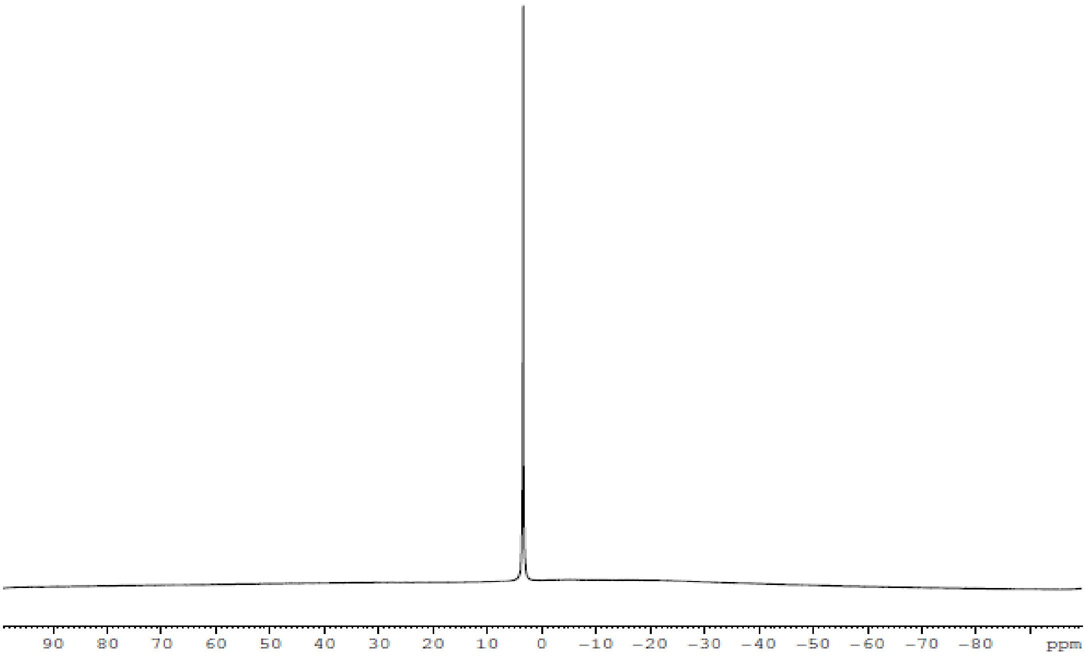

Figure SI-41. 161.99 MHz ^{31}P NMR of $[P_{6,6,6,14}]$[BMLB] in $CDCl_3$.

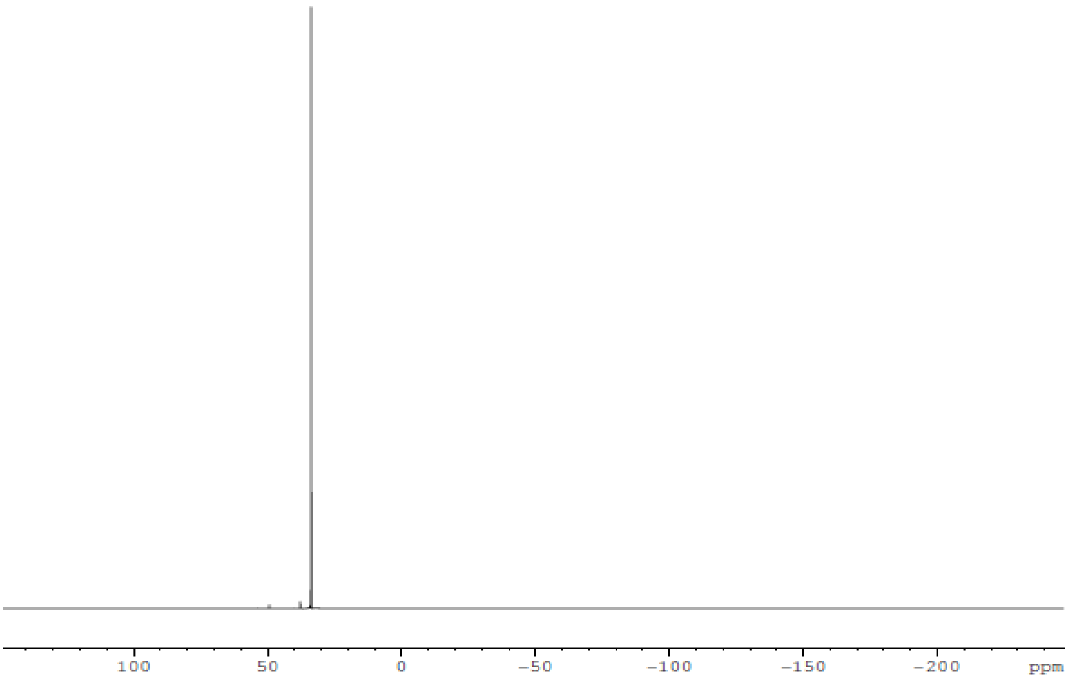

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Table SI-2. Composition, hardness and surface roughness of the alloys used.

Elemental Composition (wt%)	Alloy	
	AA2024	100Cr6
C	–	0.98–1.10
Cu	3.8–4.9	–
Si	0.5 max	0.15–0.3
Mn	0.3–0.9	0.25–0.45
Mg	1.2–1.8	–
Cr	0.1 max	1.3–1.6
Zn	0.25 max	
Ti	0.15 max	
S	–	0.025 max
P	–	0.025 max
Others	0.15 max	–
Fe	0.5 max	Balance
Al	Balance	–
Hardness (Vickers)	145	850
R_a (µm)	0.09	0.05 max

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Figure SI-42. 3D images of the worn aluminium discs lubricated with 15W-50 oil and $[P_{6,6,6,14}]$[BMB] (load 40 N and 100 m distance at a speed of 0.2 m/s). These images clearly indicate a better antiwear performance of $[P_{6,6,6,14}]$[BMB] in comparison with that of 15W-50 oil. A disc lubricated with 15W-50 oil has acquired significantly larger wear scar (Ra = 1.77 μm) compared to the one lubricated with $[P_{6,6,6,14}]$[BMB] (Ra = 0.829 μm).