

Supporting Information

Metallacarborane Synthons for Molecular Construction - Functionalization of Cobalt bis(1,2-dicarbollide) with Extendable Ligands

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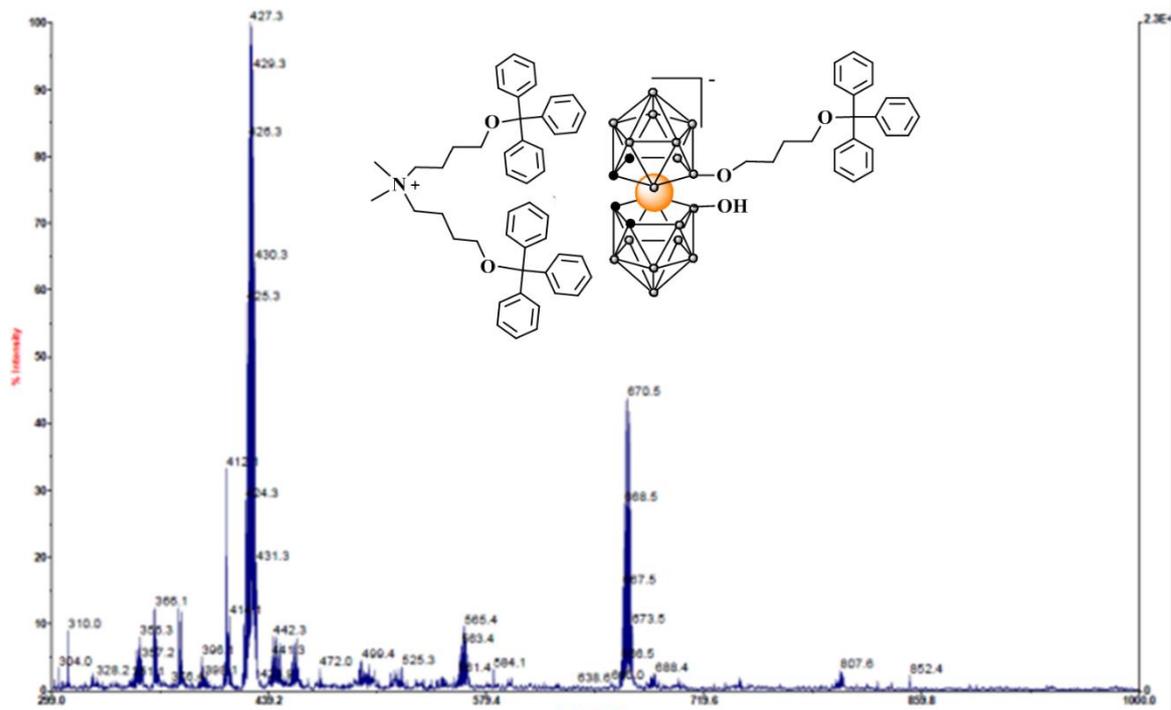


Figure S1. MS (ESI) spectrum of 3,3'-Co{[(8-O(CH₂)₄OCPH₃]-1,2-C₂B₉H₁₀)}(8'-OH-1',2'-C₂B₉H₁₀)(CH₃)₂N[(CH₂)₄OPh₃]₂ (**5**)

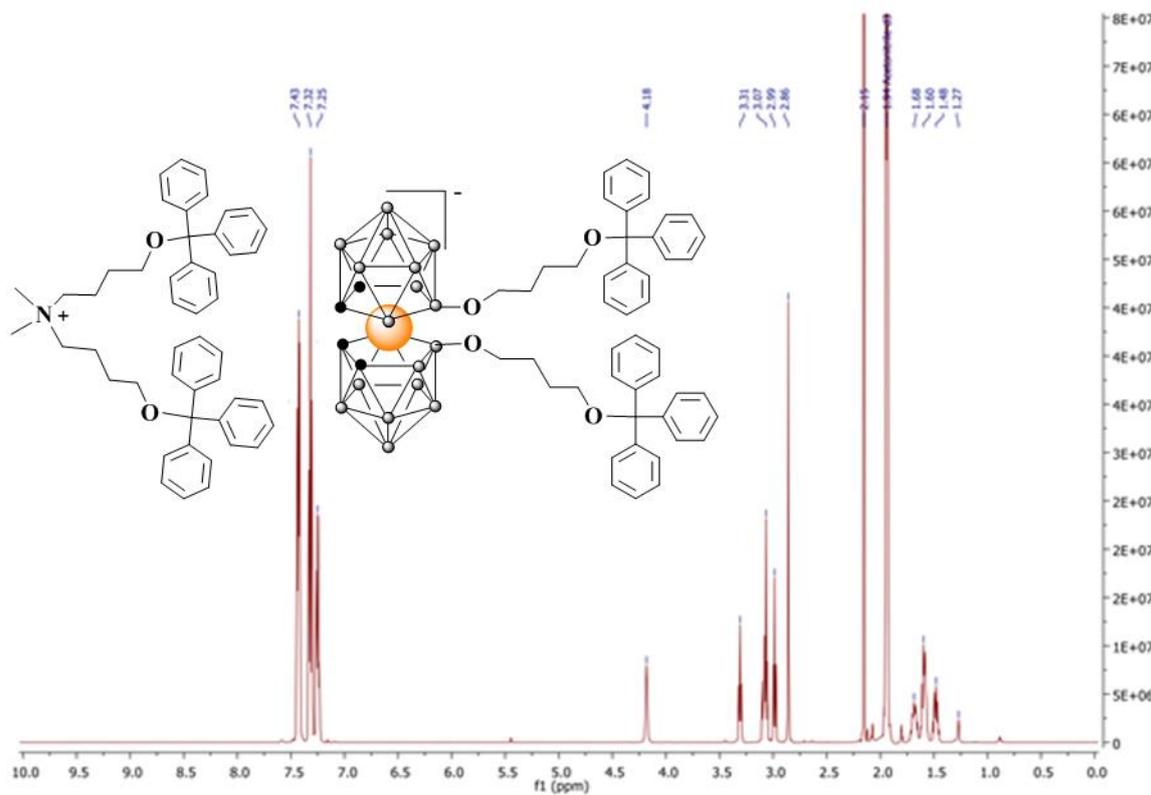


Figure S2. ¹H NMR spectrum of 3,3'-Co[(8-O(CH₂)₄OCPH₃-1,2-C₂B₉H₁₀)]₂(CH₃)₂N[(CH₂)₄OPh₃]₂ (**7**)

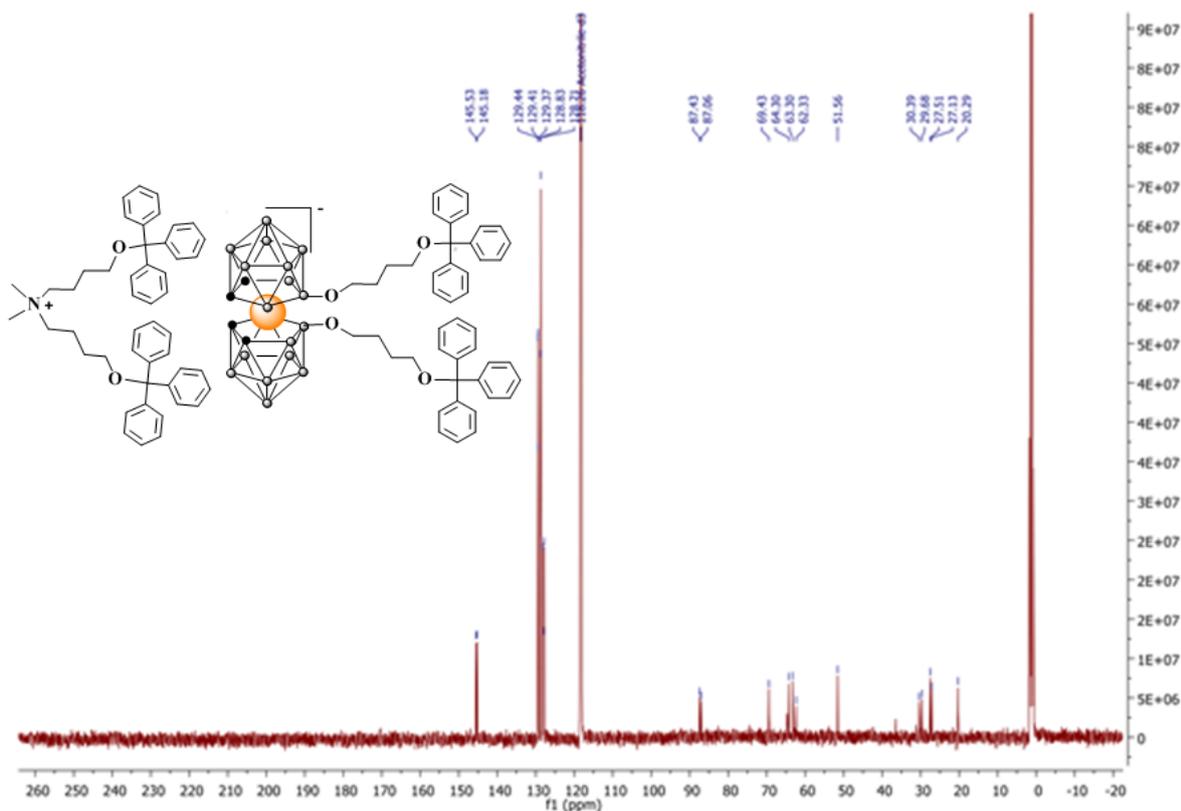


Figure S3. ¹³C NMR spectrum of $3,3'$ -Co[(8-O(CH₂)₄OCPh₃-1,2-C₂B₉H₁₀)]₂(CH₃)₂N[(CH₂)₄OPh₃]₂ (7)

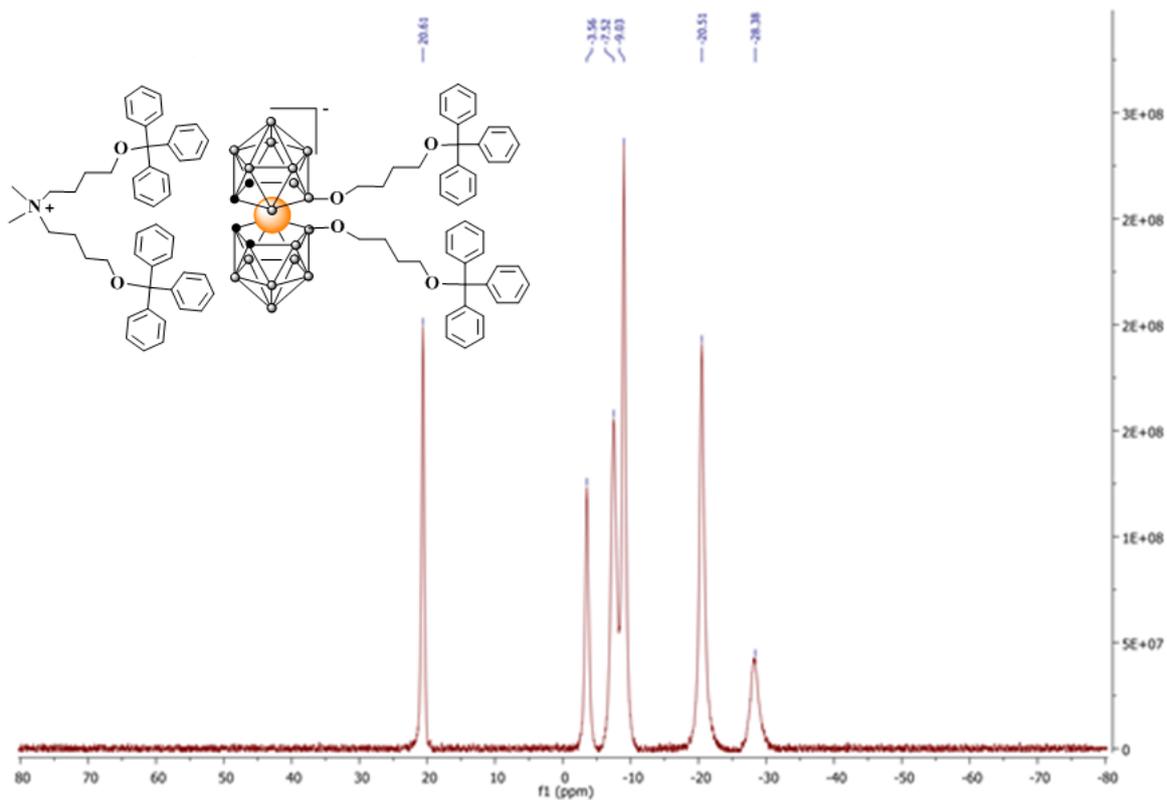


Figure S4. ¹¹B{¹H} NMR spectrum of $3,3'$ -Co[(8-O(CH₂)₄OCPh₃-1,2-C₂B₉H₁₀)]₂(CH₃)₂N[(CH₂)₄OPh₃]₂ (7)

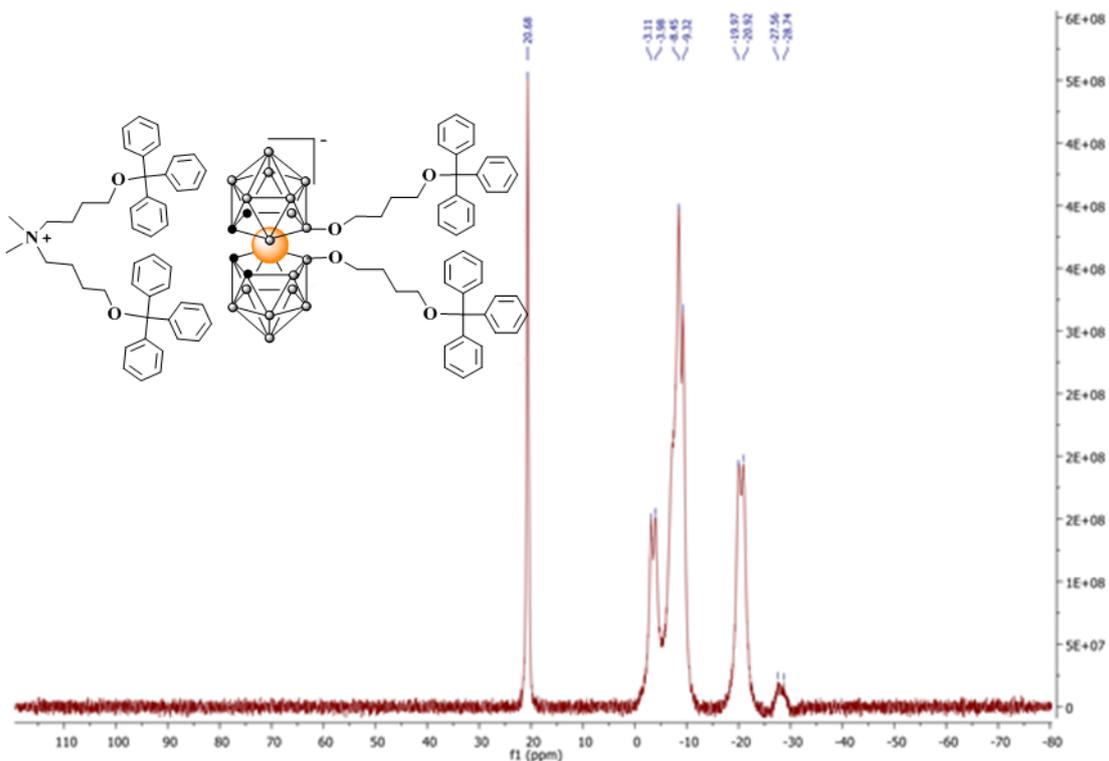


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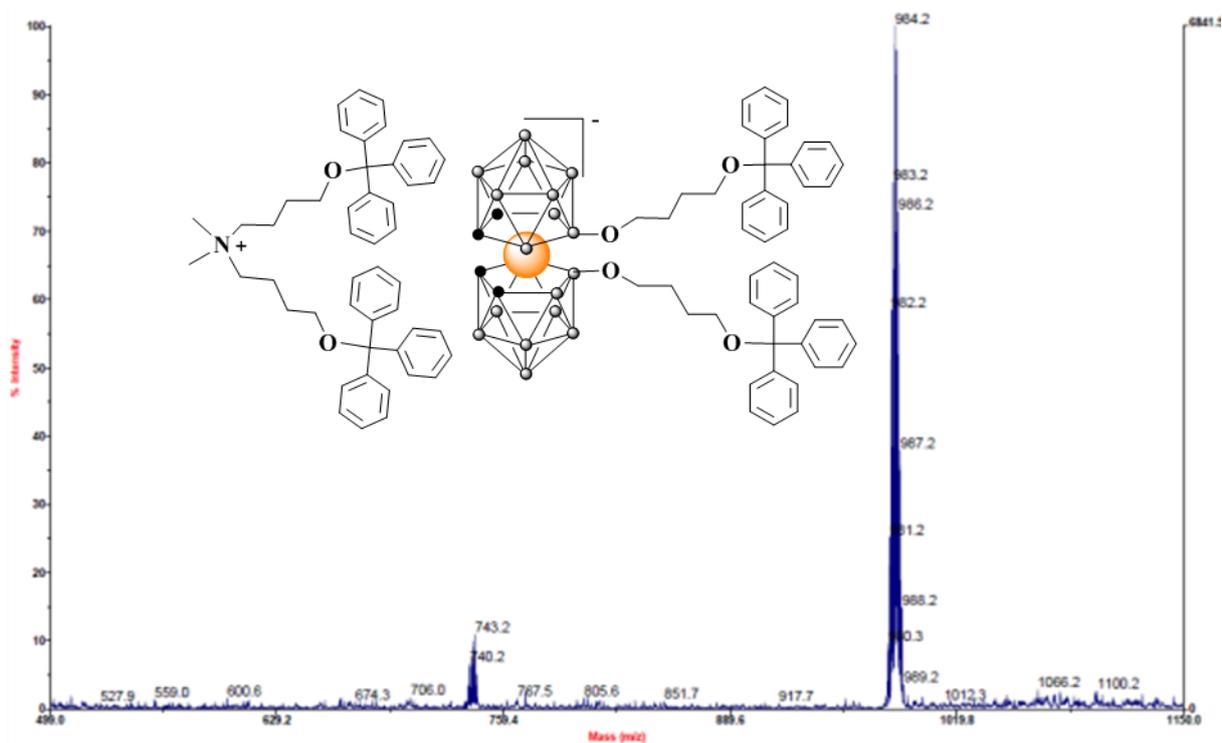


Figure S6. MS (ESI) spectrum of $3,3'$ -Co[(8-O(CH₂)₄OCPh₃-1,2-C₂B₉H₁₀)₂ (CH₃)₂N[(CH₂)₄OPh₃]₂ (**7**)

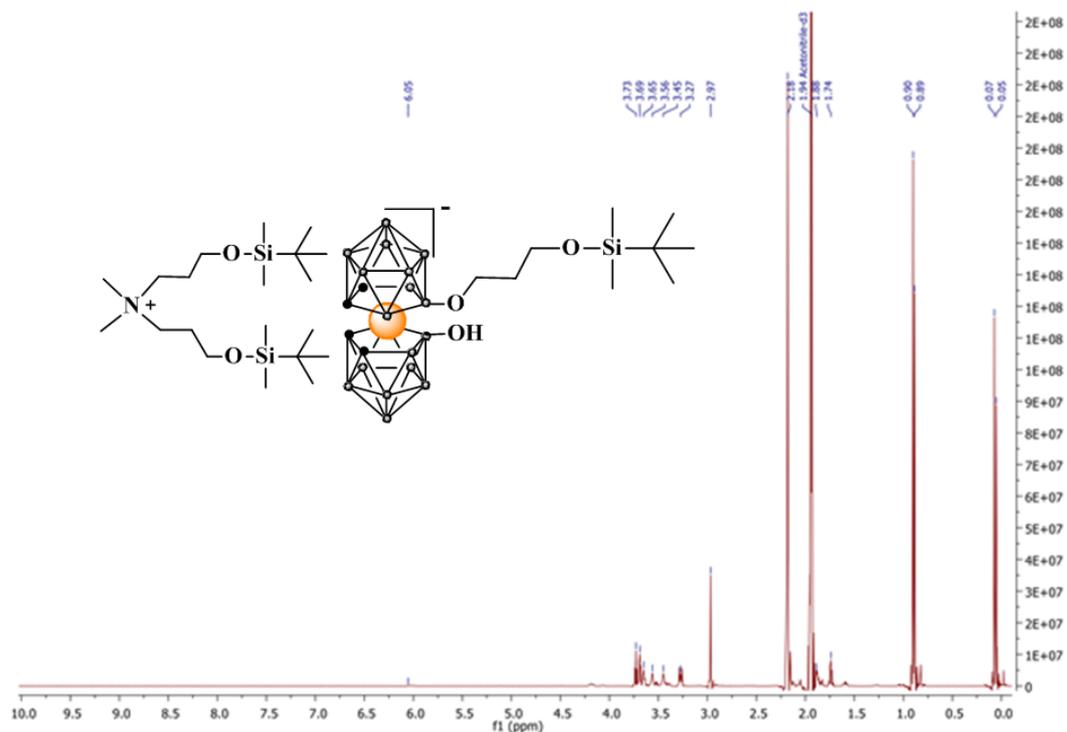


Figure S7. ^1H NMR spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS]-1,2-C₂B₉H₁₀(8'-OH-1,2-C₂B₉H₁₀) (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**6**)

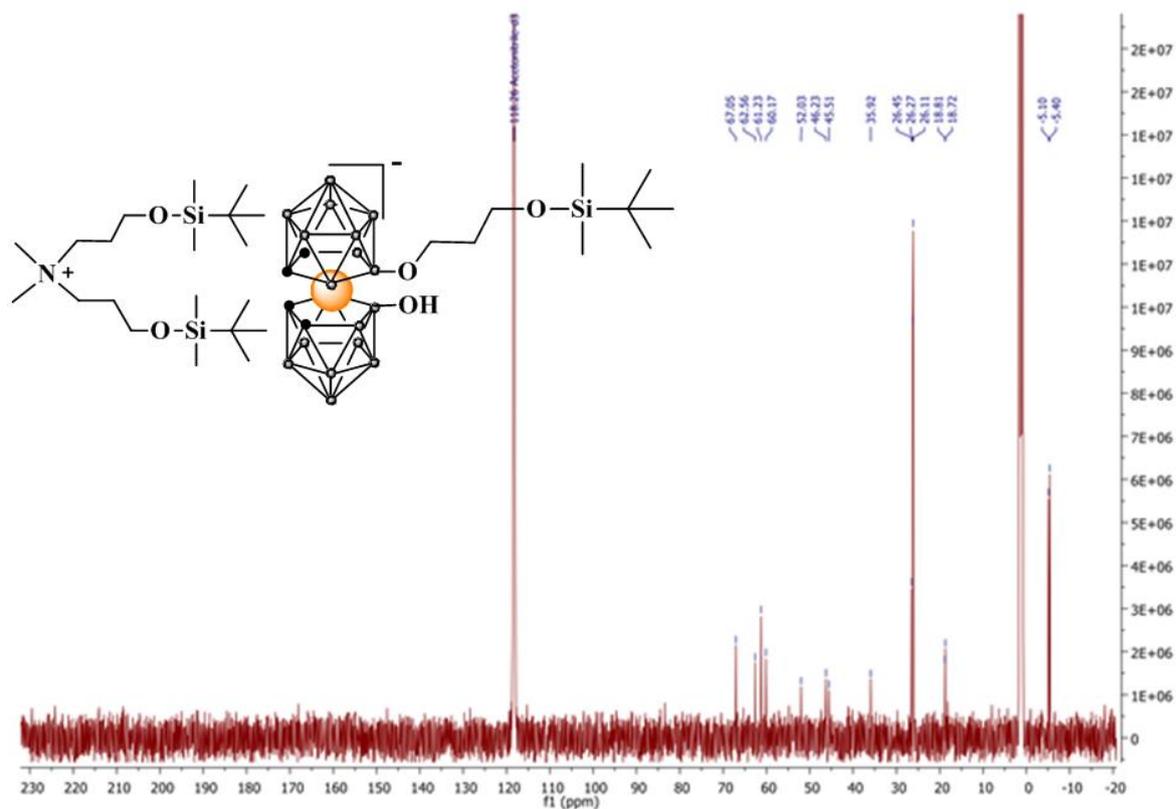


Figure S8. ^{13}C NMR spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS]-1,2-C₂B₉H₁₀(8'-OH-1,2-C₂B₉H₁₀) (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**6**)

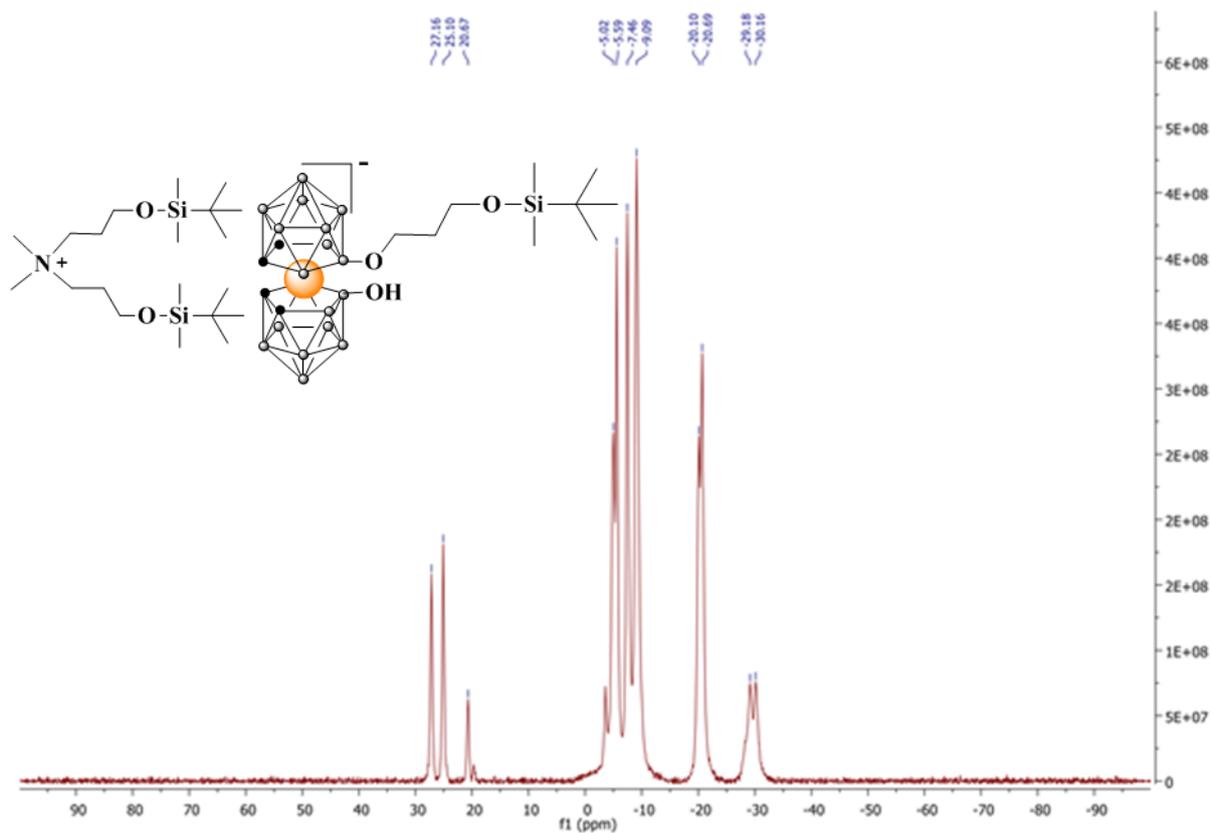


Figure S9. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS]-1,2-C₂B₉H₁₀(8'-OH-1,2-C₂B₉H₁₀) (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**6**)

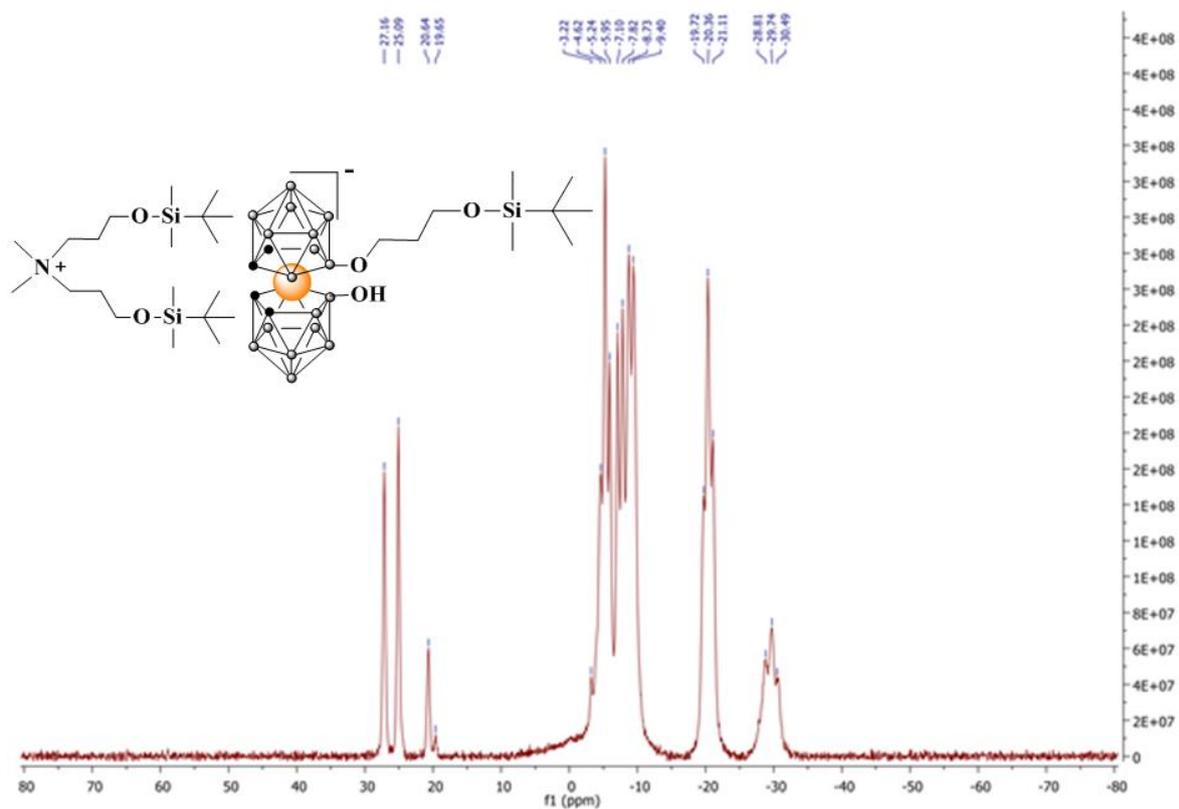


Figure S10. ^{11}B NMR spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS]-1,2-C₂B₉H₁₀(8'-OH-1,2-C₂B₉H₁₀) (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**6**)

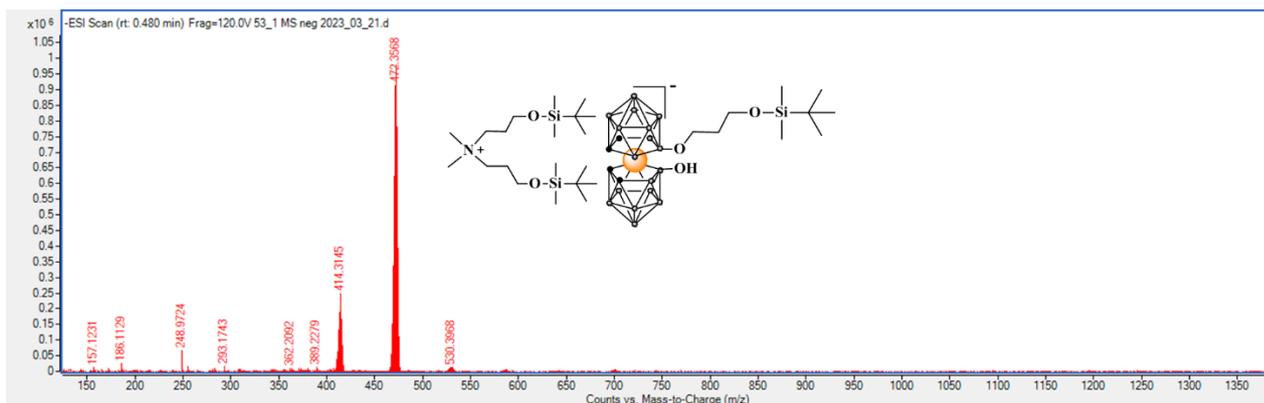


Figure S11. MS(ESI) spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS]-1,2-C₂B₉H₁₀)(8'-OH-1,2-C₂B₉H₁₀) (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**6**)

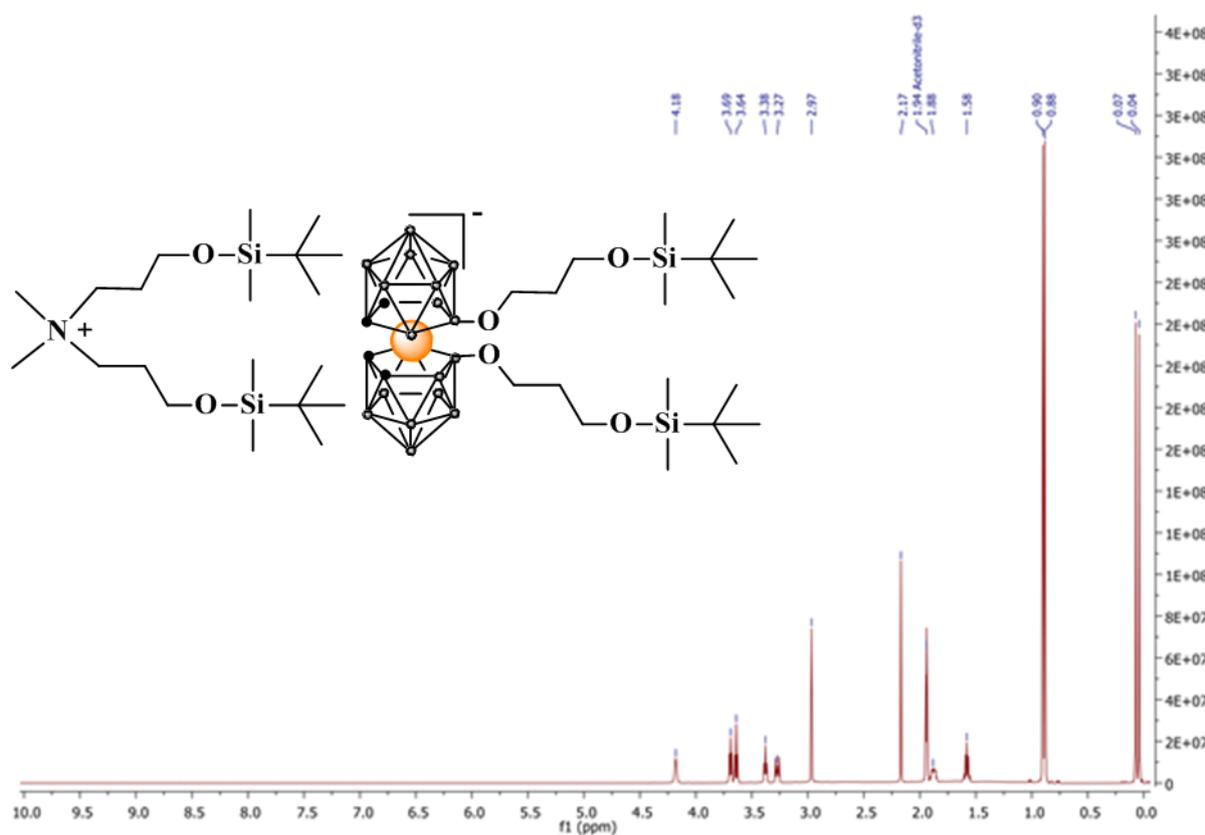


Figure S12. ¹H NMR spectrum 3,3'-Co[8-O(CH₂)₃OTBDMS]-1,2-C₂B₉H₁₀]₂ (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**8**).

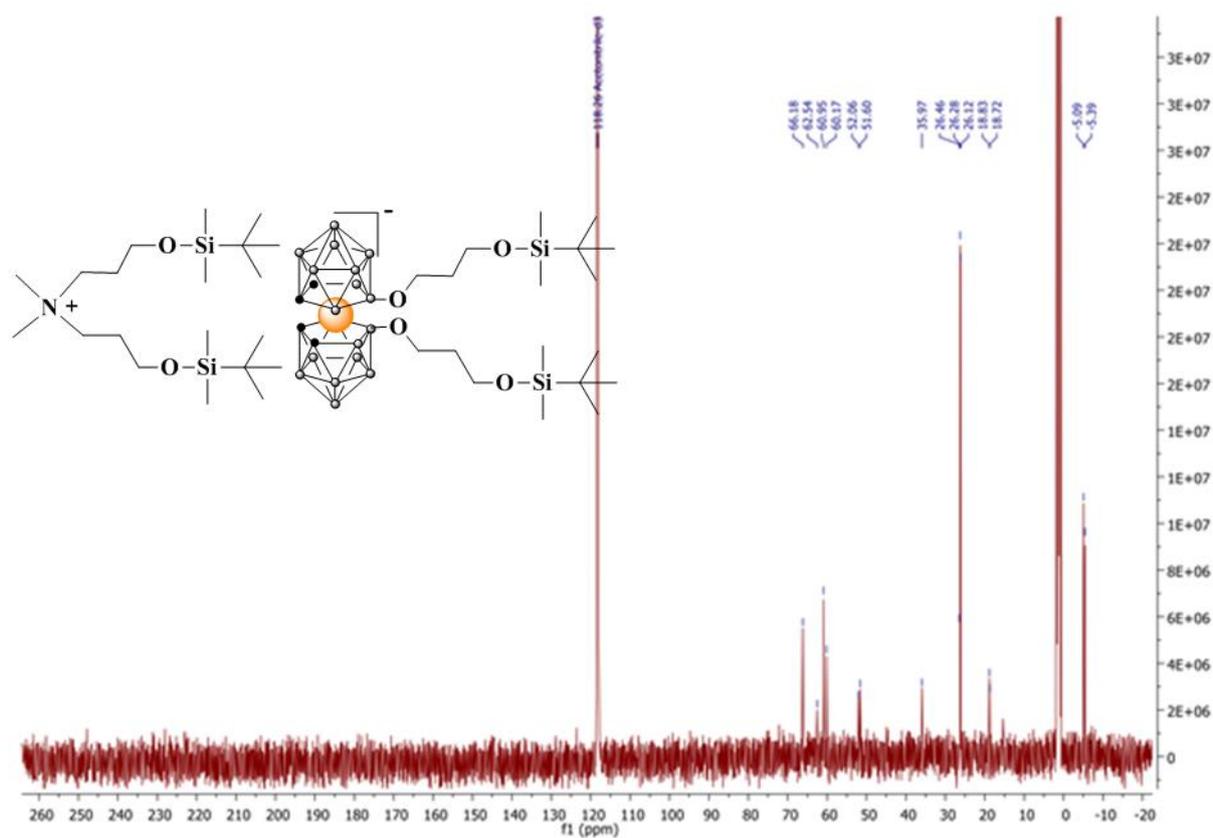


Figure S13. ^{13}C NMR spectrum of $3,3',3''\text{-Co}[8\text{-O}(\text{CH}_2)_3\text{OTBDMS-1,2-C}_2\text{B}_9\text{H}_{10}]_2(\text{CH}_3)_2\text{N}[(\text{CH}_2)_3\text{OTBDMS}]_2$ (8).

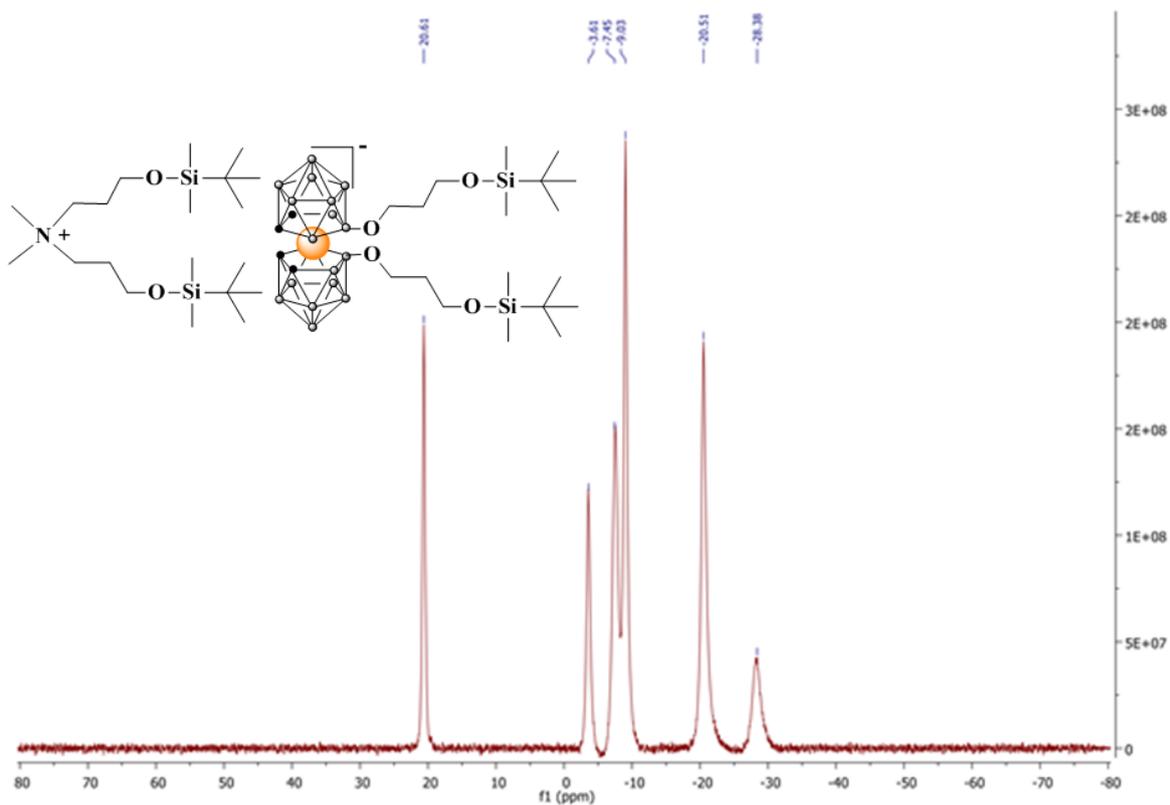


Figure S14. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of $3,3',3''\text{-Co}[8\text{-O}(\text{CH}_2)_3\text{OTBDMS-1,2-C}_2\text{B}_9\text{H}_{10}]_2(\text{CH}_3)_2\text{N}[(\text{CH}_2)_3\text{OTBDMS}]_2$ (8).

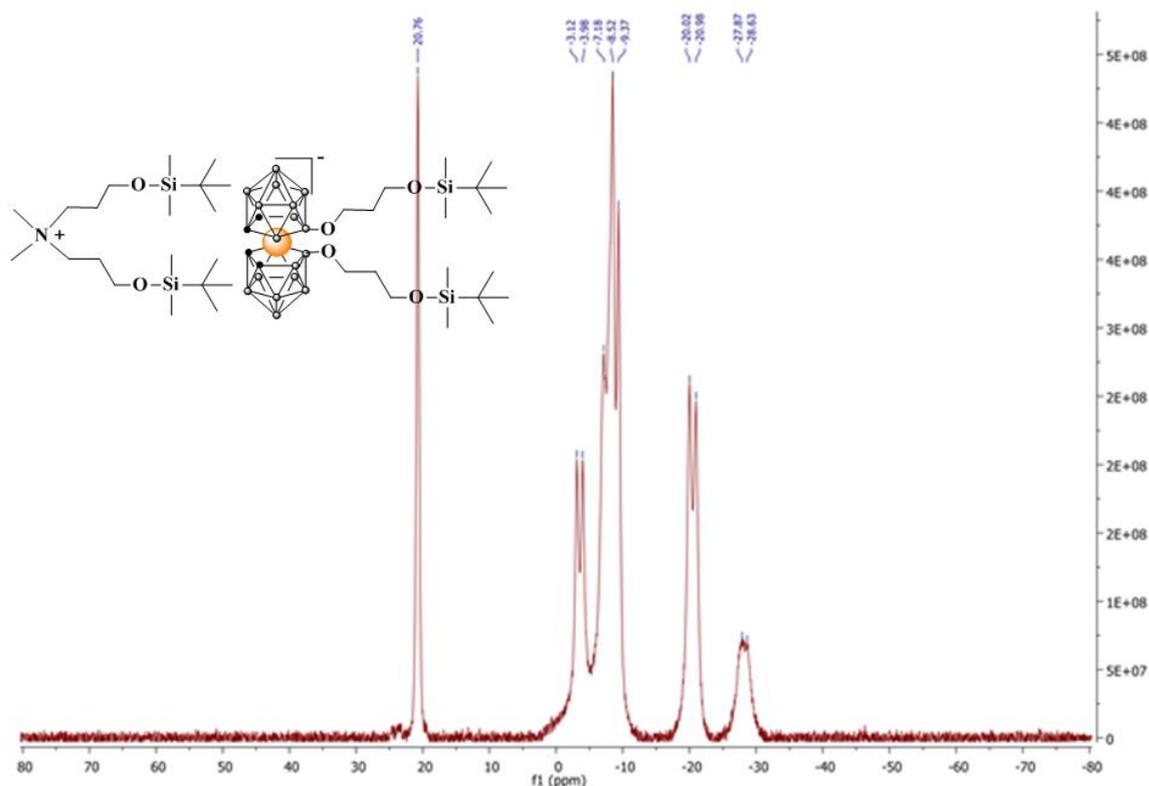


Figure S15. ^{11}B NMR spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS-1,2-C₂B₉H₁₀]₂ (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**8**).

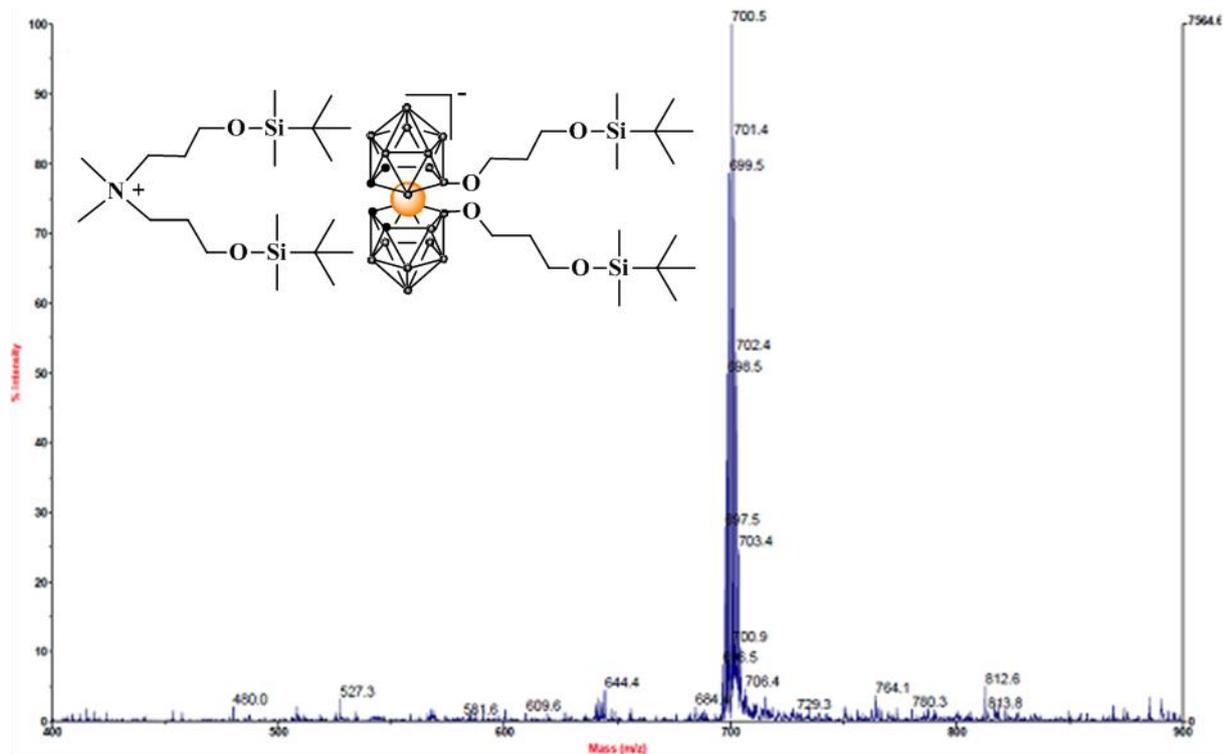
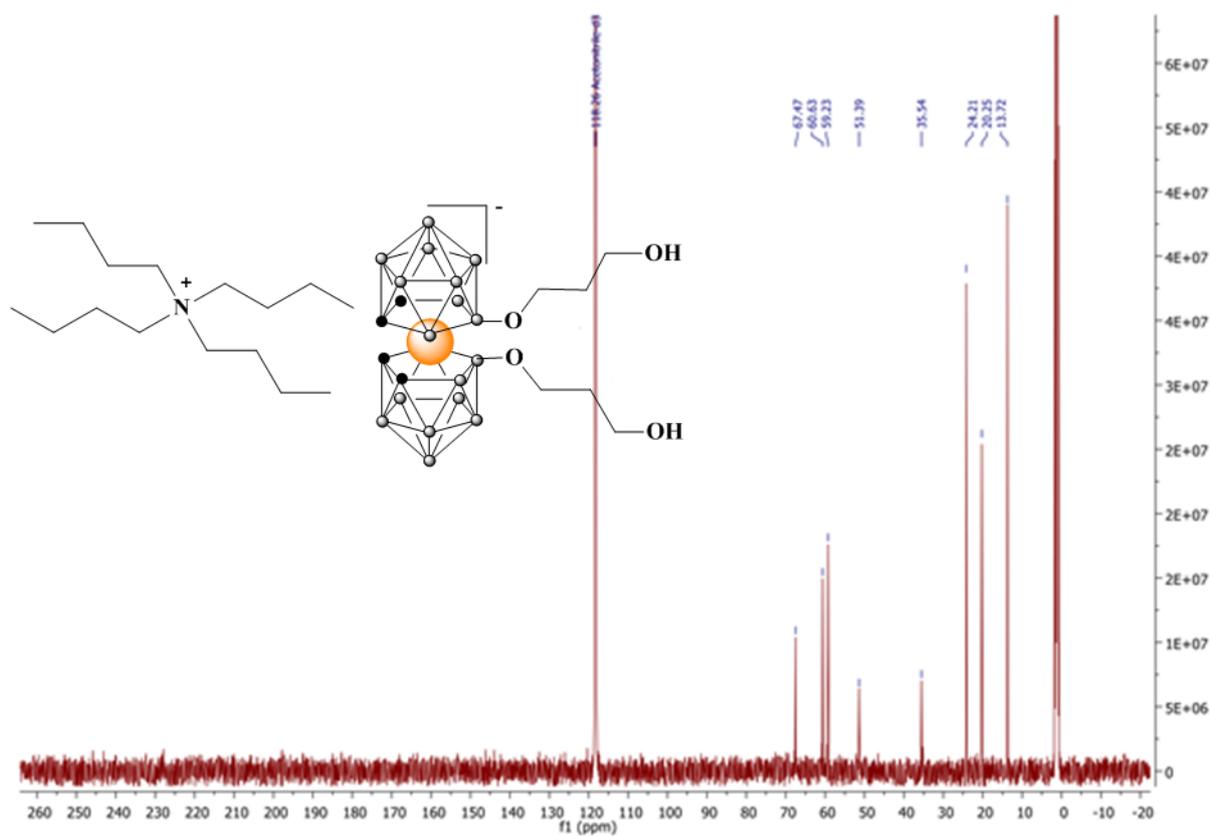
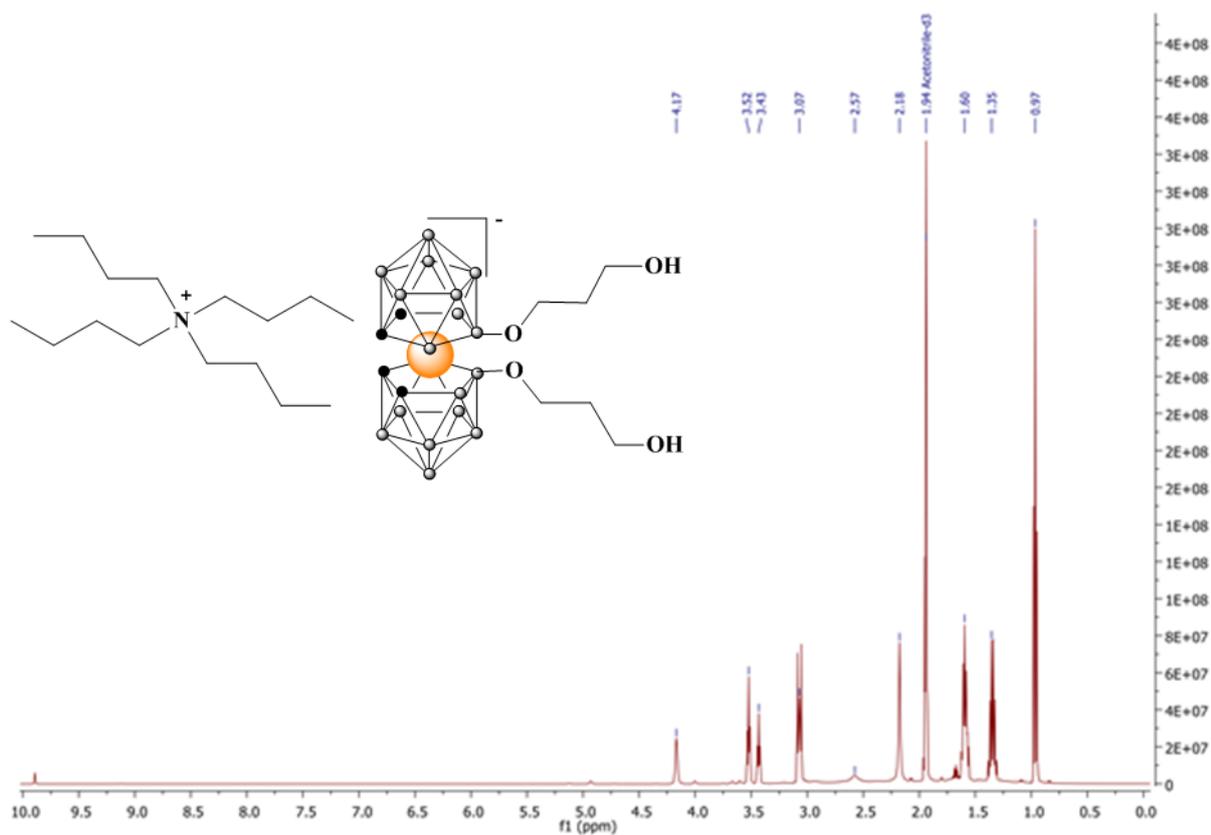


Figure S16. MS(EI) spectrum of 3,3'-Co[8-O(CH₂)₃OTBDMS-1,2-C₂B₉H₁₀]₂ (CH₃)₂N[(CH₂)₃OTBDMS]₂ (**8**).



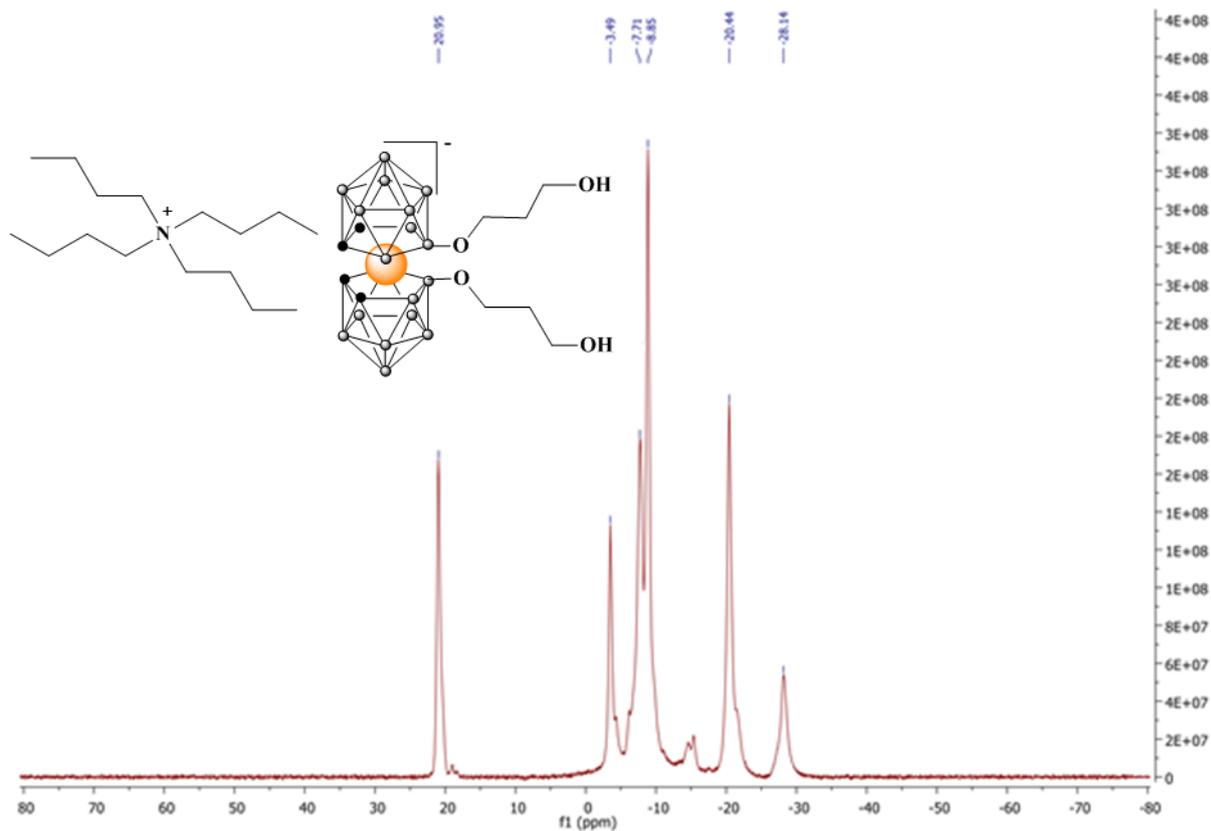


Figure S19. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of $[3,3'\text{-Co}(8\text{-O}(\text{CH}_2)_3\text{OH}\text{-}1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ TBA (9).

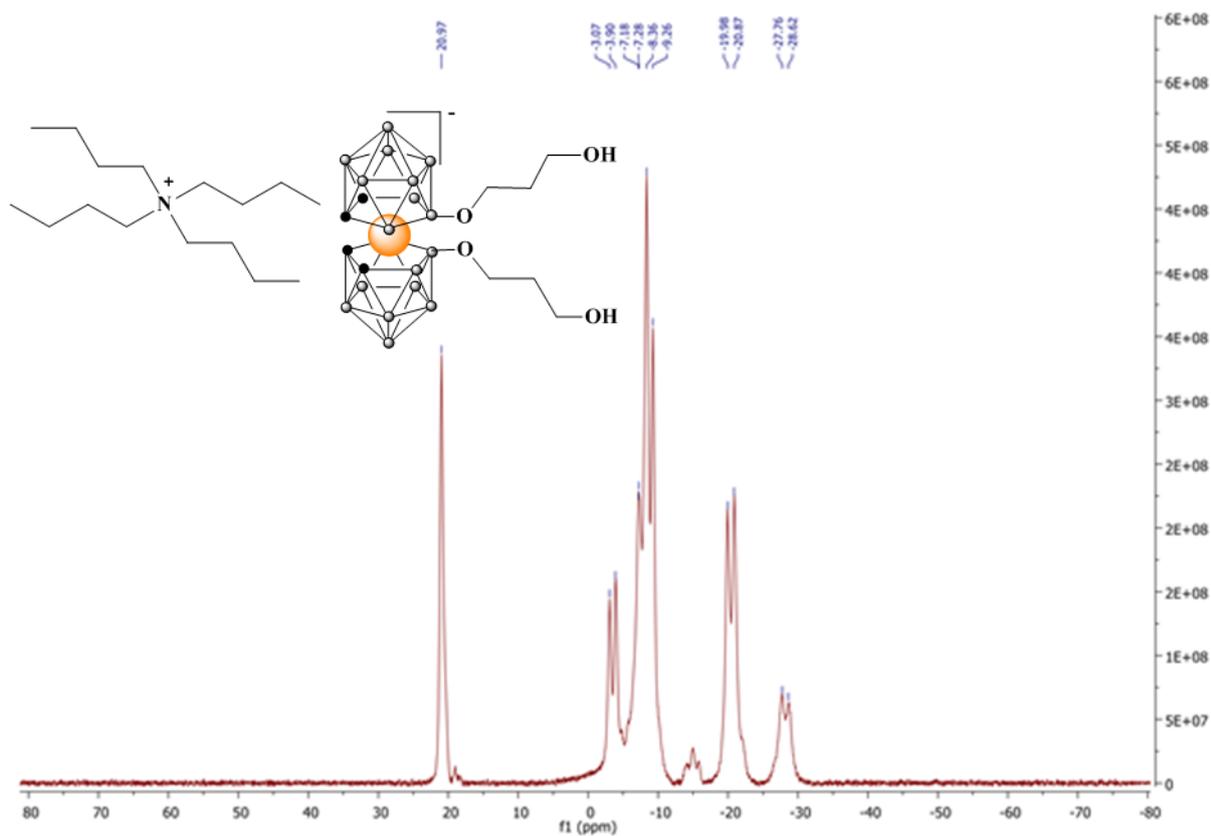


Figure S20. ^{11}B NMR spectrum of $[3,3'\text{-Co}(8\text{-O}(\text{CH}_2)_3\text{OH}\text{-}1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ TBA (9).

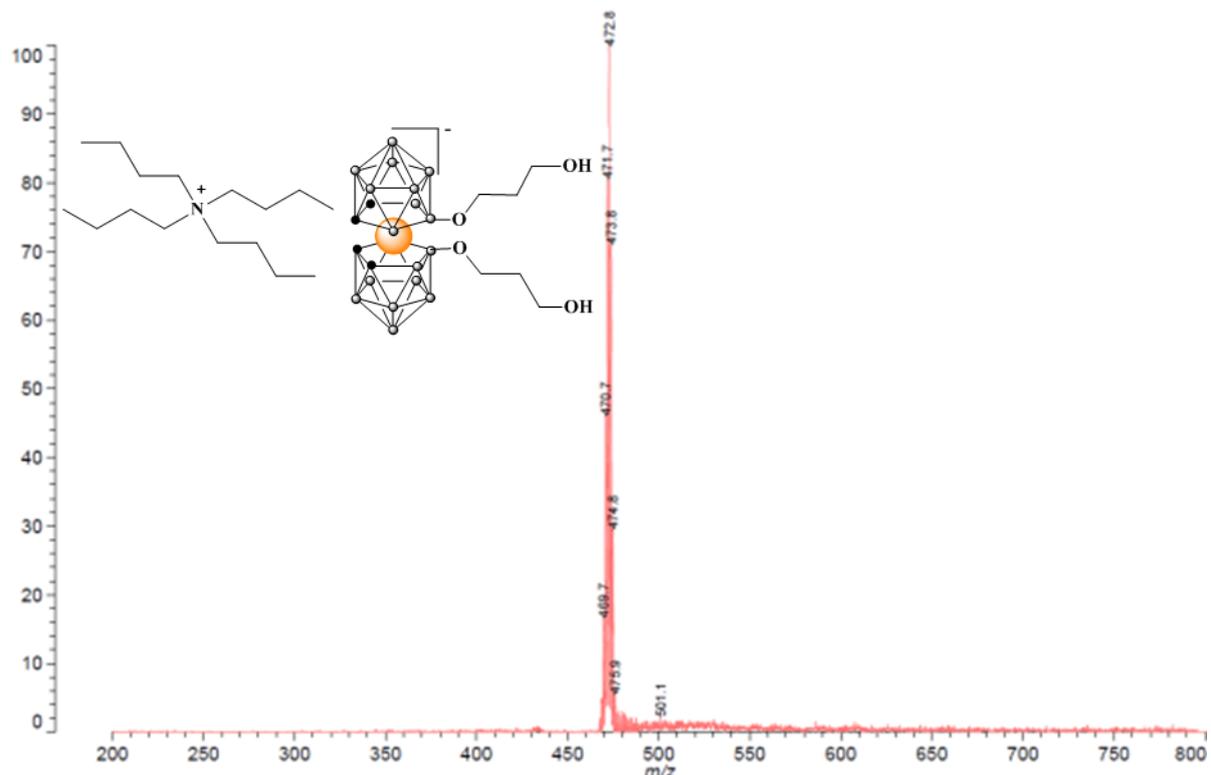


Figure S21. MS(ESI) spectrum of $[3,3'\text{-Co}(8\text{-O}(\text{CH}_2)_3\text{OH}\text{-}1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ TBA (**9**).

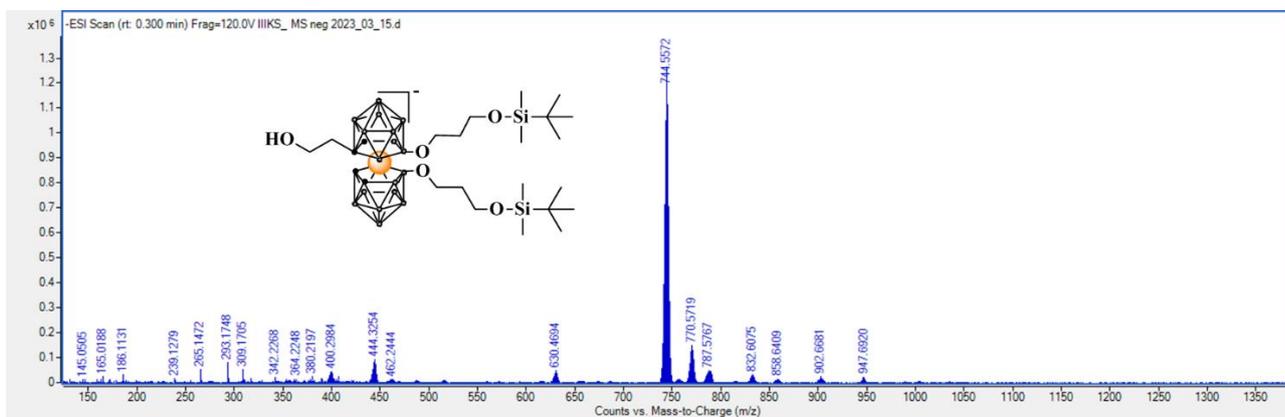


Figure S22. MS (ESI) spectrum of $3,3'\text{-Co}\{[8\text{-O}(\text{CH}_2)_3\text{OTBDMS}\text{-}1\text{-}(\text{CH}_2)_2\text{OH}]\text{-}1,2\text{-C}_2\text{B}_9\text{H}_9\}\{[8'\text{-O}(\text{CH}_2)_3\text{OTBDMS}\text{-}1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\}^-$ (**10**)

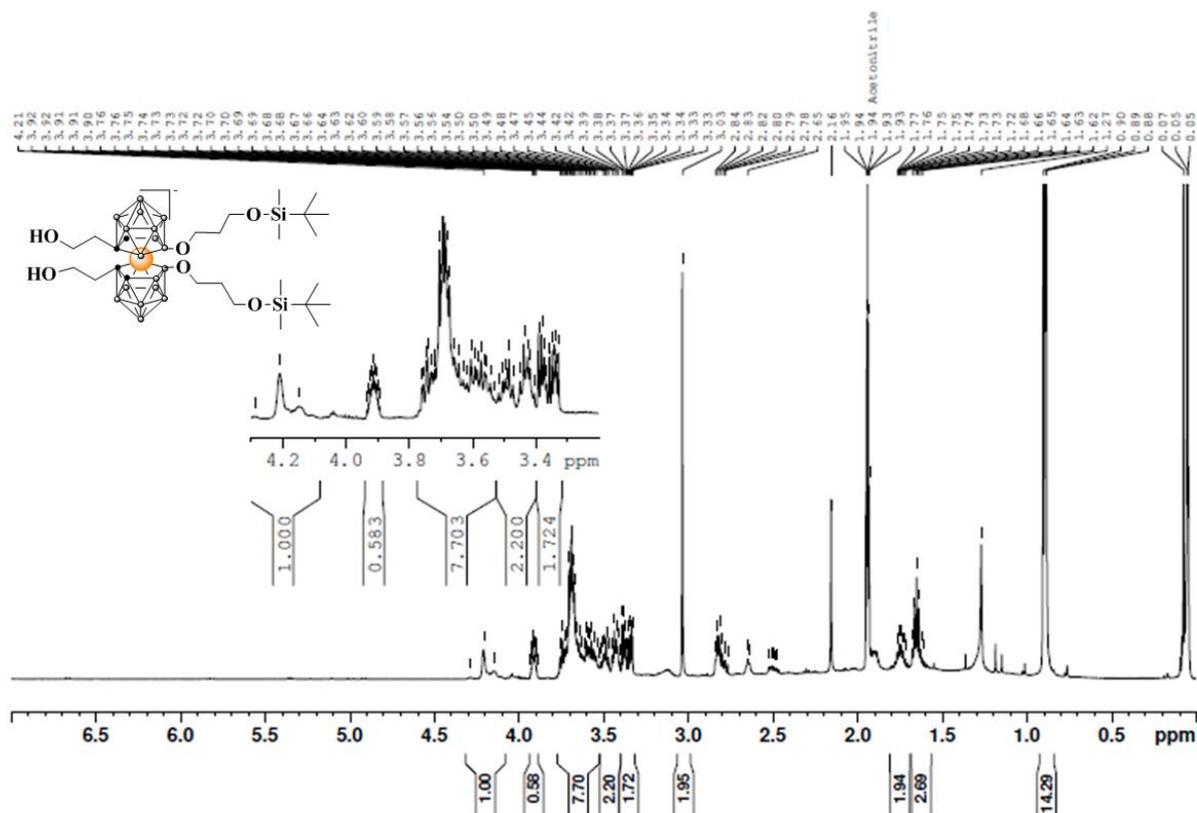


Figure S23. ¹H NMR spectrum of $3,3'$ -Co[(8-O(CH₂)₃OTBDMS-1-(CH₂)₂OH-1,2-C₂B₉H₉)]₂⁻ (**11**)

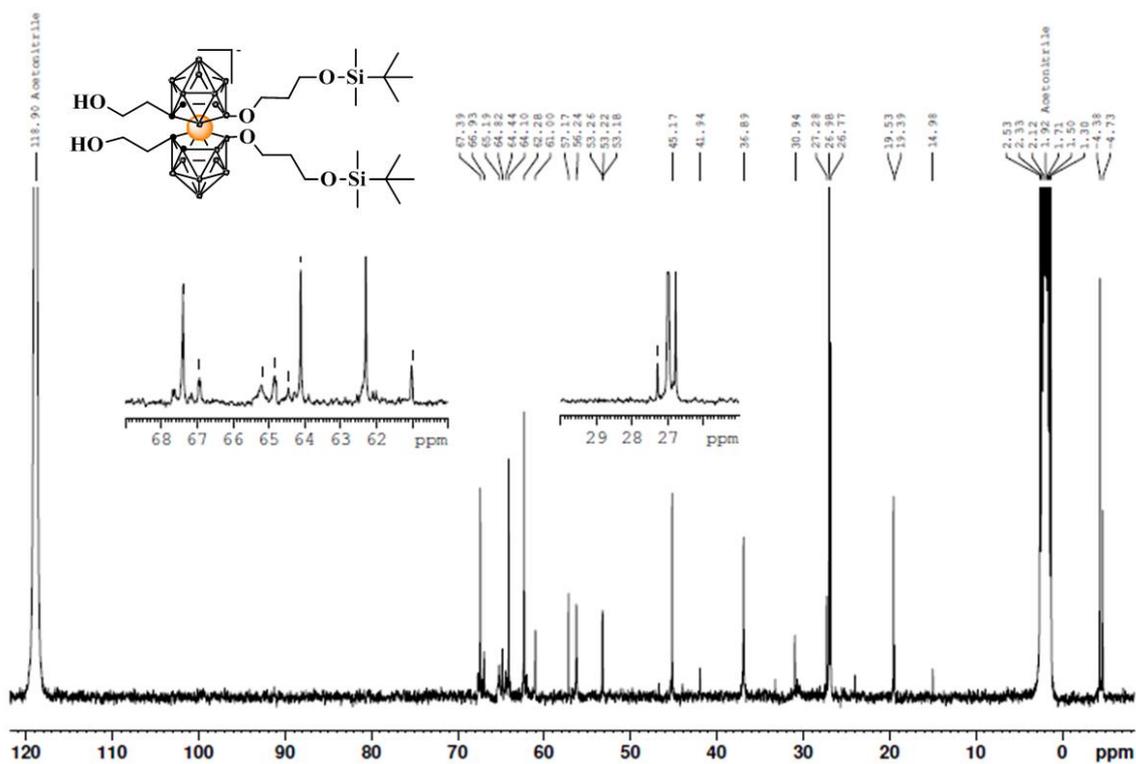
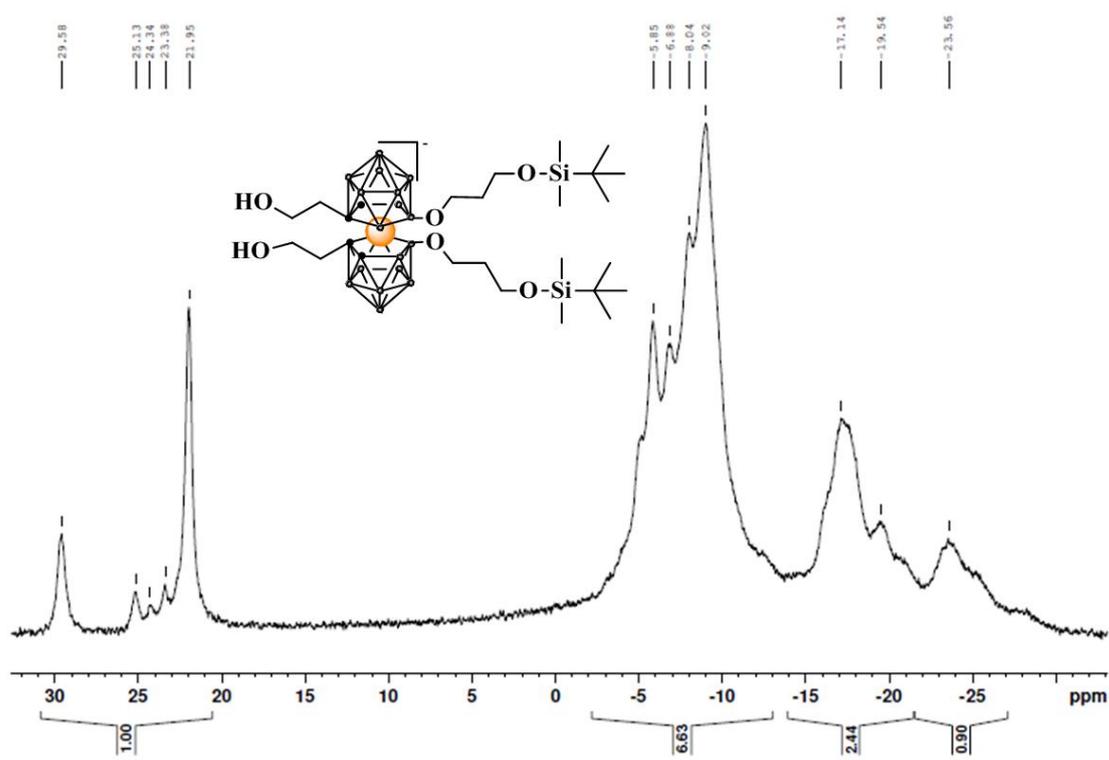
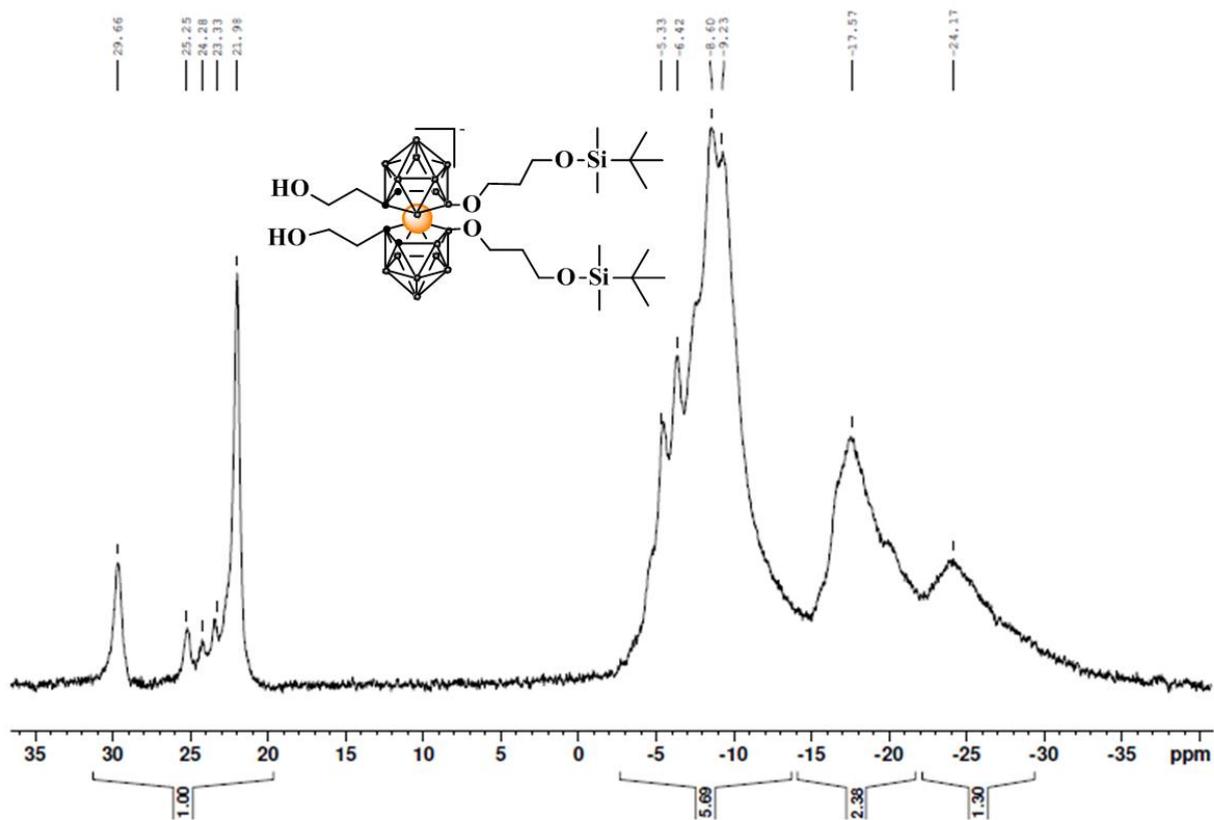


Figure S24. ¹³C NMR spectrum of $3,3'$ -Co[(8-O(CH₂)₃OTBDMS-1-(CH₂)₂OH-1,2-C₂B₉H₉)]₂⁻ (**11**)



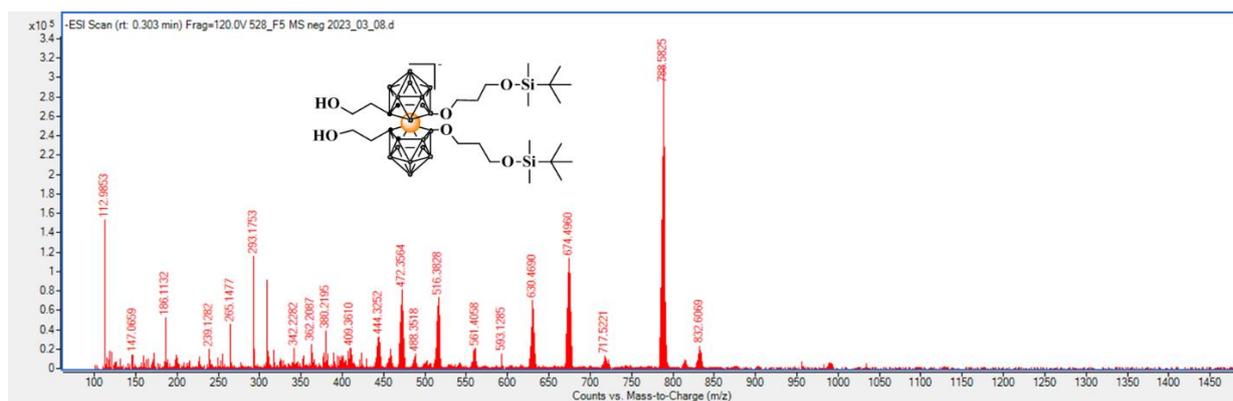


Figure S27. MS (ESI) spectrum of 3,3'-Co[(8-O(CH₂)₃OTBDMS-1-(CH₂)₂OH-1,2-C₂B₉H₉)]₂⁻ (**11**)

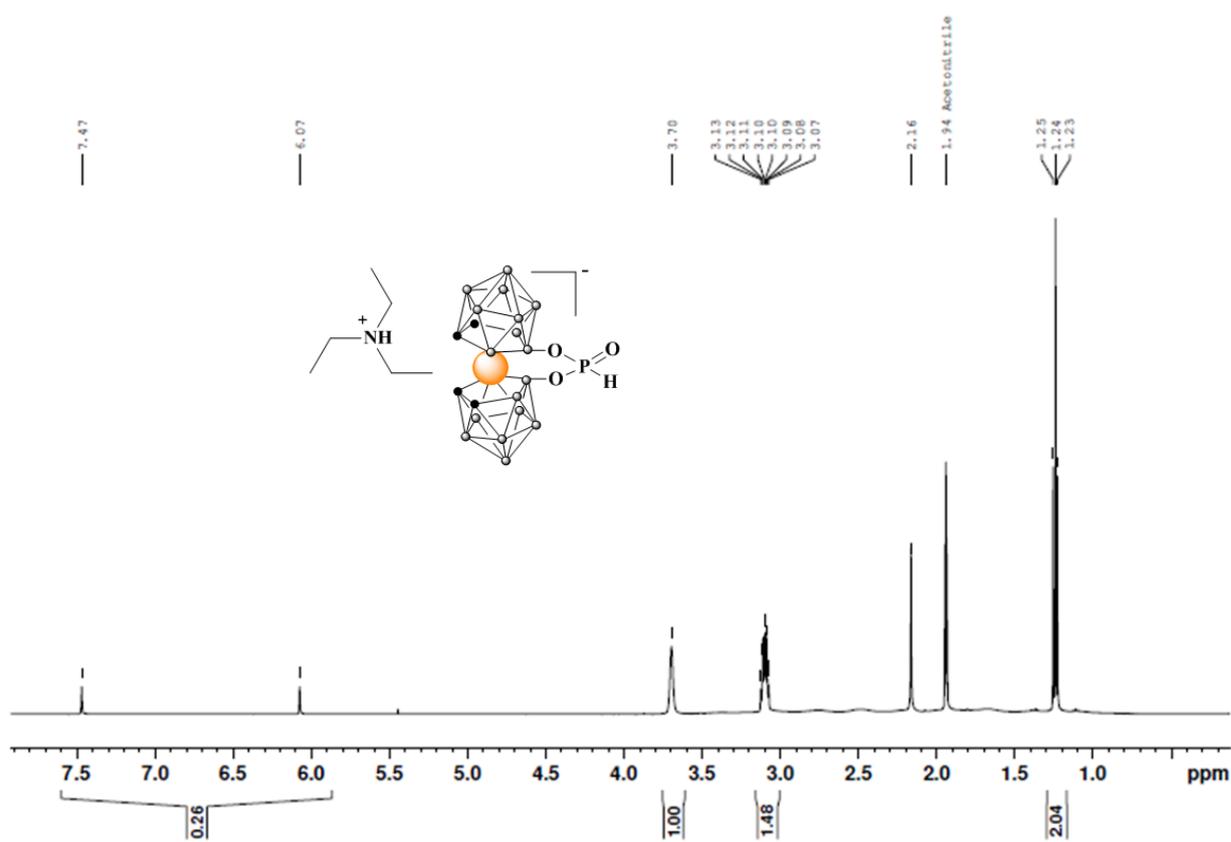


Figure S28. ¹H NMR spectrum of 8,8'-bridged [8,8'-O₂P(O)H-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ H-phosphonate (**12**)

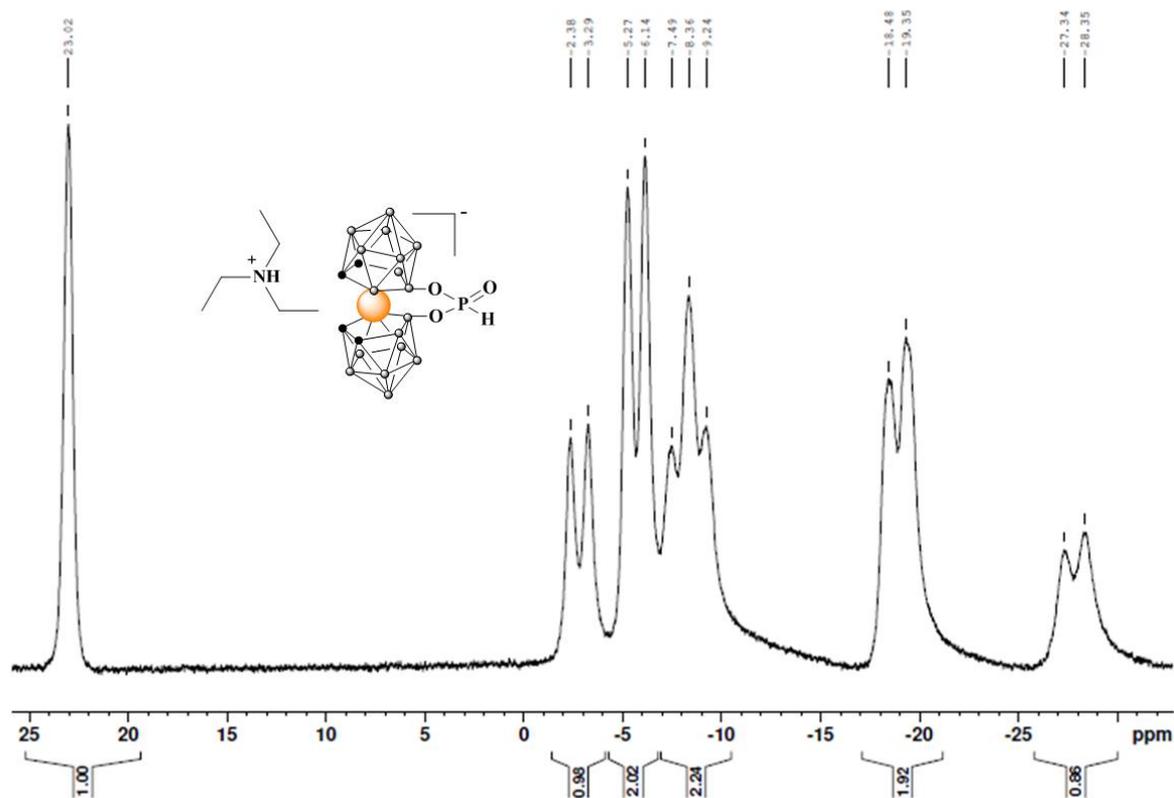


Figure S31. ^{11}B NMR spectrum of 8,8'-bridged $[8,8'\text{-O}_2\text{P(O)H-3,3}'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 H-phosphonate (**12**)

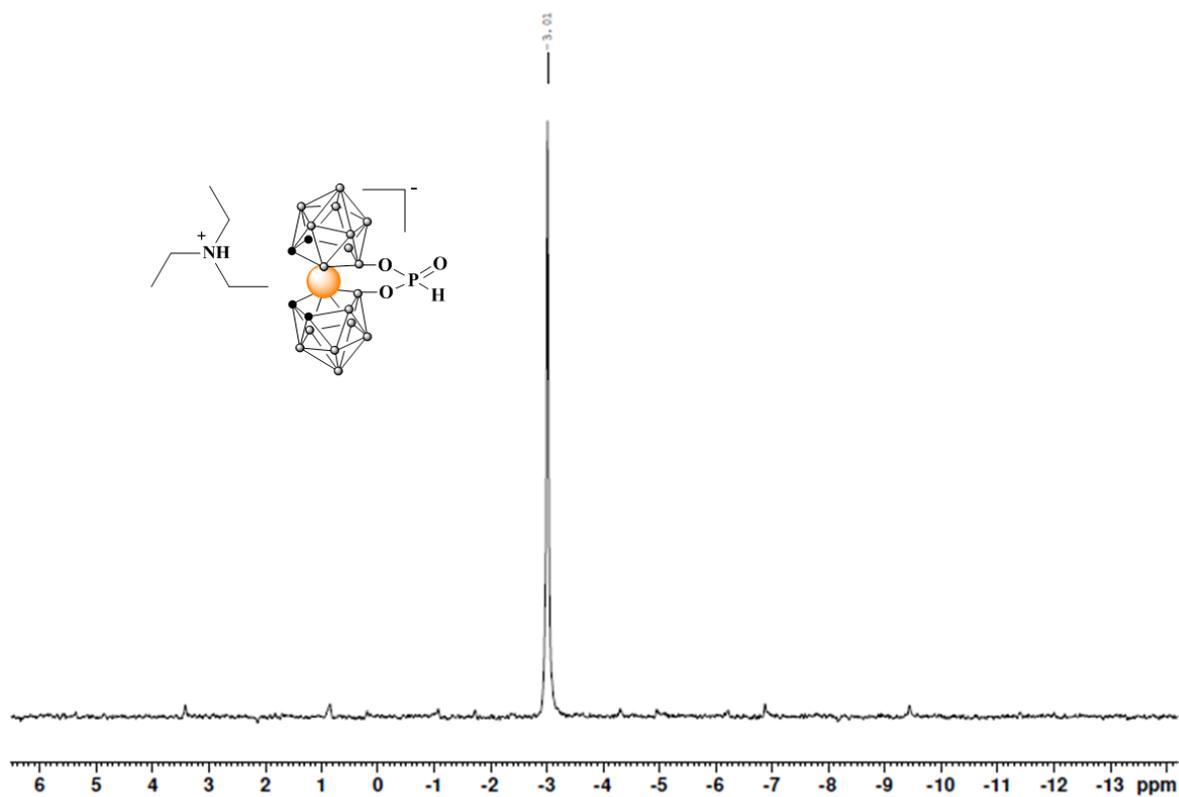


Figure S32. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of 8,8'-bridged $[8,8'\text{-O}_2\text{P(O)H-3,3}'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 H-phosphonate (**12**)

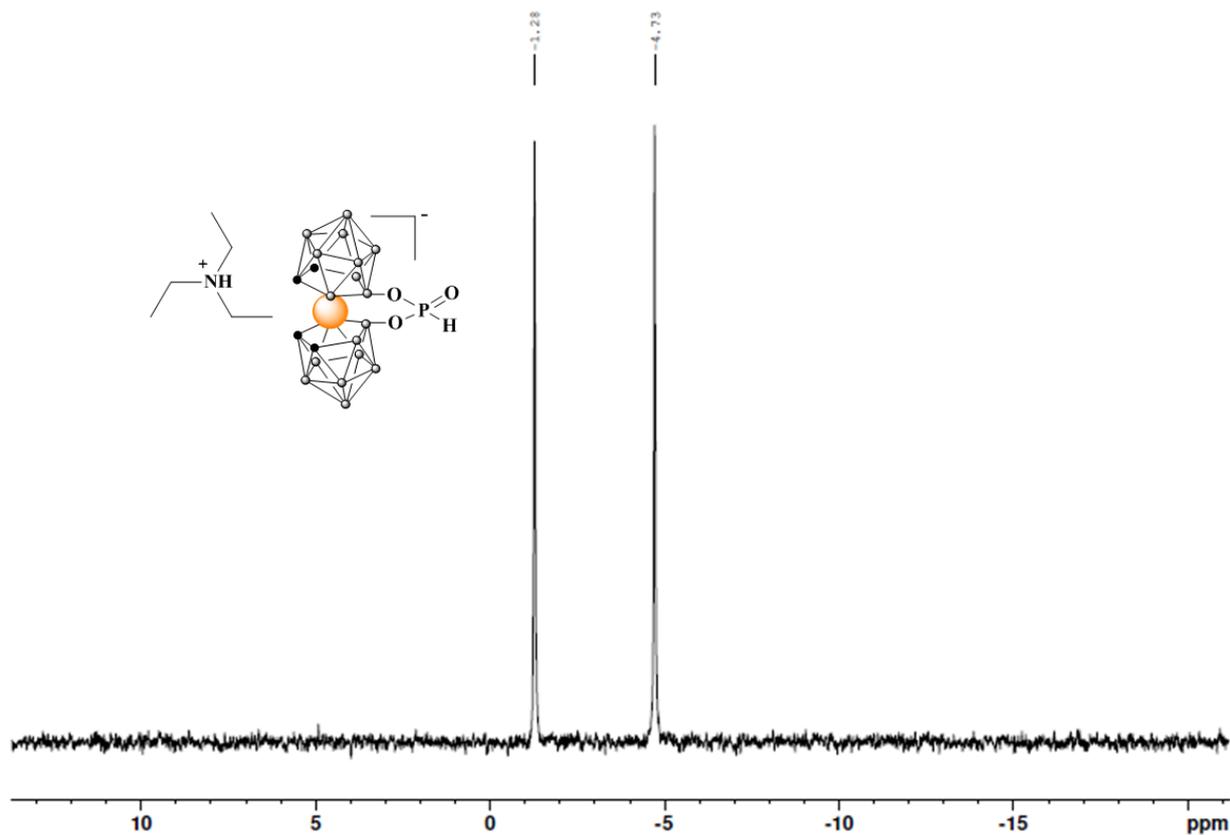


Figure S33. ^{31}P NMR spectrum of 8,8'-bridged $[8,8'\text{-O}_2\text{P}(\text{O})\text{H}\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 H-phosphonate (**12**).

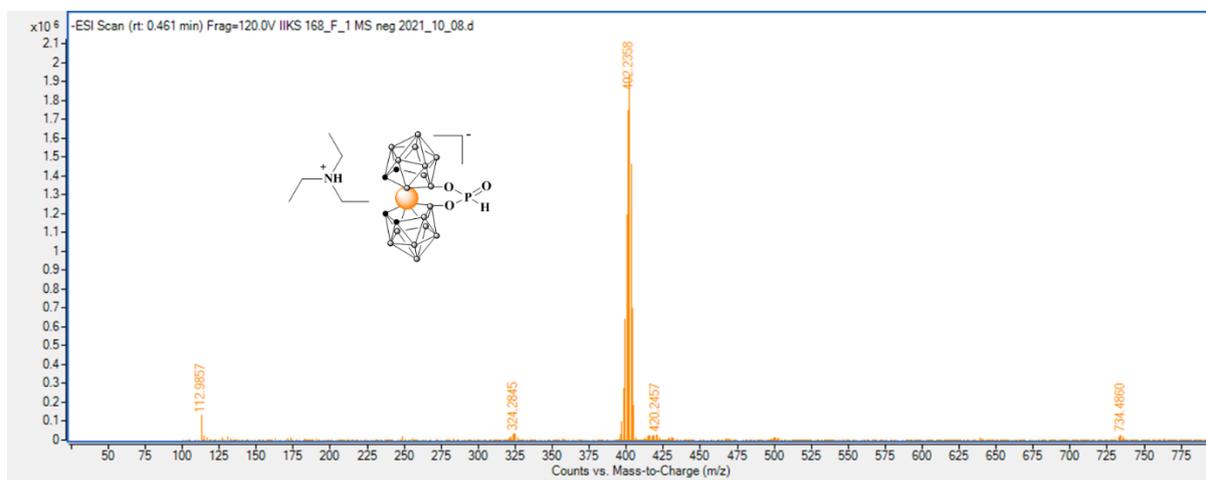


Figure S34. MS (ESI) spectrum of 8,8'-bridged $[8,8'\text{-O}_2\text{P}(\text{O})\text{H}\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 H-phosphonate (**12**).

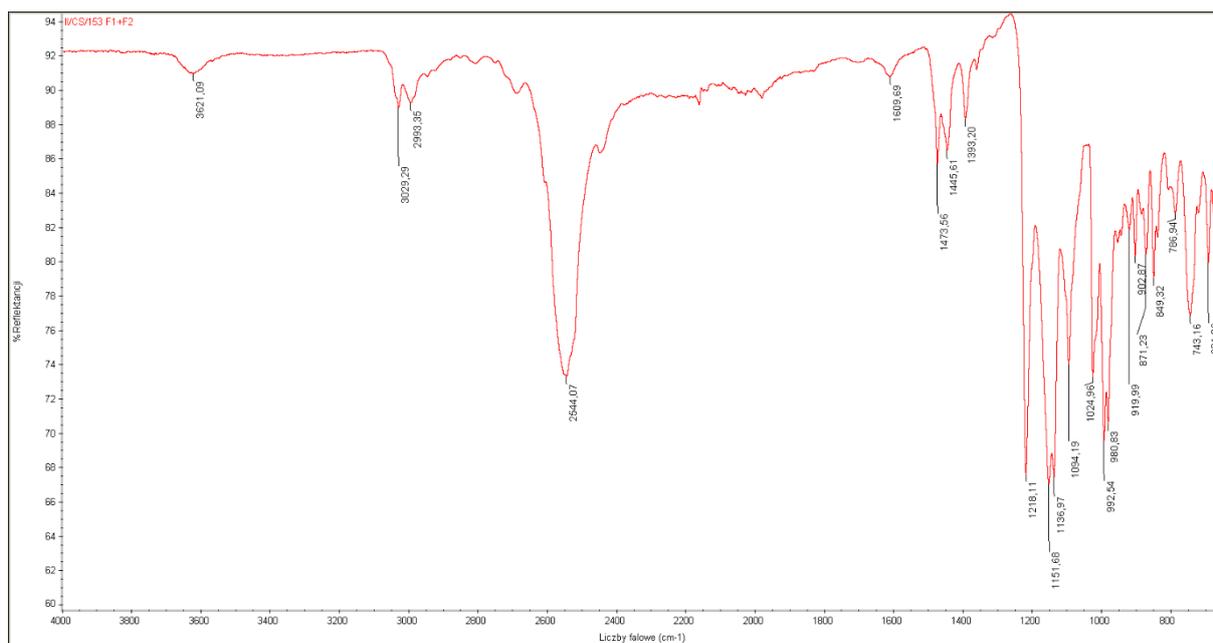


Figure S35. FT-IR spectrum of 8,8'-bridged [8,8'-O₂P(O)H-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNet₃ H-phosphonate (**12**)

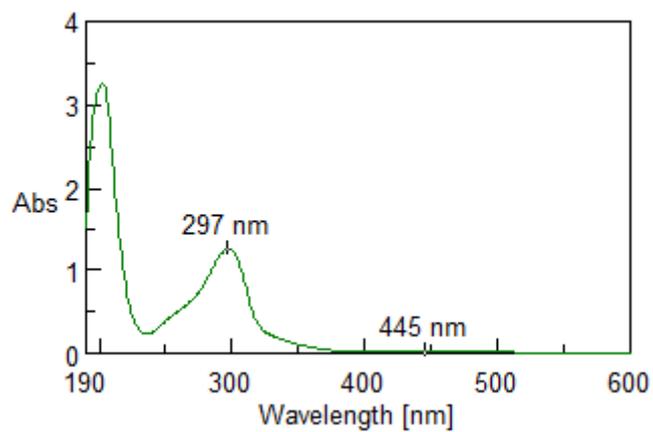


Figure S36. UV-VIS spectrum of 8,8'-bridged [8,8'-O₂P(O)H-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNet₃ H-phosphonate (**12**)

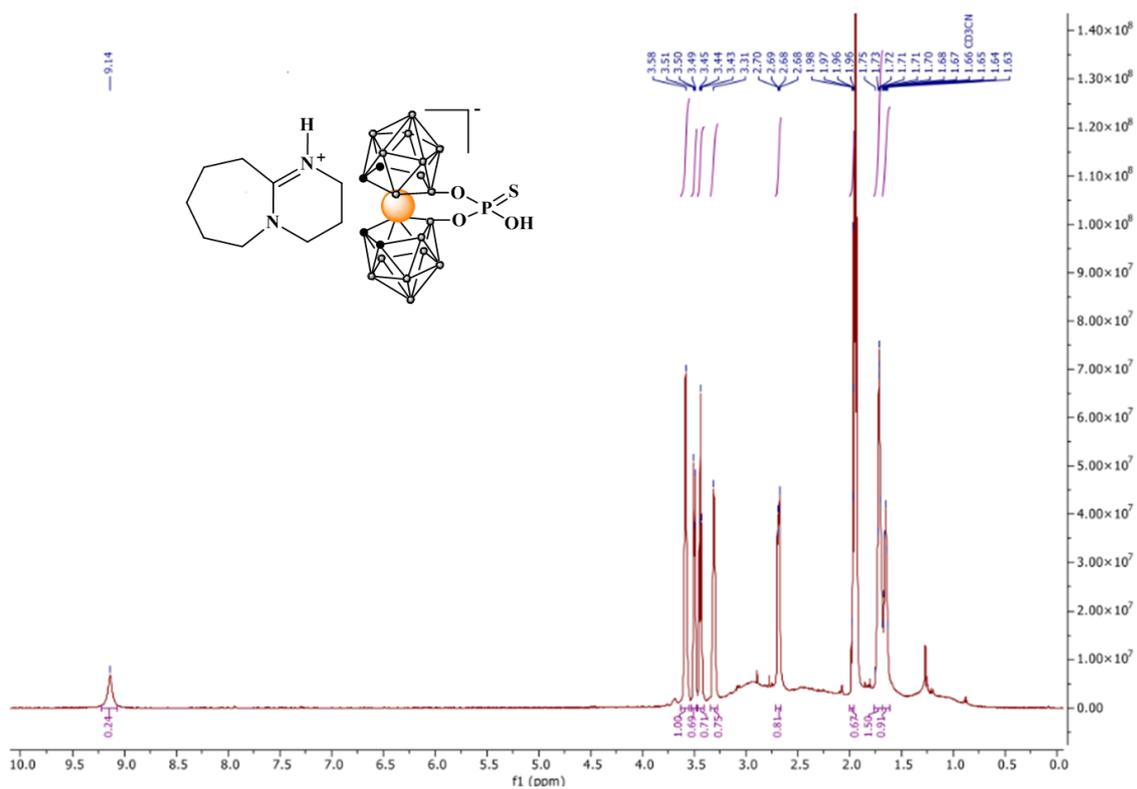


Figure S37. ^1H NMR spectrum of 8,8'-bridged $[8,8'\text{-O}_2\text{P(O)SH-3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HDBU phosphorothioate (**13**).

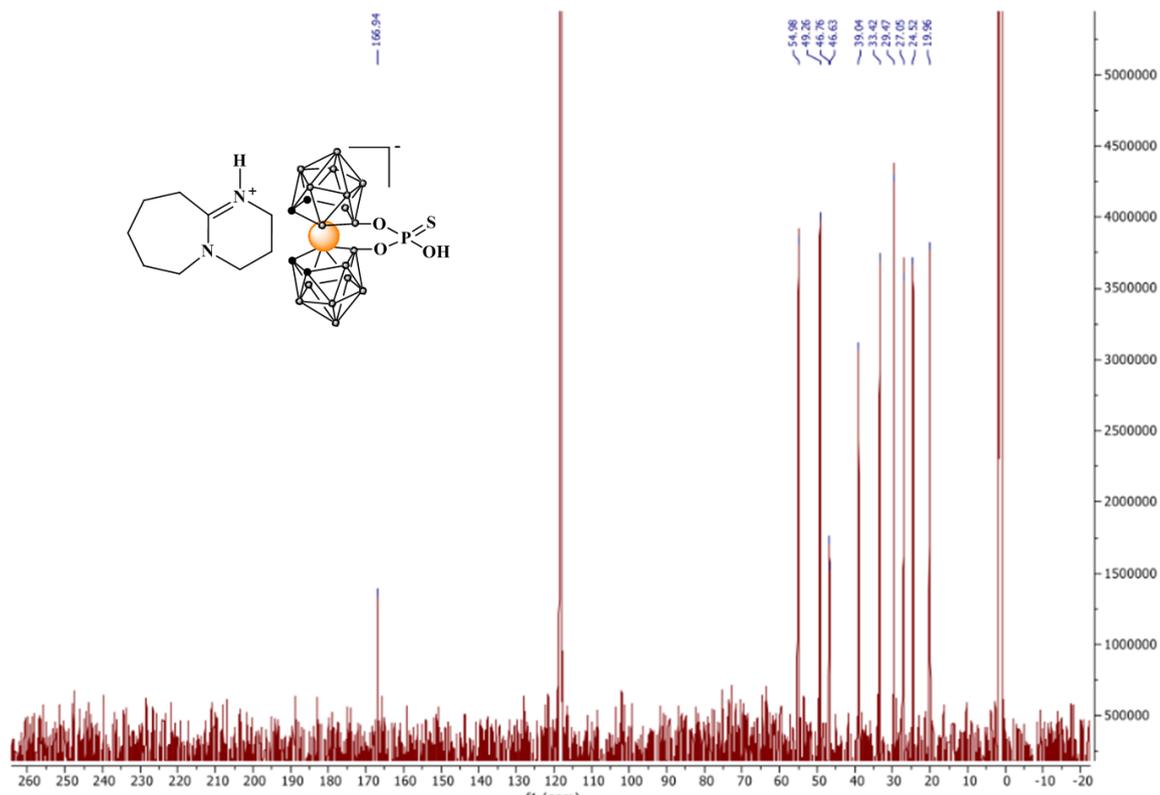


Figure S38. ^{13}C NMR spectrum of 8,8'-bridged $[8,8'\text{-O}_2\text{P(O)SH-3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HDBU phosphorothioate (**13**).

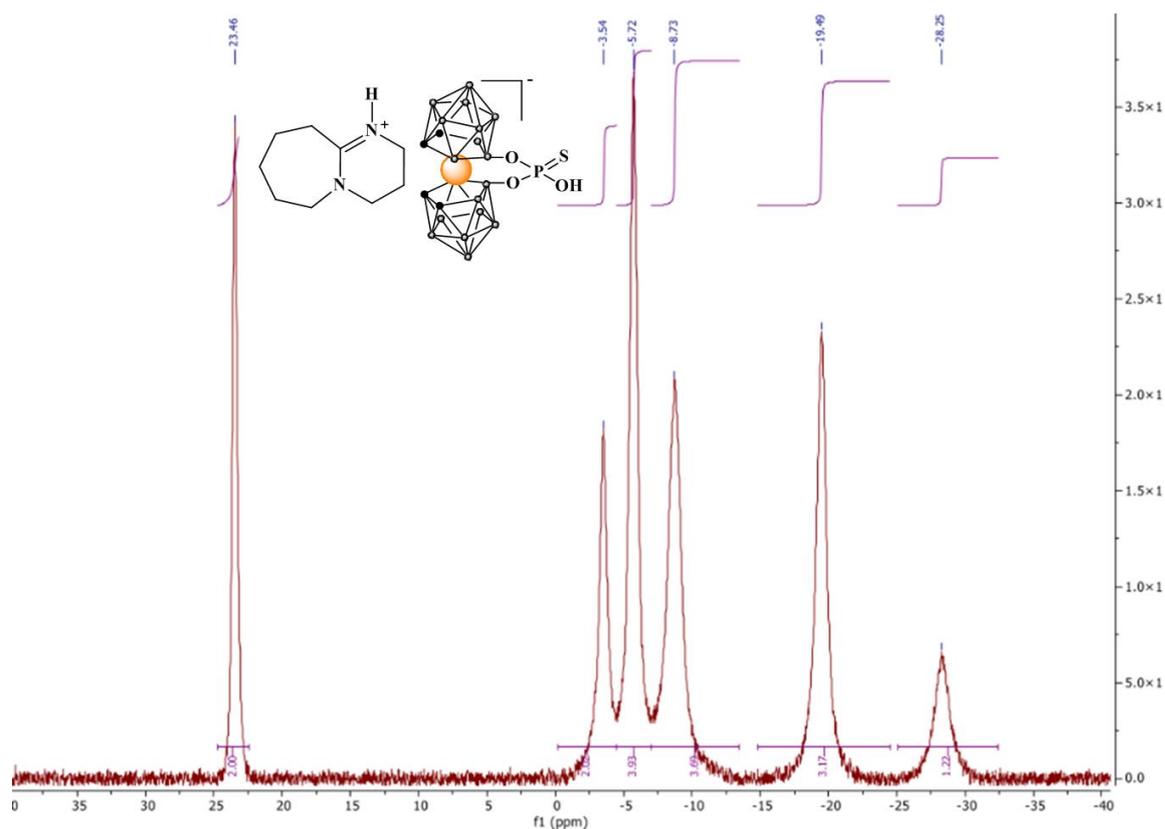


Figure S39. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

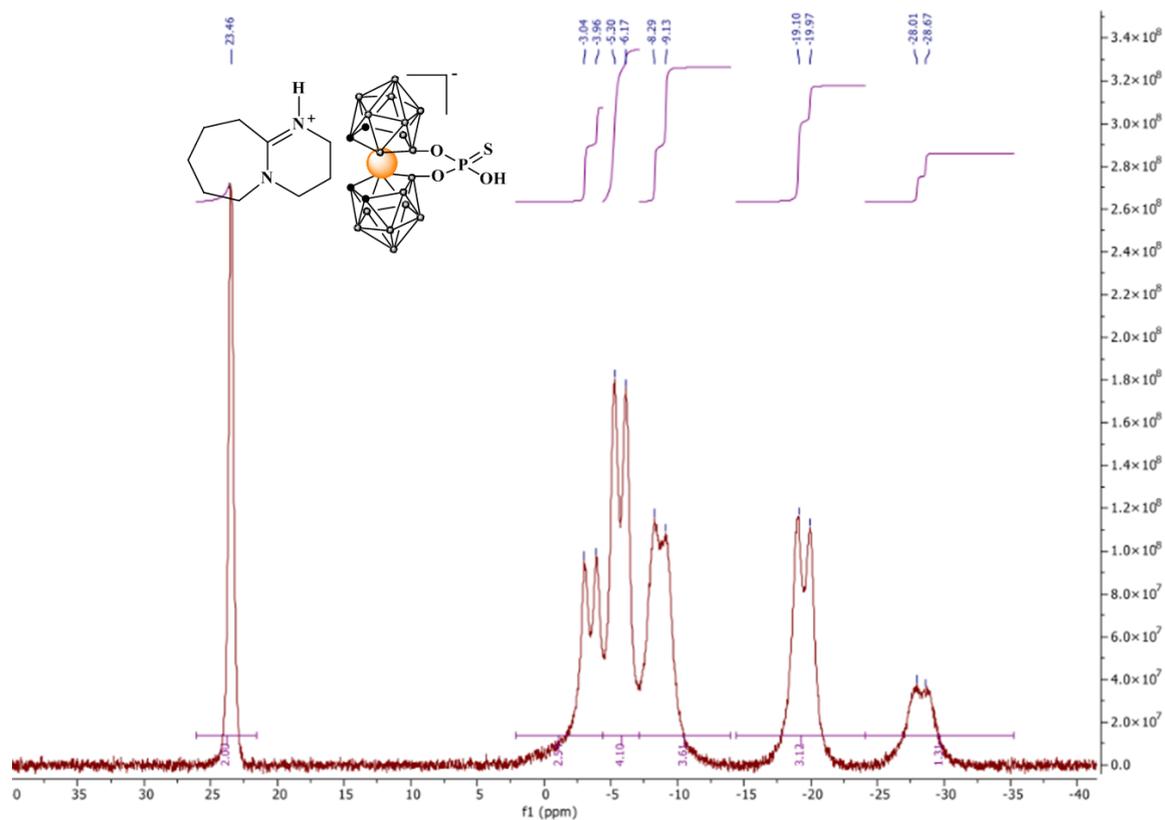


Figure S40. ^{11}B NMR spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

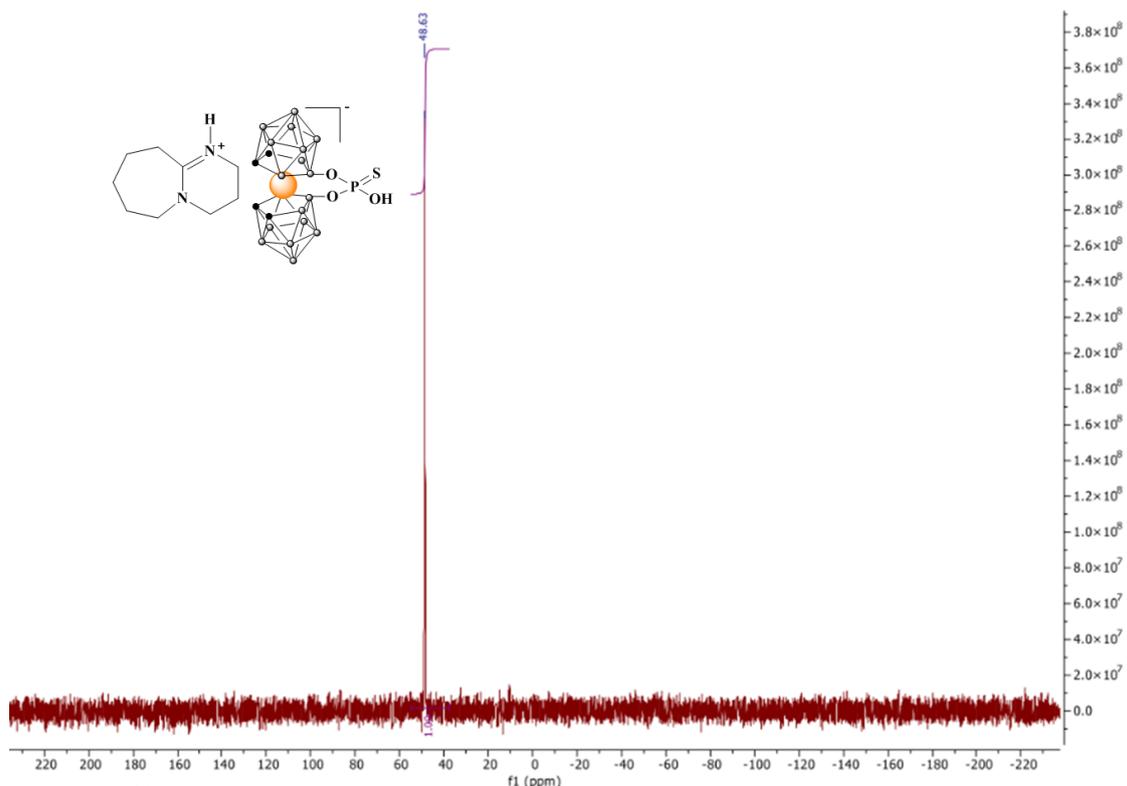


Figure S41. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

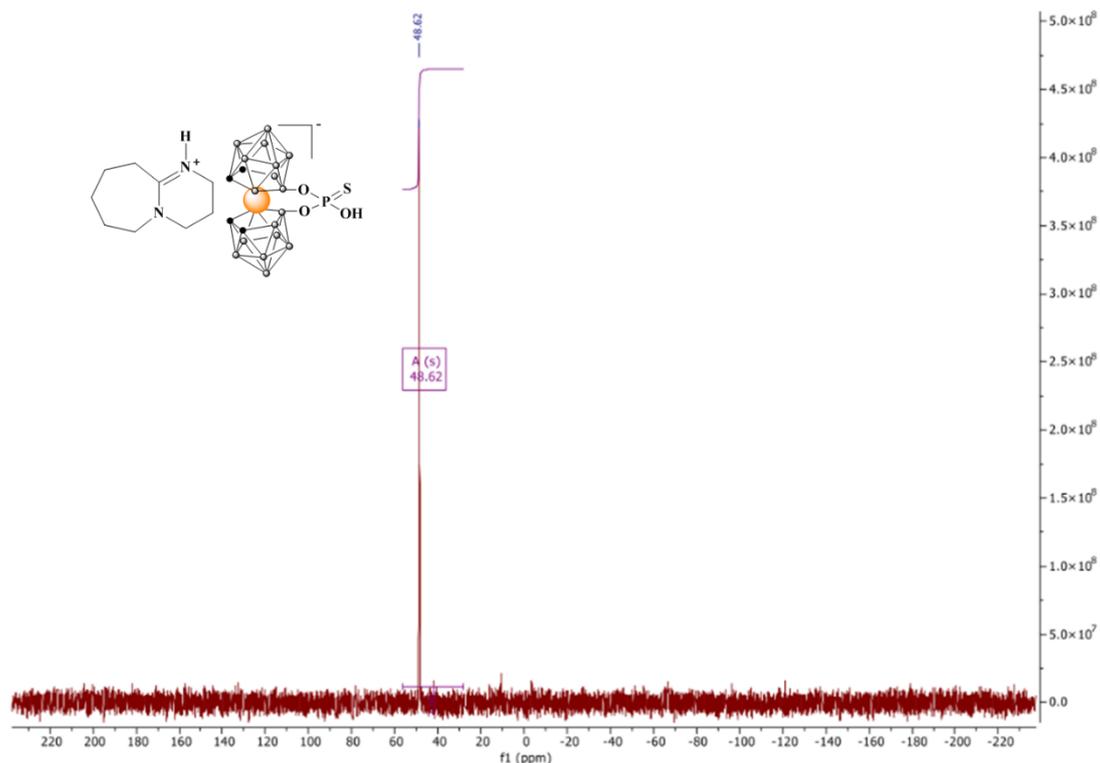


Figure S42. ^{31}P NMR spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

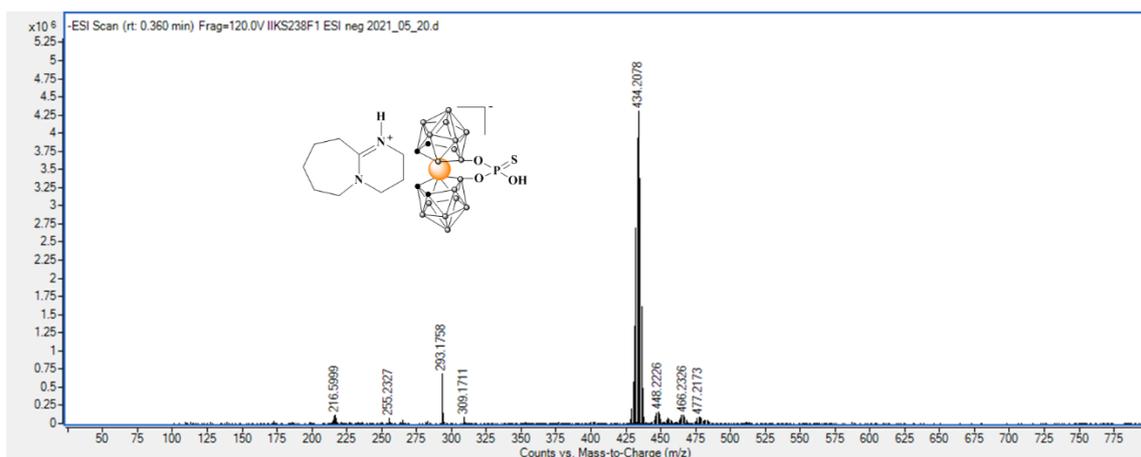


Figure S43. MS (ESI) spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

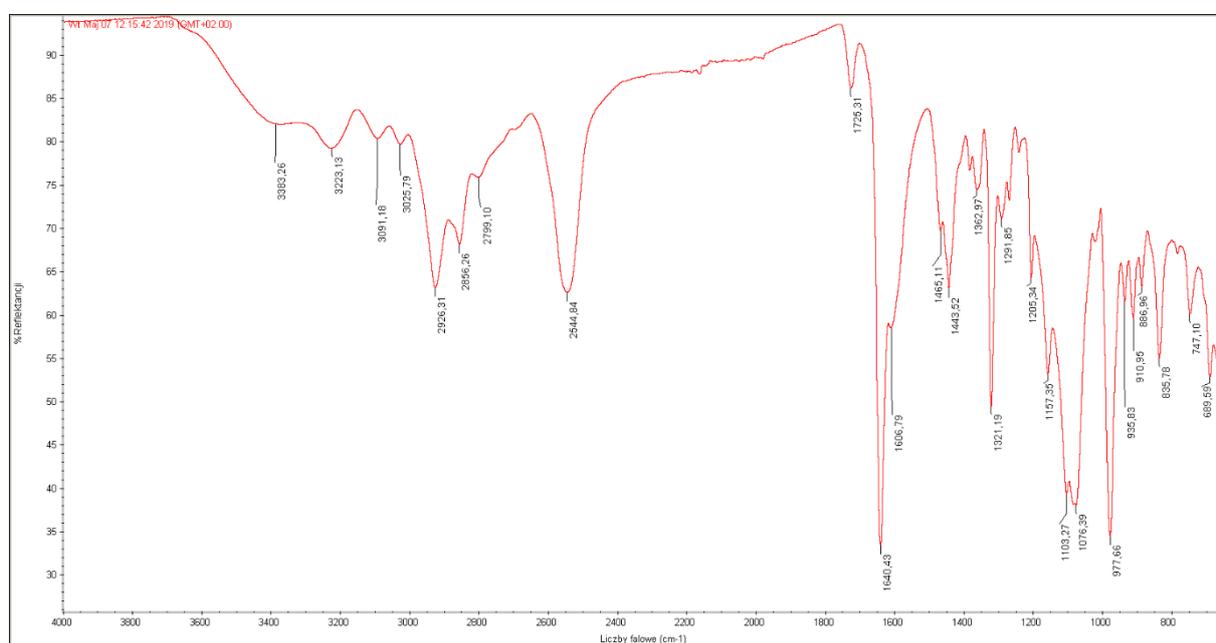


Figure S44. FT-IR spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

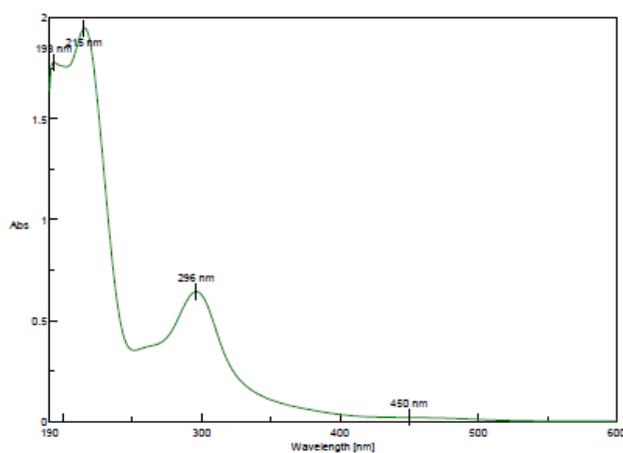


Figure S45. UV-VIS spectrum of spectrum of 8,8'-bridged [8,8'-O₂P(O)SH-3,3'-Co(1,2-C₂B₉H₁₀)₂] HDBU phosphorothioate (**13**).

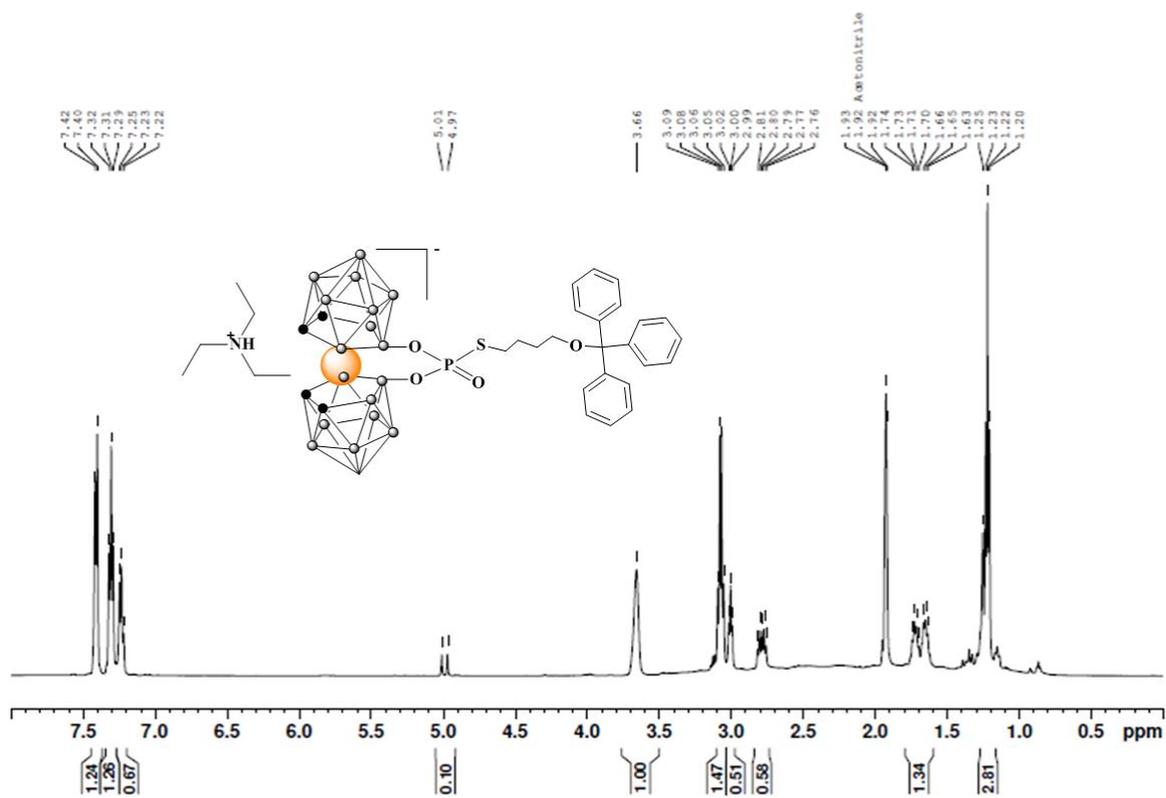


Figure S46. ^1H NMR spectrum of $[8,8'\text{-O}_2\text{P(O)S(CH}_2)_4\text{OCPh}_3\text{-}3,3'\text{-Co(1,2-C}_2\text{B}_9\text{H}_{10})_2] \text{HNEt}_3$ (15)

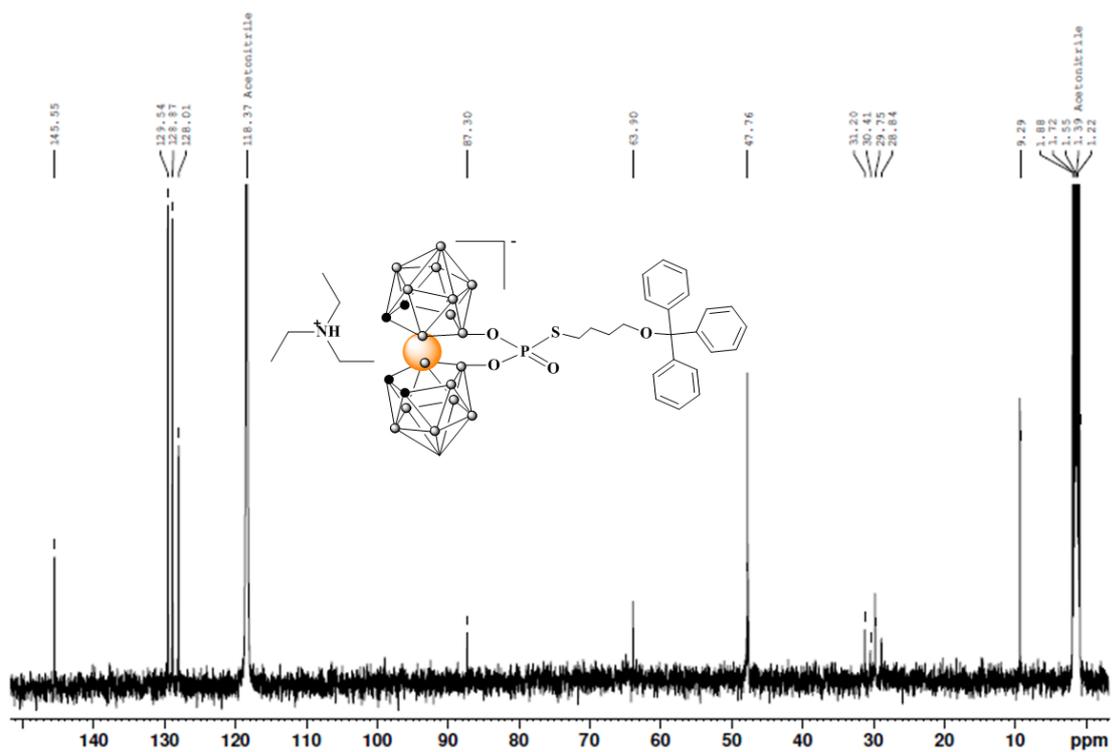


Figure S47. ^{13}C NMR spectrum of $[8,8'\text{-O}_2\text{P(O)S(CH}_2)_4\text{OCPh}_3\text{-}3,3'\text{-Co(1,2-C}_2\text{B}_9\text{H}_{10})_2] \text{HNEt}_3$ (15)

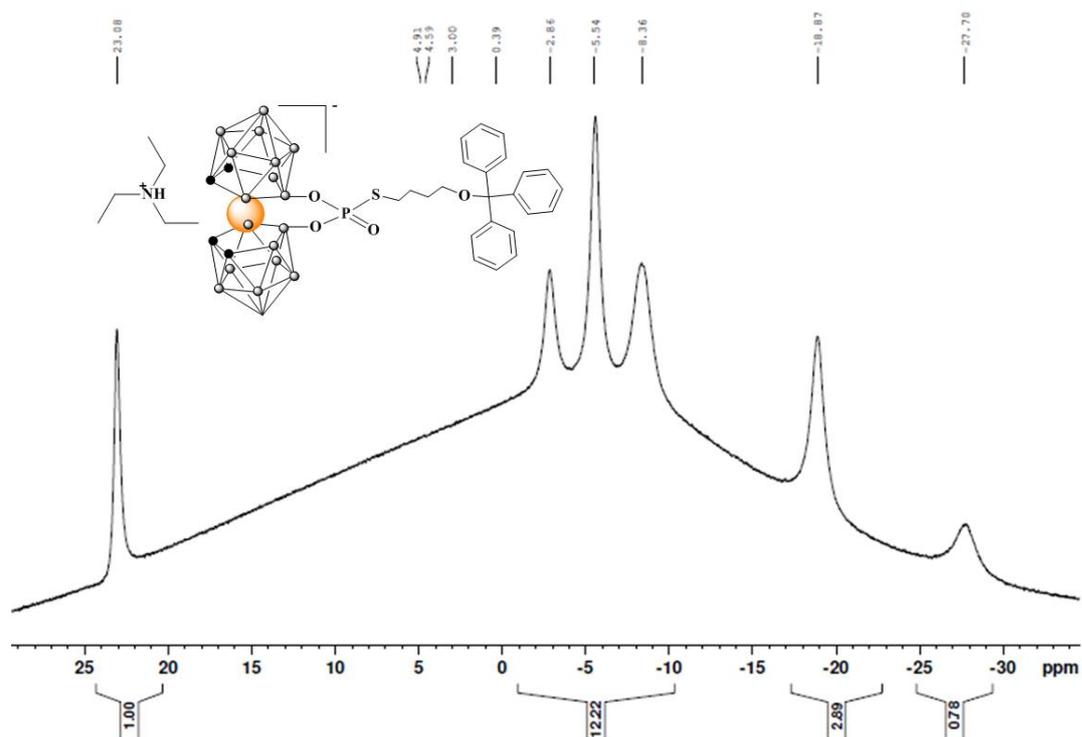


Figure S48. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of $[8,8'\text{-O}_2\text{P(O)S(CH}_2)_4\text{OCPh}_3\text{-}3,3'\text{-Co(1,2-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 (**15**)

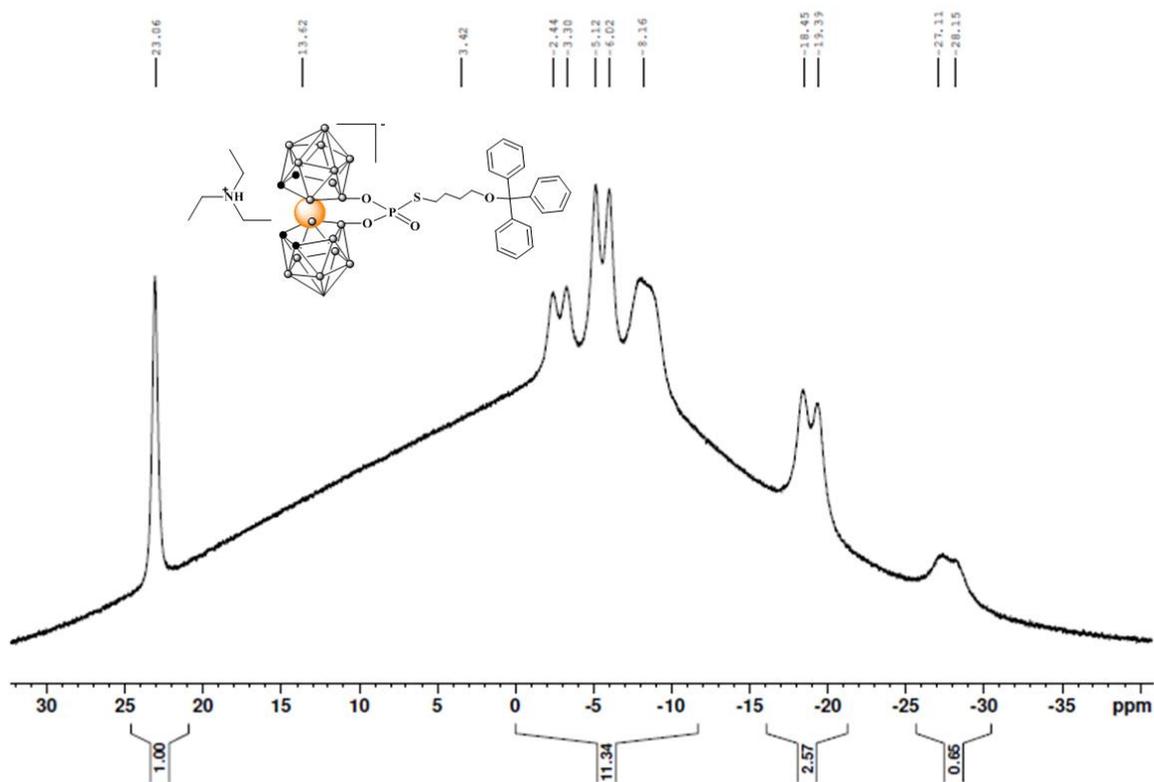


Figure S49. ^{11}B NMR spectrum of $[8,8'\text{-O}_2\text{P(O)S(CH}_2)_4\text{OCPh}_3\text{-}3,3'\text{-Co(1,2-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 (**15**)

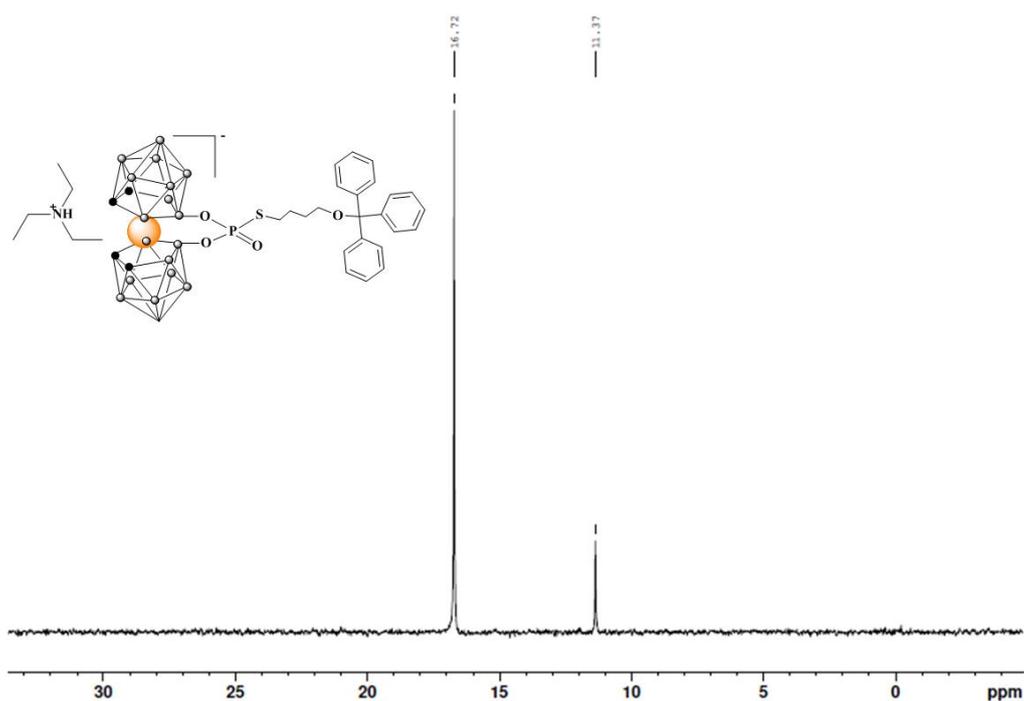


Figure S50. $^{31}P\{^1H\}$ NMR spectrum of $[8,8'-O_2P(O)S(CH_2)_4OCPh_3-3,3'-Co(1,2-C_2B_9H_{10})_2] HNEt_3$ (**15**)

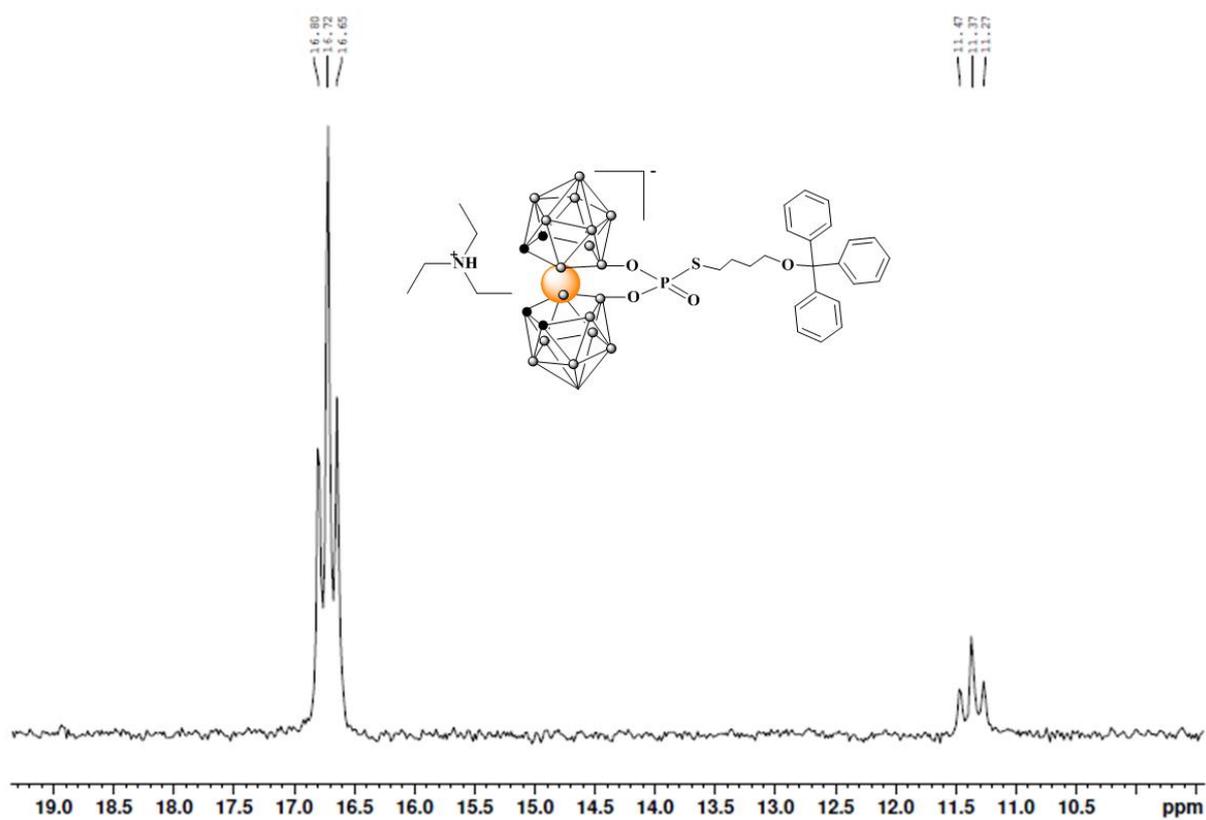


Figure S51. ^{31}P NMR spectrum of $[8,8'-O_2P(O)S(CH_2)_4OCPh_3-3,3'-Co(1,2-C_2B_9H_{10})_2] HNEt_3$ (**15**)

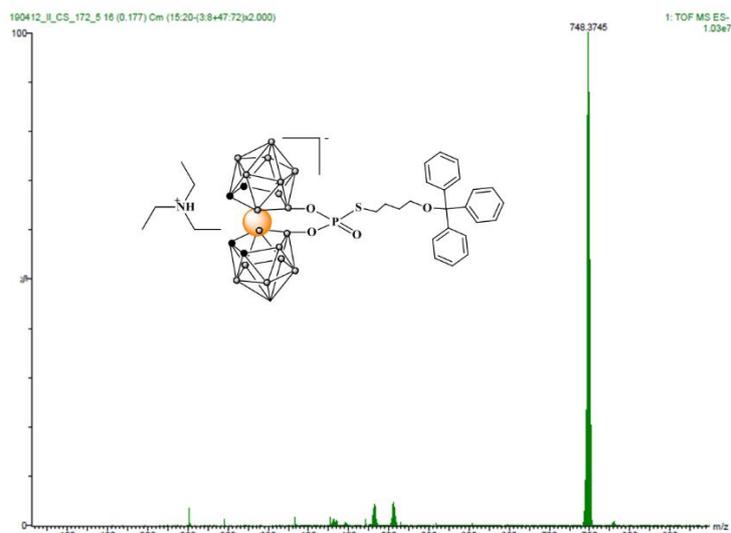


Figure S52. MS (ESI) spectrum of [8,8'-O₂P(O)S(CH₂)₄OCPh₃-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ (**15**)

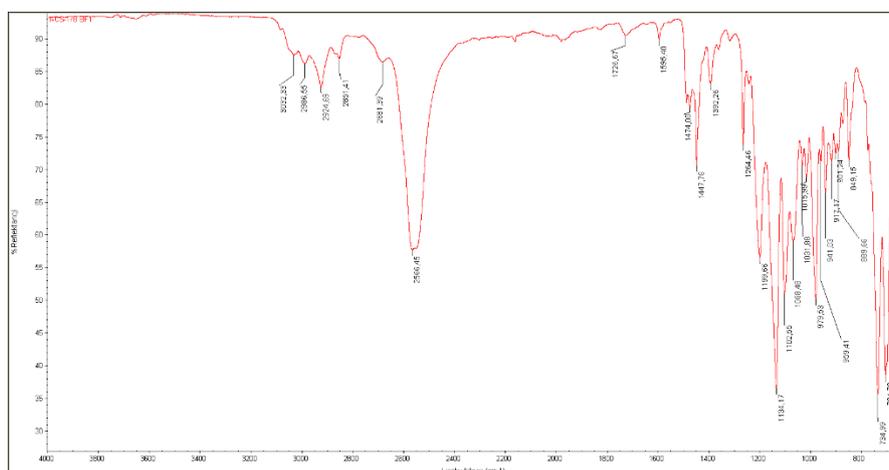


Figure S53. FT-IR spectrum of [8,8'-O₂P(O)S(CH₂)₄OCPh₃-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ (**15**)

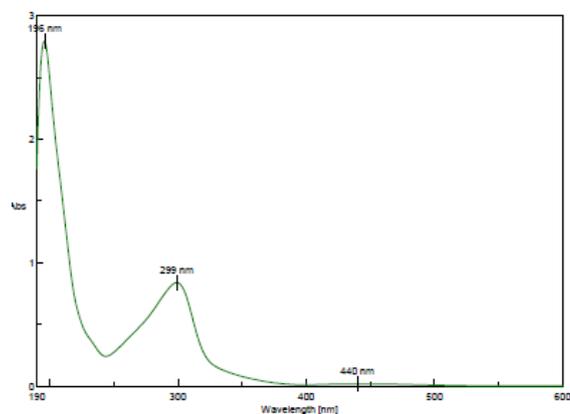


Figure S54. UV-VIS spectrum of [8,8'-O₂P(O)S(CH₂)₄OCPh₃-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ (**15**)

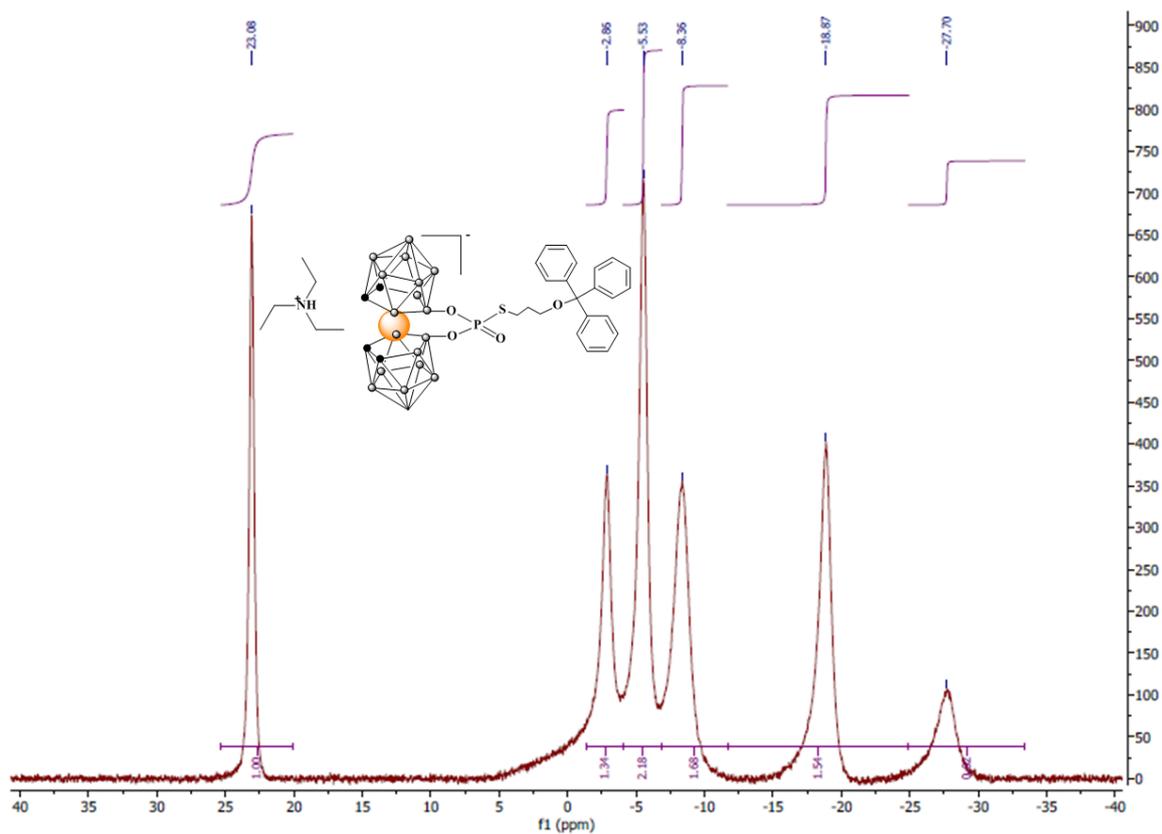


Figure S57. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_3\text{OCPh}_3\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 (**16**)

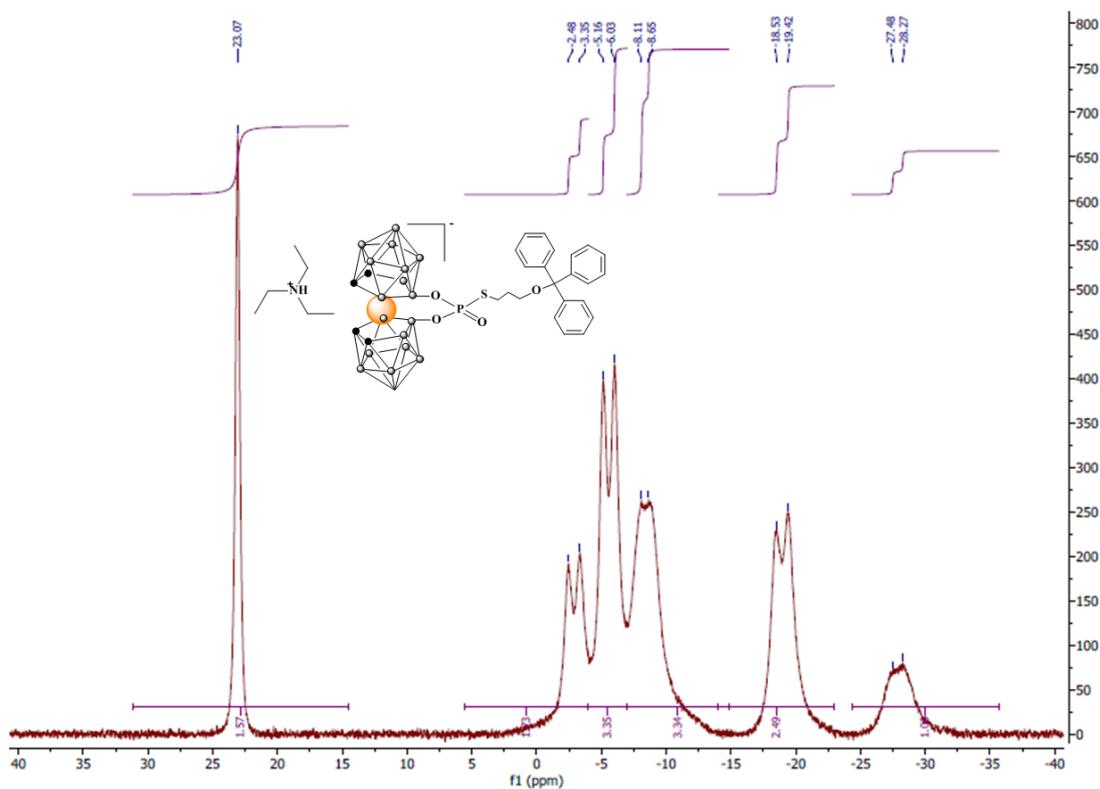


Figure S58. ^{11}B NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_3\text{OCPh}_3\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 (**16**)

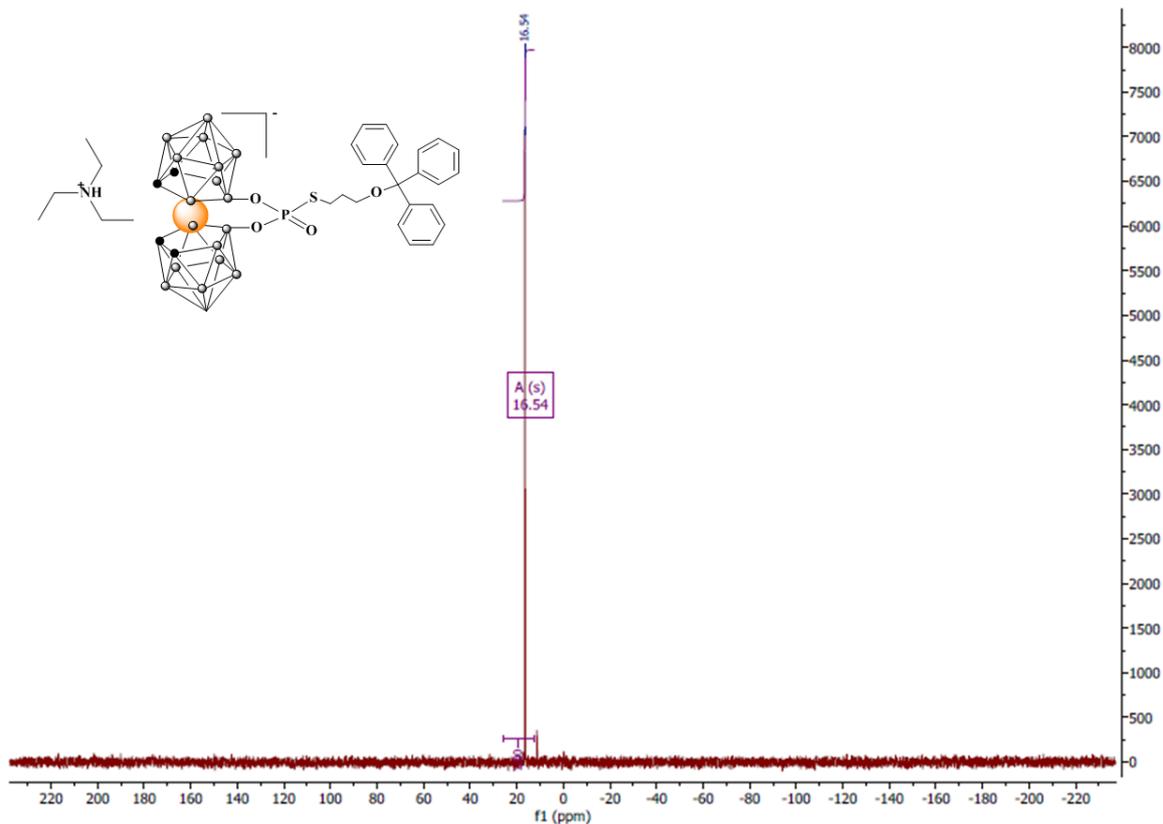


Figure S59. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_3\text{OCPh}_3\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 (**16**)

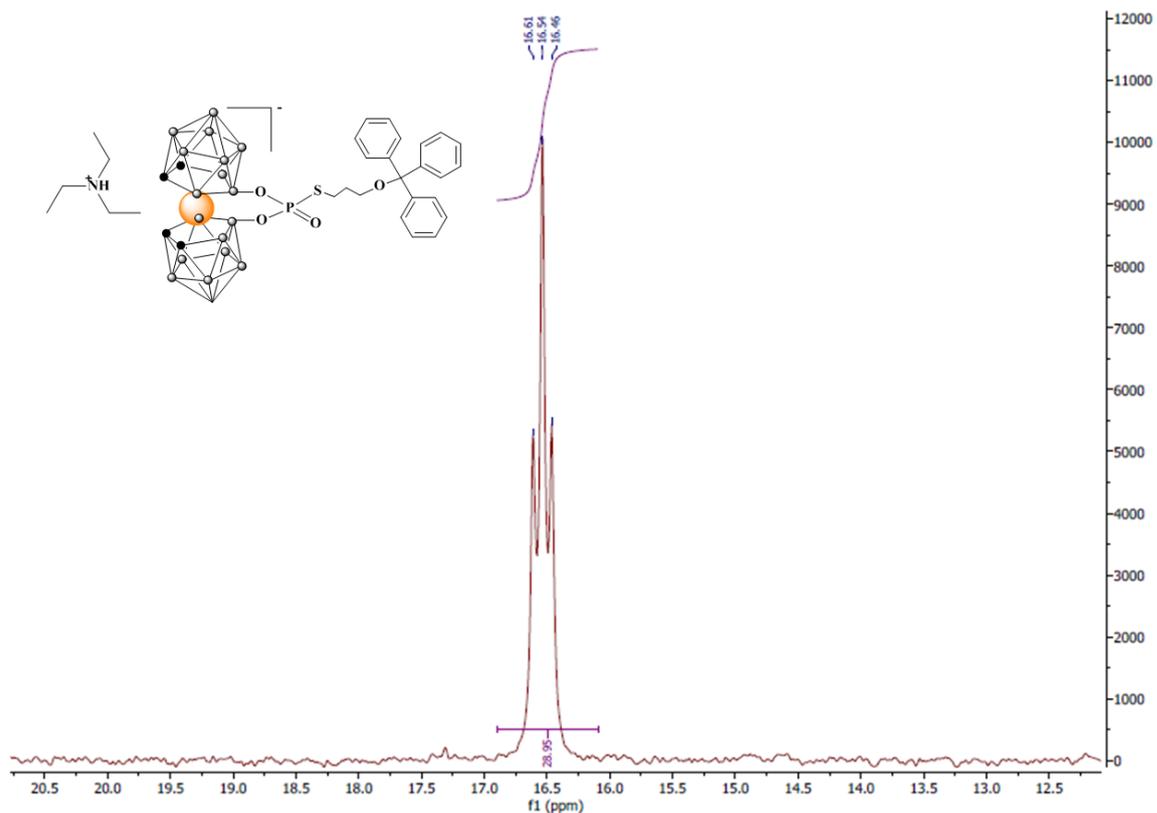


Figure S60. ^{31}P NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_3\text{OCPh}_3\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]$ HNEt_3 (**16**)

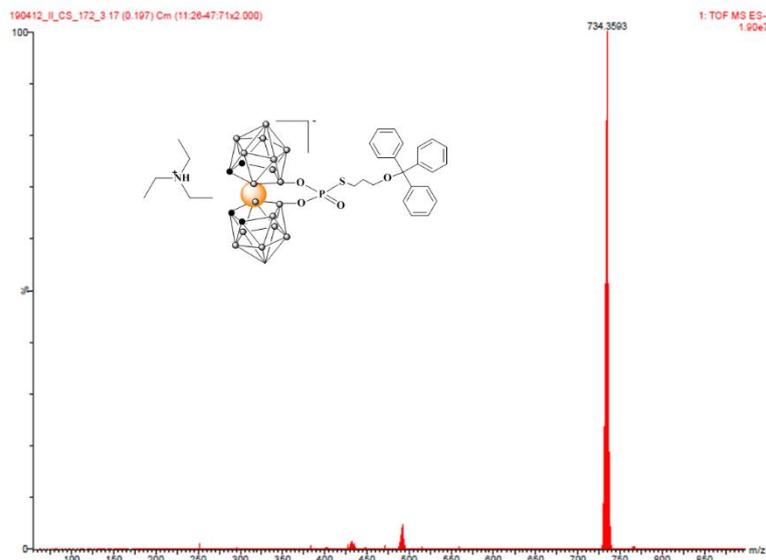


Figure S61. MS (ESI) spectrum of [8,8'-O₂P(O)S(CH₂)₃OCPh₃-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ (**16**)

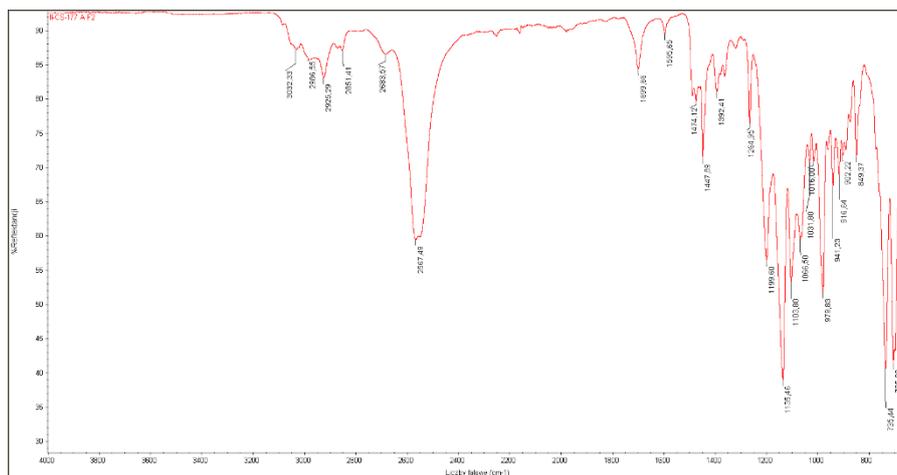


Figure S62. FT-IR spectrum of [8,8'-O₂P(O)S(CH₂)₃OCPh₃-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ (**16**)

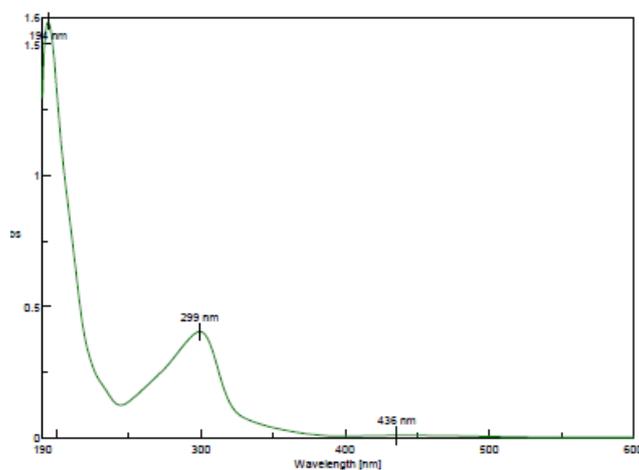


Figure S63. UV-VIS spectrum of [8,8'-O₂P(O)S(CH₂)₃OCPh₃-3,3'-Co(1,2-C₂B₉H₁₀)₂] HNEt₃ (**16**)

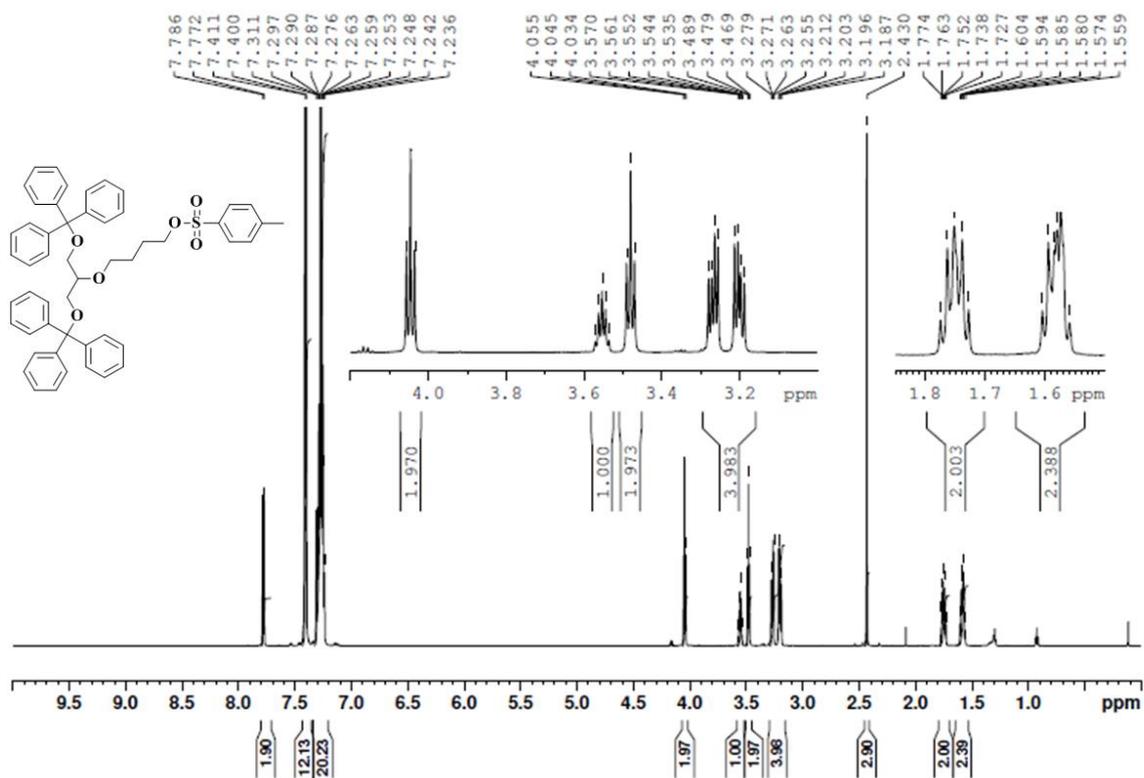


Figure S64. ¹H NMR spectrum of 4-(1,3-bis(trityloxy)propan-2-yloxy)butyl 4-methylbenzenesulfonate (**20**)

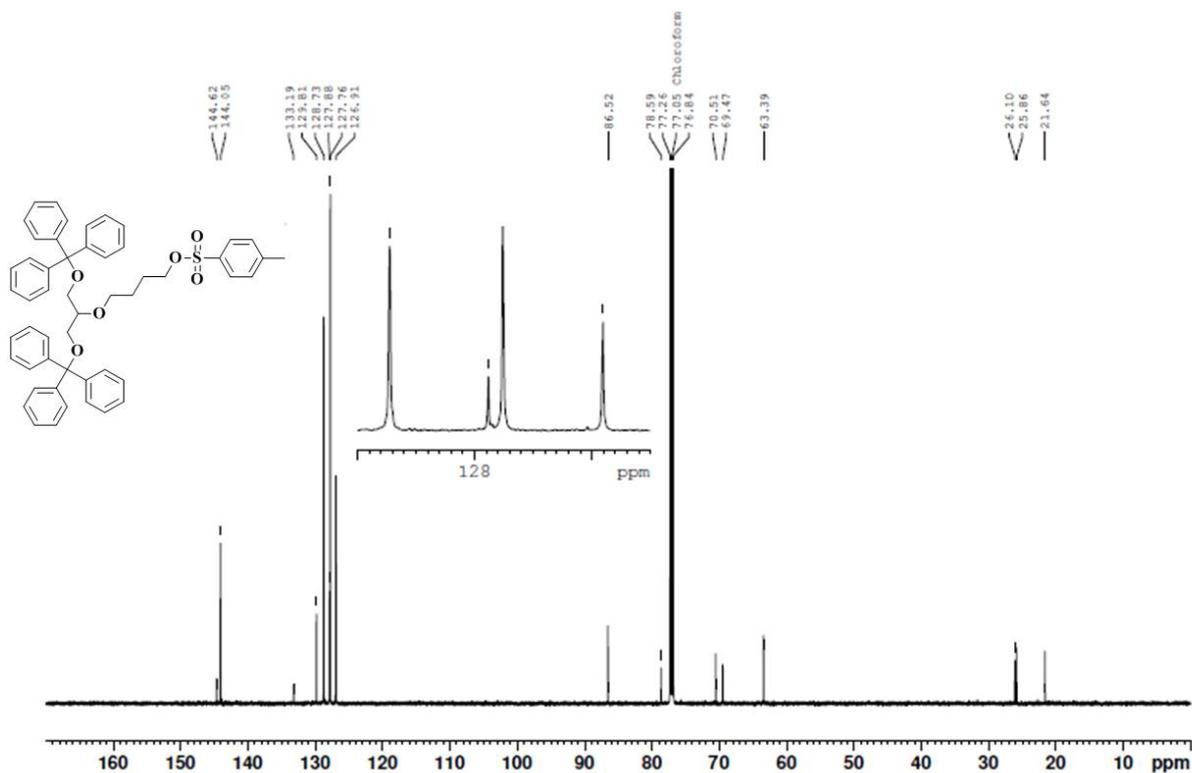


Figure S65. ¹³C NMR spectrum of 4-(1,3-bis(trityloxy)propan-2-yloxy)butyl 4-methylbenzenesulfonate (**20**)

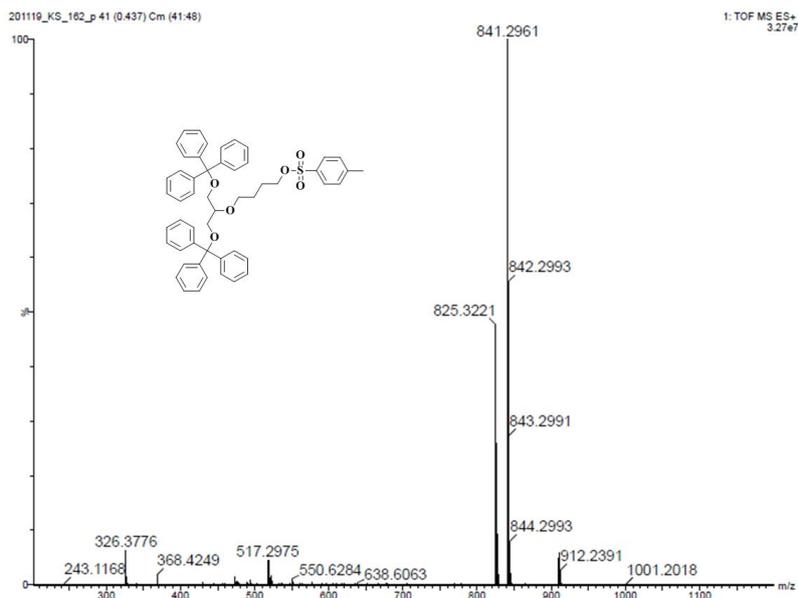


Figure S66. MS (ESI) spectrum of 4-(1,3-bis(trityloxy)propan-2-yloxy)butyl 4-methylbenzenesulfonate (**20**)

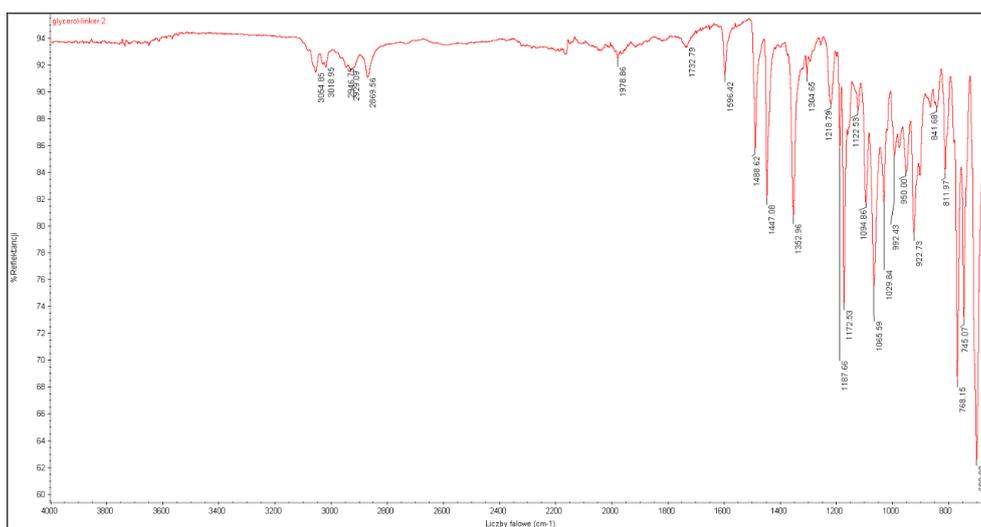


Figure S67. FT-IR spectrum of spectrum of 4-(1,3-bis(trityloxy)propan-2-yloxy)butyl 4-methylbenzenesulfonate (**20**)

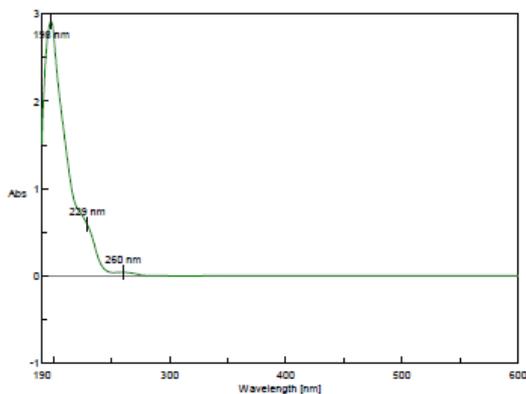


Figure S67. UV-VIS spectrum of 4-(1,3-bis(trityloxy)propan-2-yloxy)butyl 4-methylbenzenesulfonate (**20**)

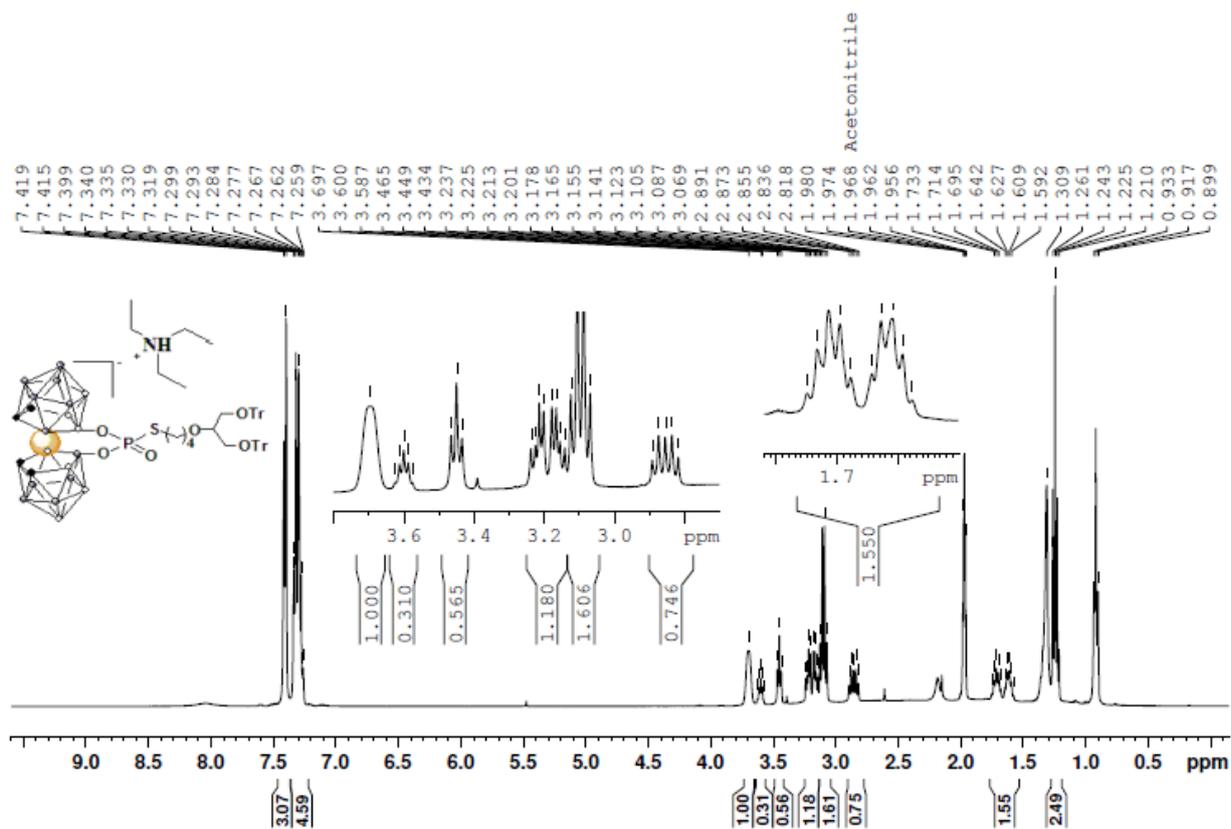


Figure S68. ^1H NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OTr})_2\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]\text{HNEt}_3$ (**21**)

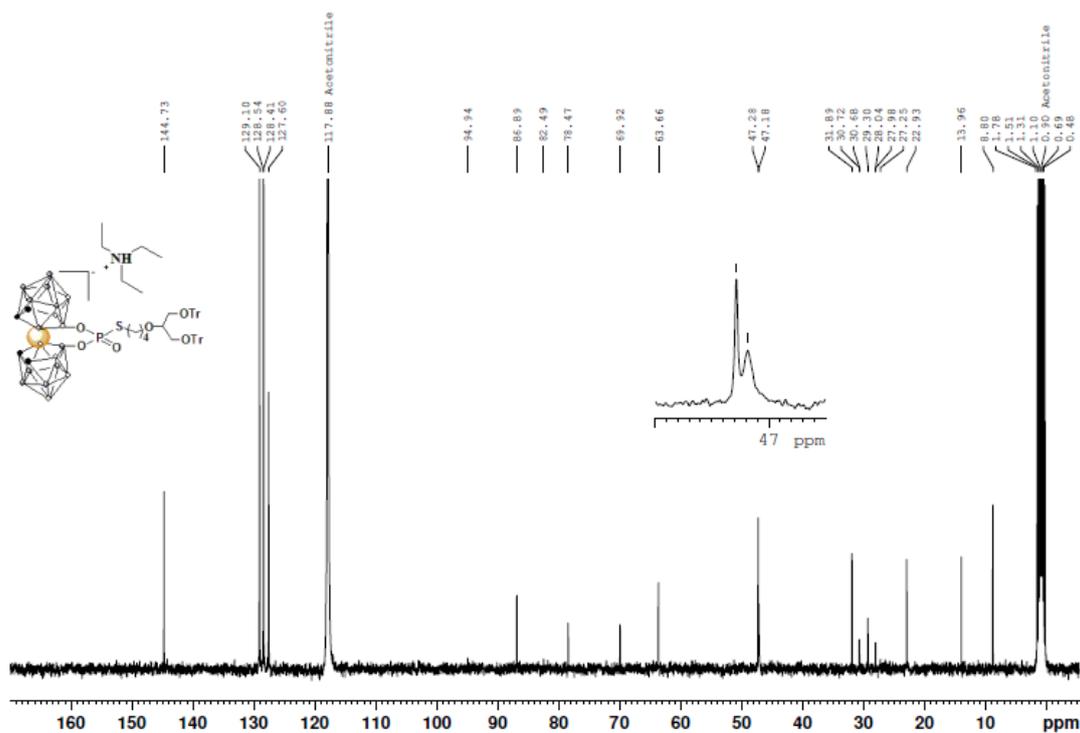


Figure S69. ^{13}C NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OTr})_2\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]\text{HNEt}_3$ (**21**)

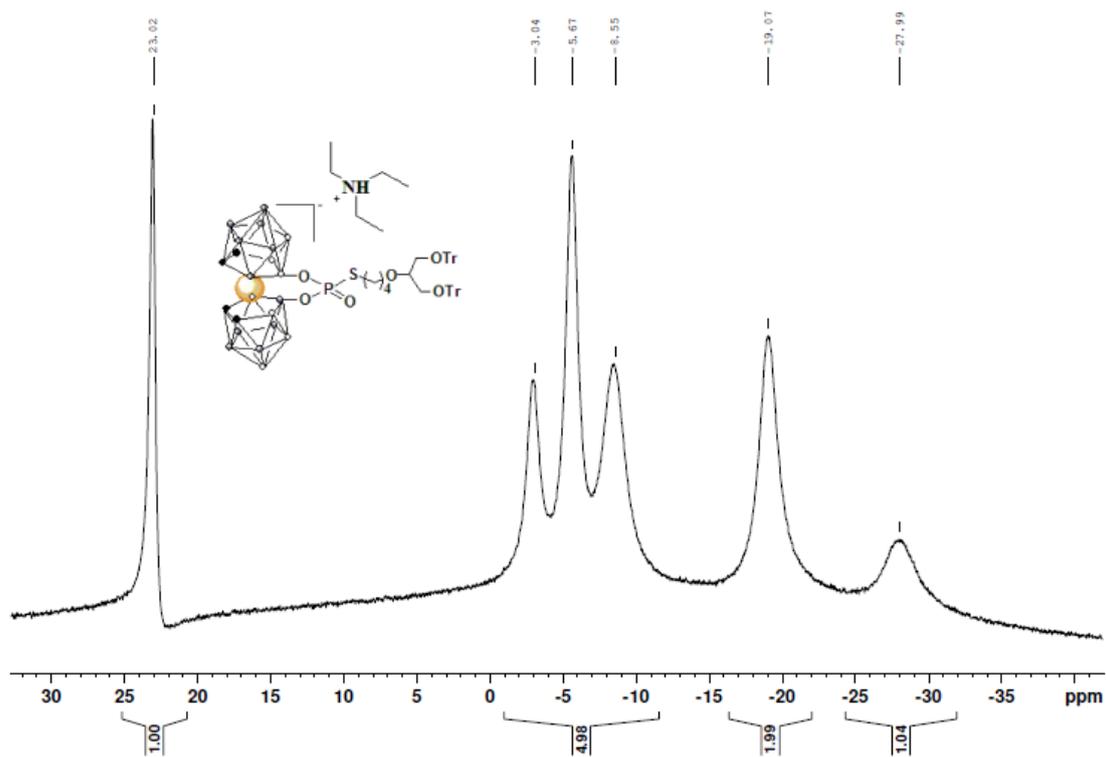


Figure S70. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OTr})_2\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]\text{HNEt}_3$ (**21**)

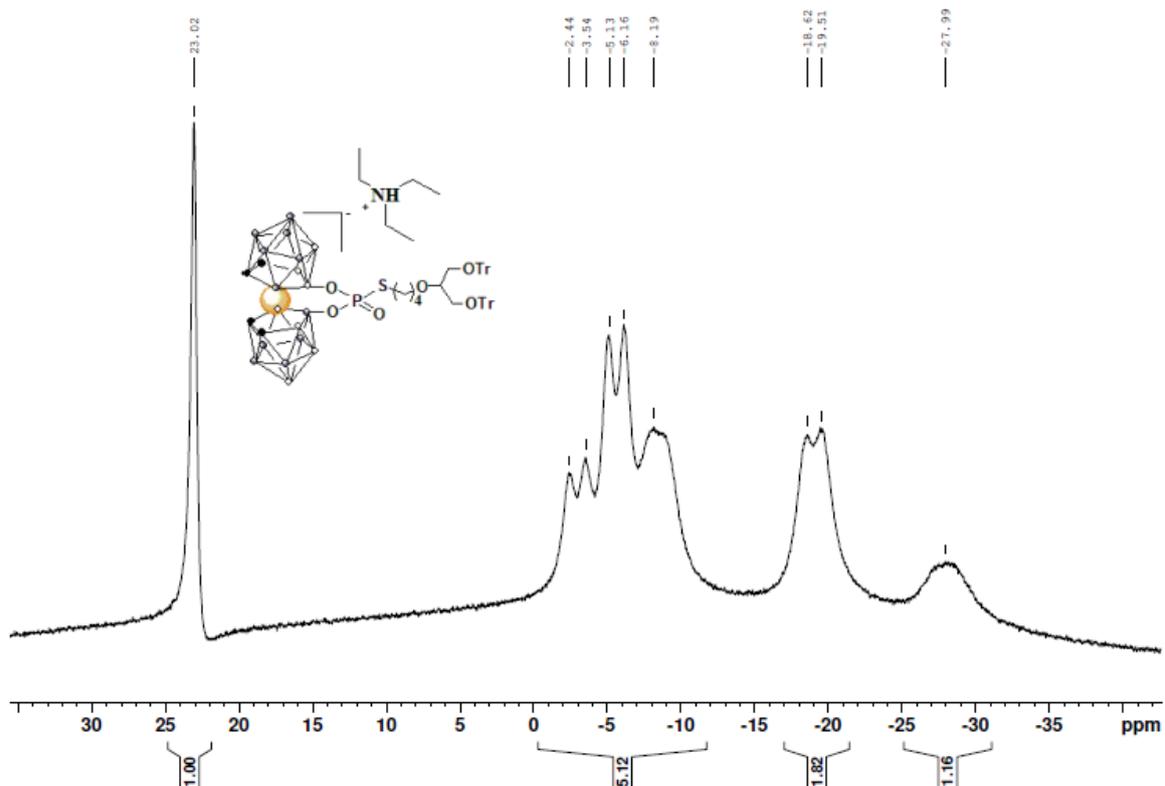


Figure S71. ^{11}B NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OTr})_2\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]\text{HNEt}_3$ (**21**)

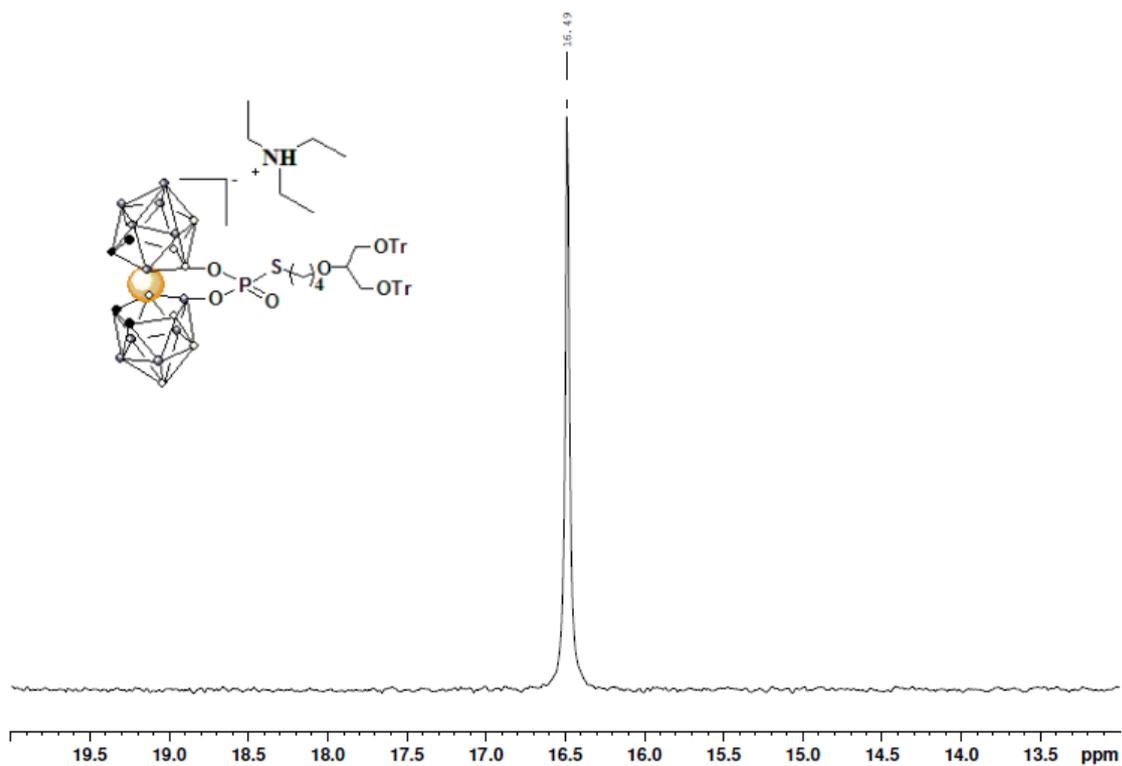


Figure S72. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OTr})_2\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]\text{HNEt}_3$ (**21**)

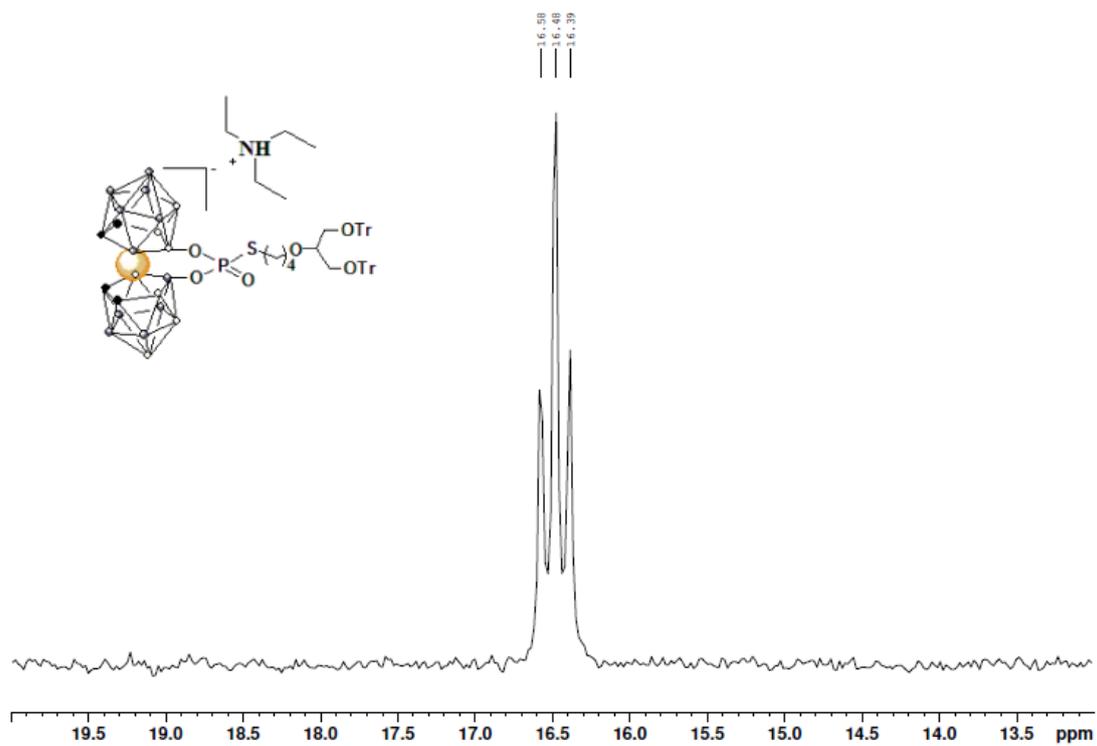


Figure S73. ^{31}P NMR spectrum of $[8,8'\text{-O}_2\text{P}(\text{O})\text{S}(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OTr})_2\text{-}3,3'\text{-Co}(1,2\text{-C}_2\text{B}_9\text{H}_{10})_2]\text{HNEt}_3$ (**21**)

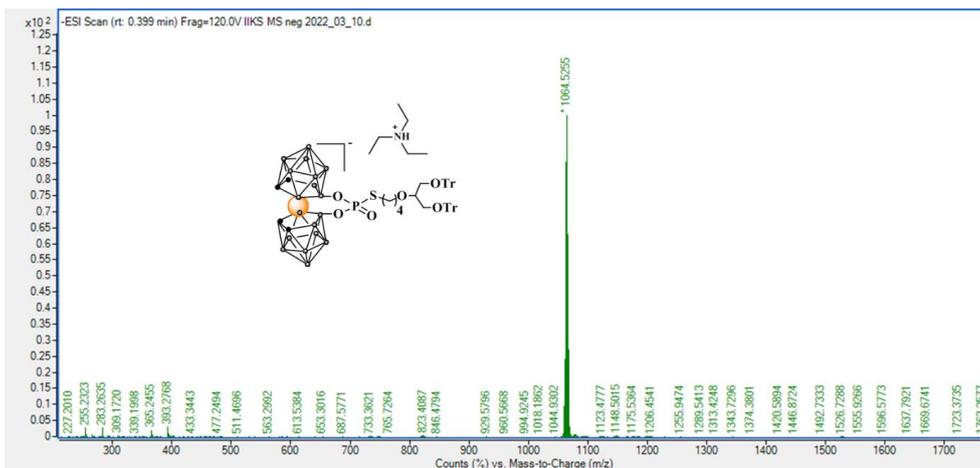


Figure S74. MS (ESI) spectrum of [8,8'-O₂P(O)S(CH₂)₄OCH(CH₂OTr)₂-3,3'-Co(1,2-C₂B₉H₁₀)₂]HNEt₃ (**21**)

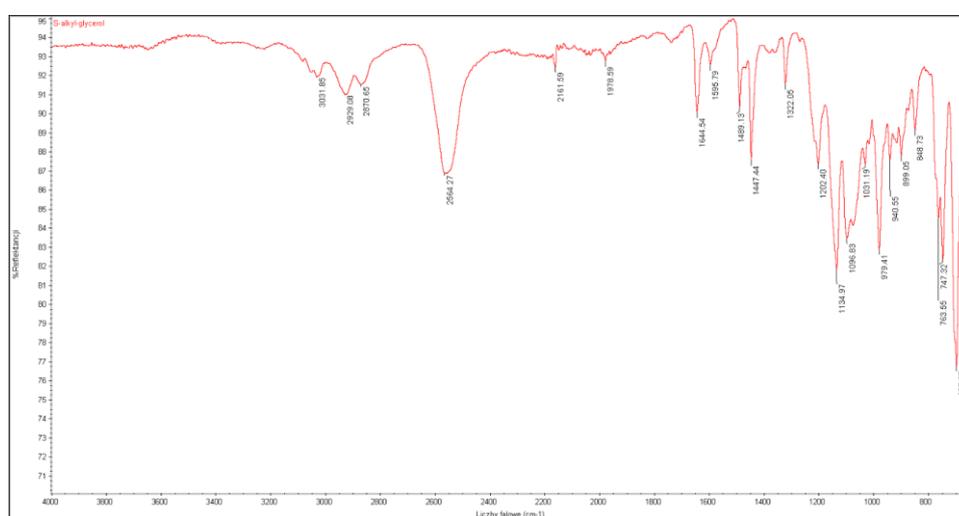


Figure S75. MS (ESI) spectrum of [8,8'-O₂P(O)S(CH₂)₄OCH(CH₂OTr)₂-3,3'-Co(1,2-C₂B₉H₁₀)₂]HNEt₃ (**21**)

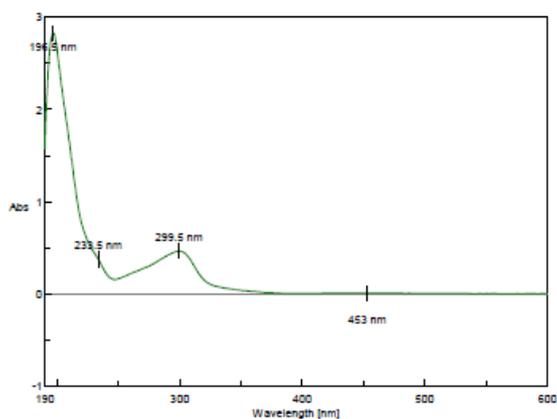


Figure S76. UV-VIS spectrum of spectrum of [8,8'-O₂P(O)S(CH₂)₄OCH(CH₂OTr)₂-3,3'-Co(1,2-C₂B₉H₁₀)₂]HNEt₃ (**21**)

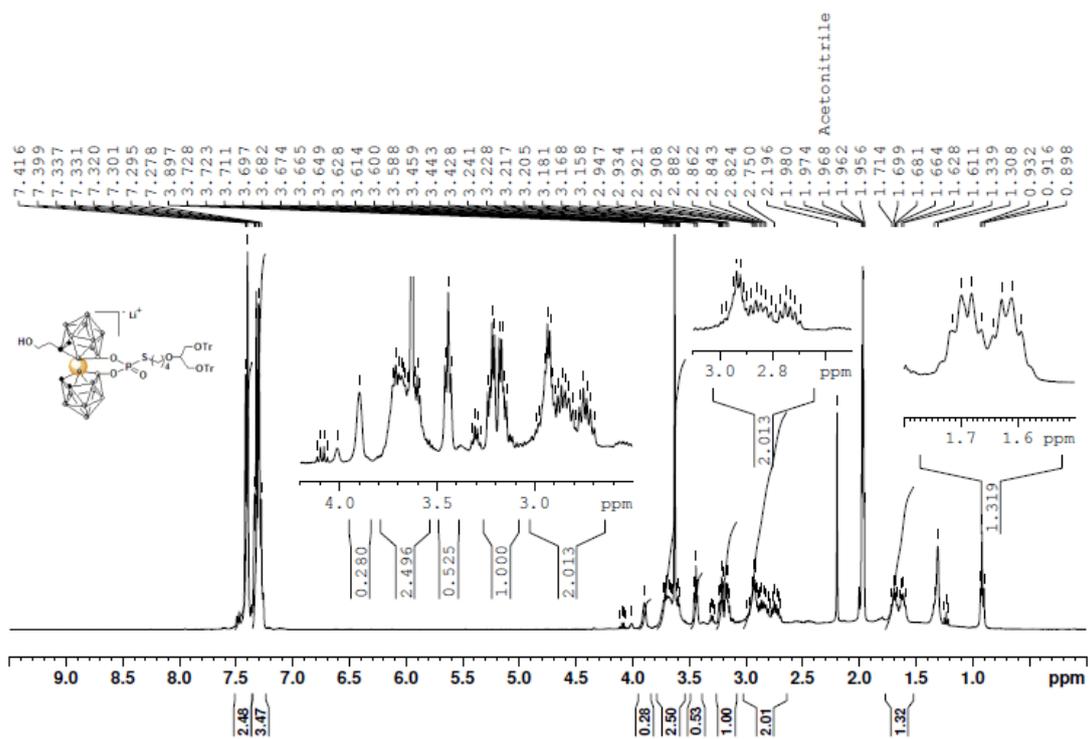


Figure S77. ^1H NMR spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-}(\text{CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\}$ (**22**)

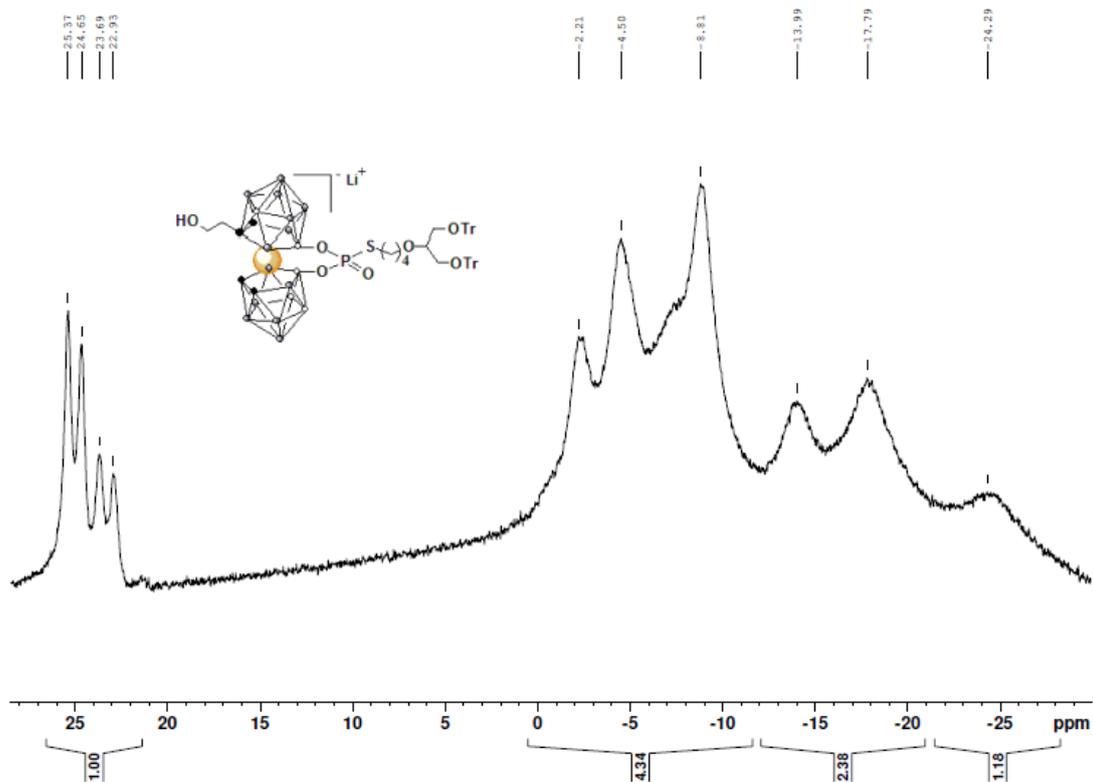


Figure S78. $^{11}\text{B}\{^1\text{H}\}$ NMR spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-}(\text{CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\}$ (**22**)

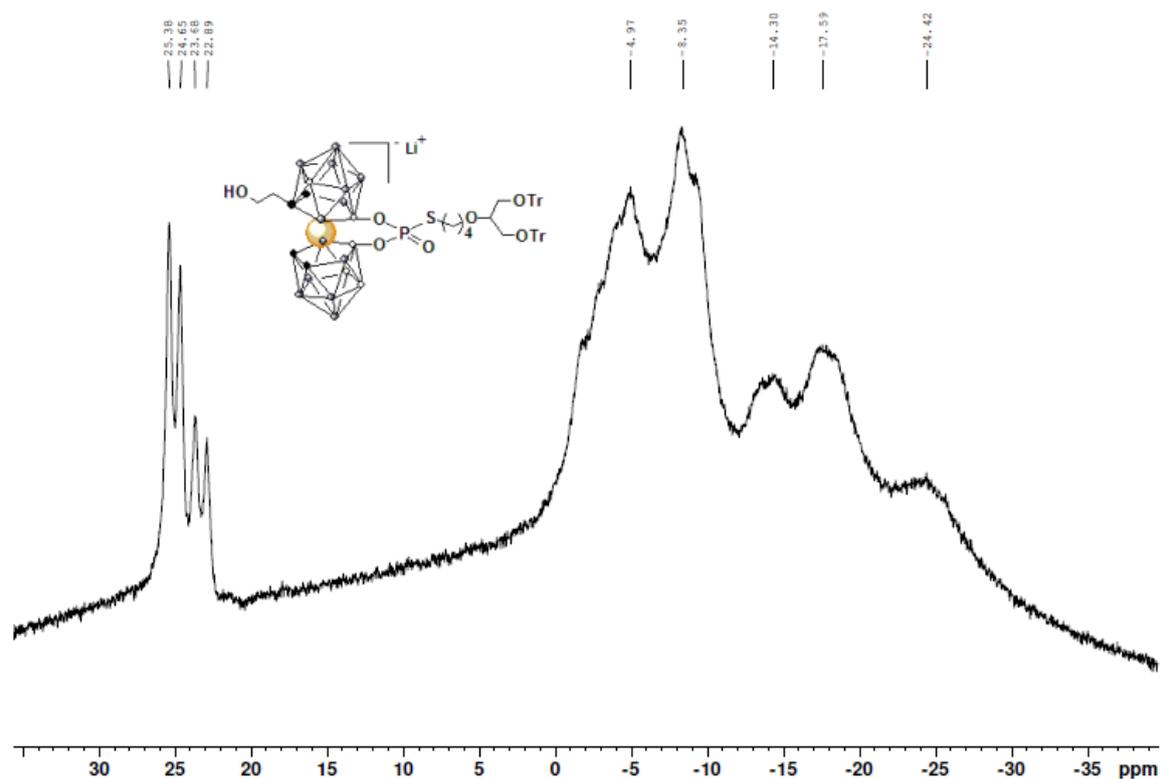


Figure S79. ¹¹B NMR spectrum of {8,8'-O₂P(O)S[(CH₂)₄OCH(CH₂OCPH₃)₂]-3,3'-Co[1-(CH₂)₂OH-1,2-C₂B₉H₁₀](1',2'-C₂B₉H₁₀)} (**22**)

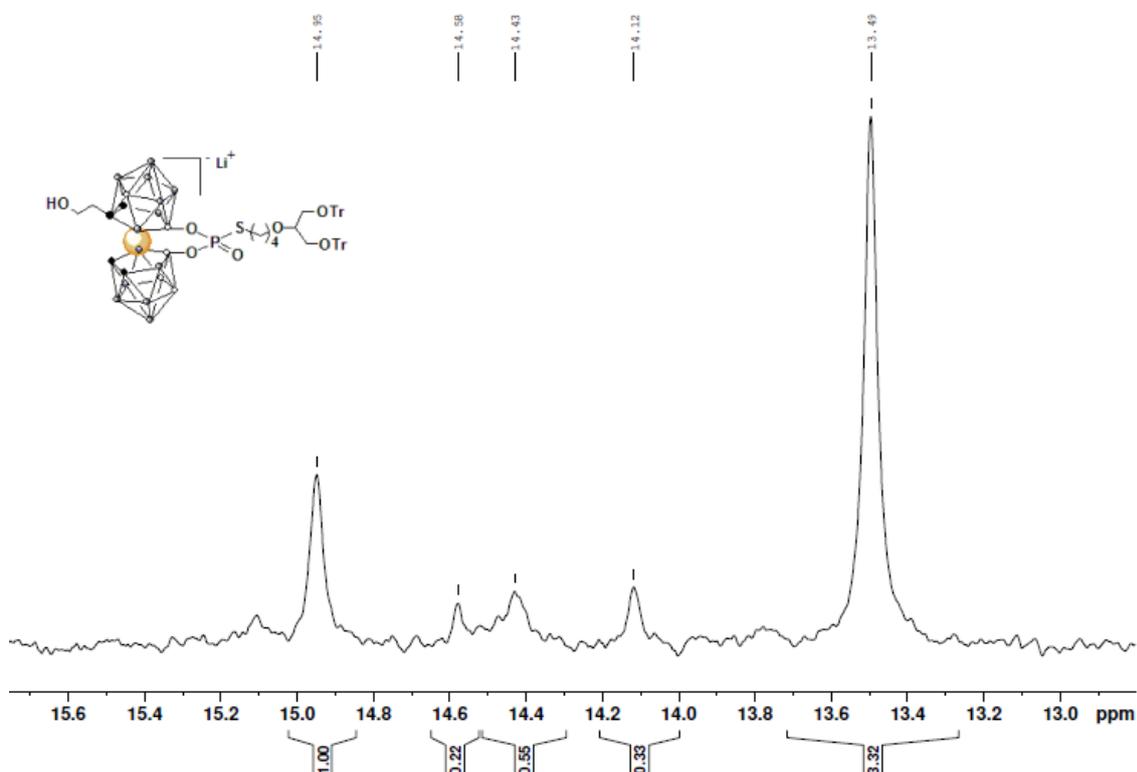


Figure S80. ³¹P{¹H} NMR spectrum of {8,8'-O₂P(O)S[(CH₂)₄OCH(CH₂OCPH₃)₂]-3,3'-Co[1-(CH₂)₂OH-1,2-C₂B₉H₁₀](1',2'-C₂B₉H₁₀)} (**22**)

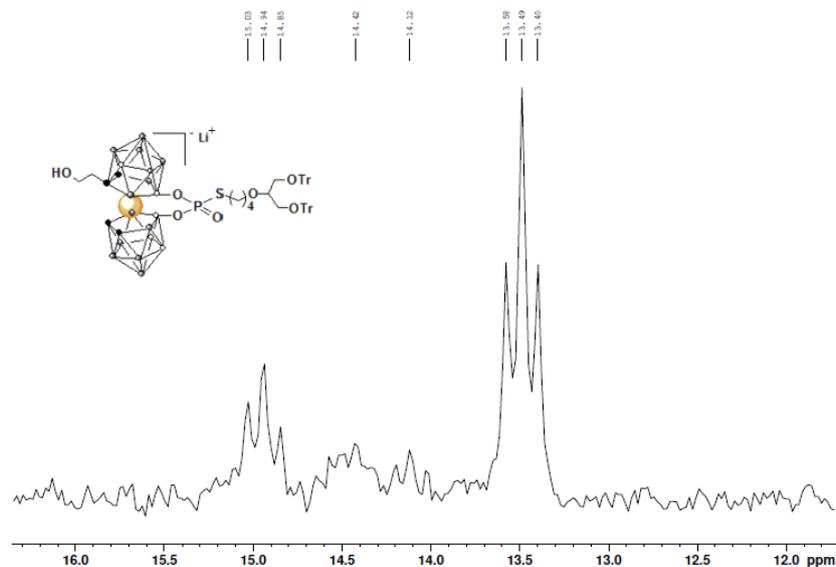


Figure S81. ^{31}P NMR spectrum of $\{8,8'-O_2P(O)S[(CH_2)_4OCH(CH_2OCPh_3)_2]-3,3'-Co[1-(CH_2)_2OH-1,2-C_2B_9H_{10}][1',2'-C_2B_9H_{10}]\}$ (**22**)

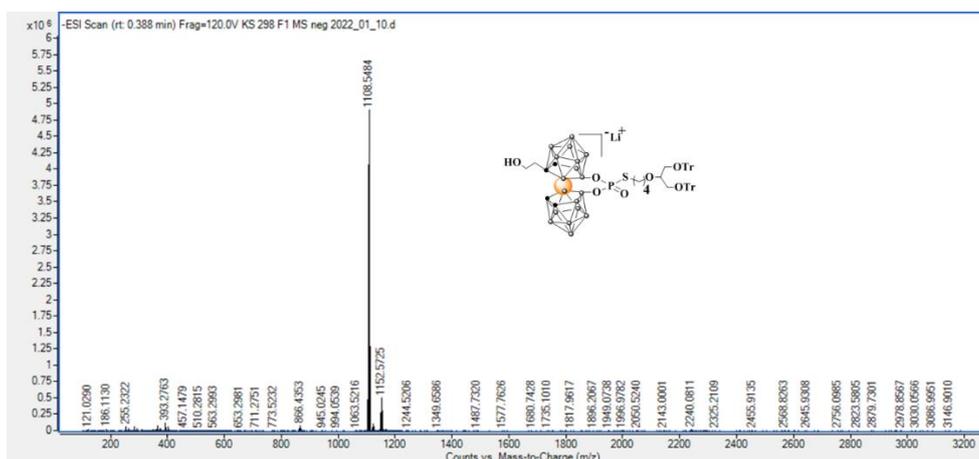


Figure S82. MS (ESI) spectrum of $\{8,8'-O_2P(O)S[(CH_2)_4OCH(CH_2OCPh_3)_2]-3,3'-Co[1-(CH_2)_2OH-1,2-C_2B_9H_{10}][1',2'-C_2B_9H_{10}]\}$ (**22**)

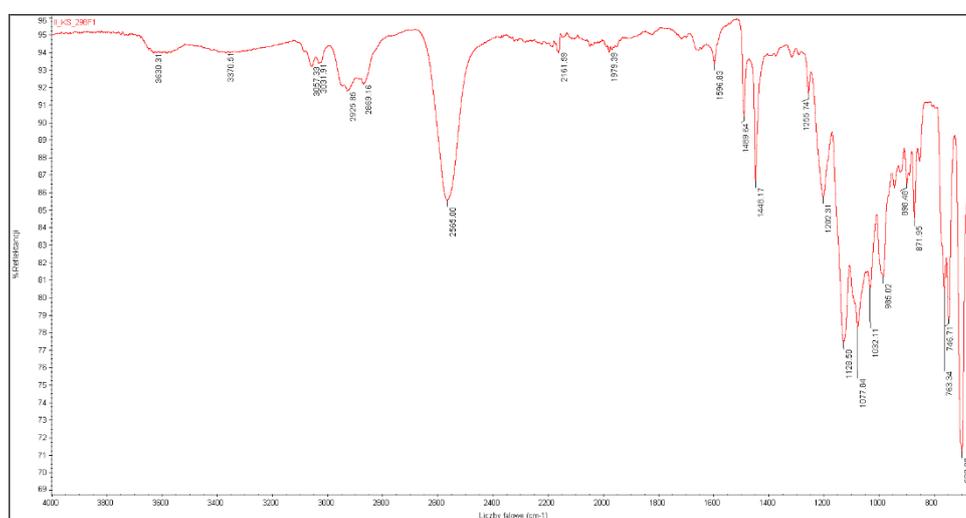


Figure S83. FT-IR spectrum of $\{8,8'-O_2P(O)S[(CH_2)_4OCH(CH_2OCPh_3)_2]-3,3'-Co[1-(CH_2)_2OH-1,2-C_2B_9H_{10}][1',2'-C_2B_9H_{10}]\}$ (**22**)

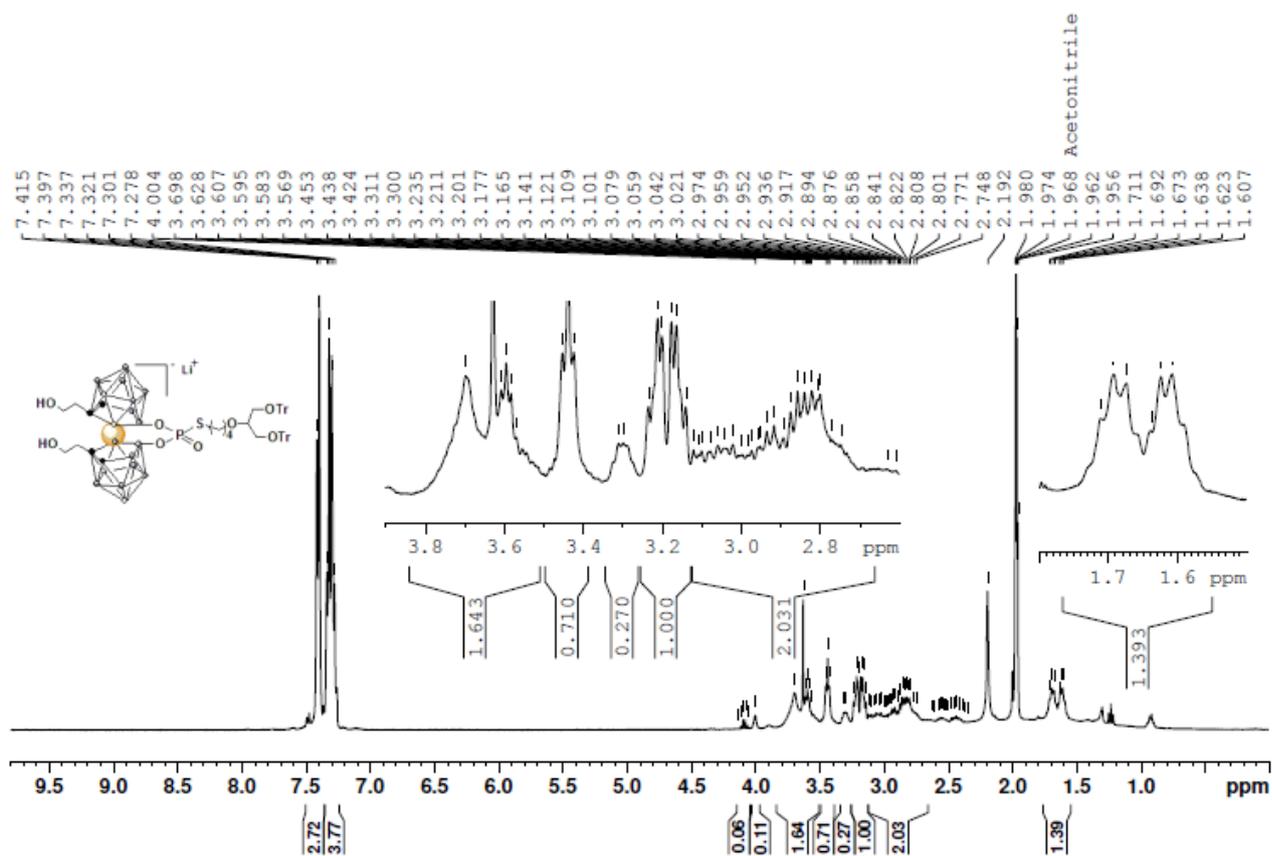


Figure S84. ¹H NMR spectrum of {8,8'-O₂P(O)S[(CH₂)₄OCH(CH₂OCPh₃)₂]-3,3'-Co[1-(CH₂)₂OH-1,2-C₂B₉H₁₀]} [1'-(CH₂)₂OH-1',2'-C₂B₉H₁₀]} HNEt₃ (**23**)

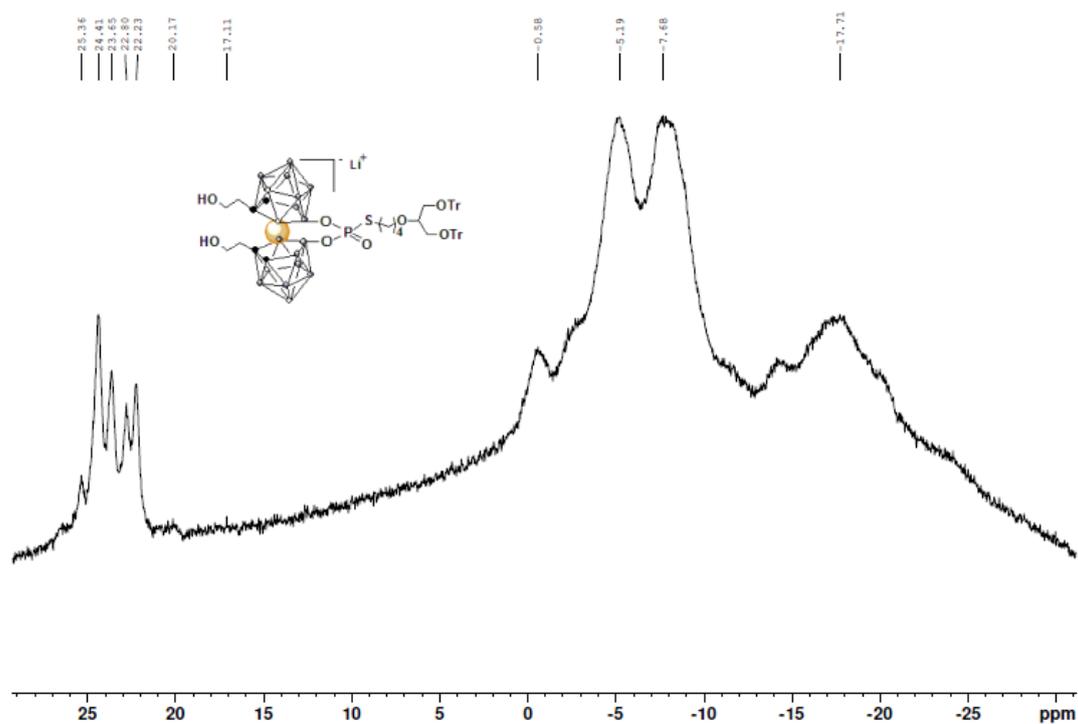


Figure S85. ¹¹B{¹H} NMR spectrum of {8,8'-O₂P(O)S[(CH₂)₄OCH(CH₂OCPh₃)₂]-3,3'-Co[1-(CH₂)₂OH-1,2-C₂B₉H₁₀]} [1'-(CH₂)₂OH-1',2'-C₂B₉H₁₀]} HNEt₃ (**23**)

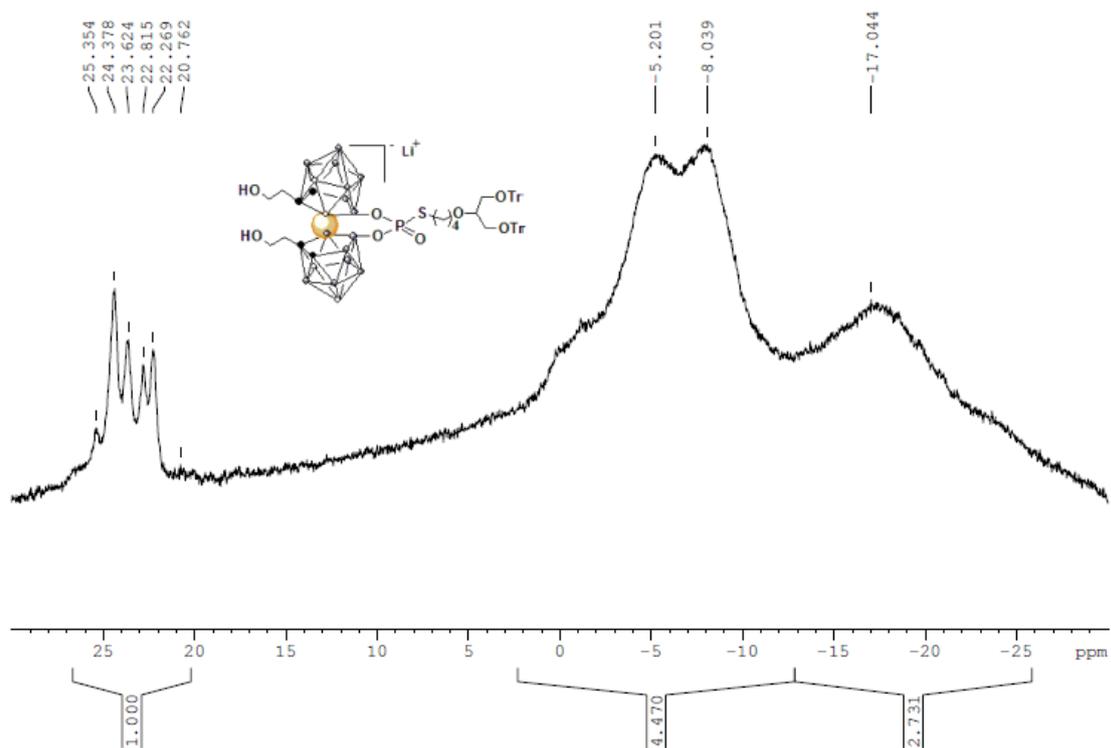


Figure S86. ^{11}B NMR spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-}(\text{CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1'\text{-}(\text{CH}_2)_2\text{OH-}1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\} \text{HNEt}_3$ (**23**)

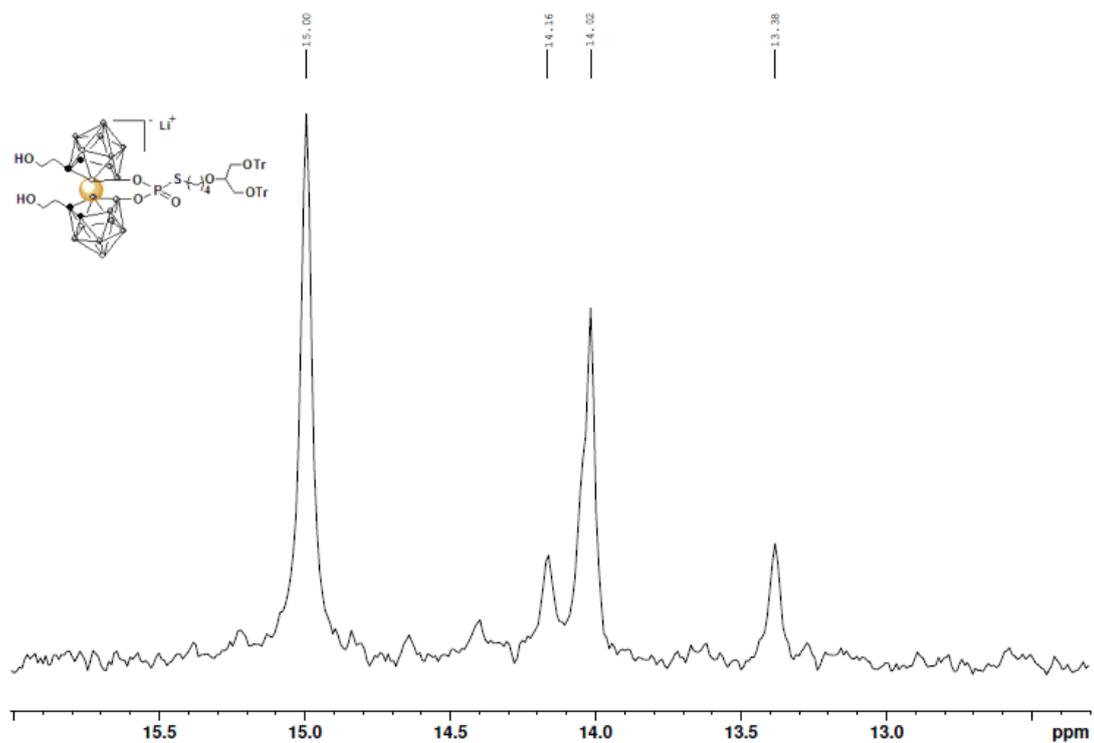


Figure S87. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-}(\text{CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1'\text{-}(\text{CH}_2)_2\text{OH-}1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\} \text{HNEt}_3$ (**23**)

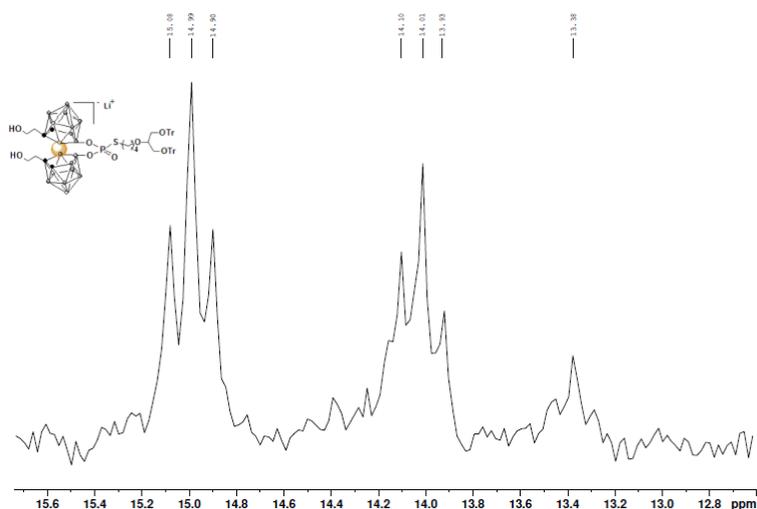


Figure S88. ^{31}P NMR spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-(CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1'\text{-(CH}_2)_2\text{OH-}1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\}$ $\text{HN}(\text{Et})_3$ (**23**)

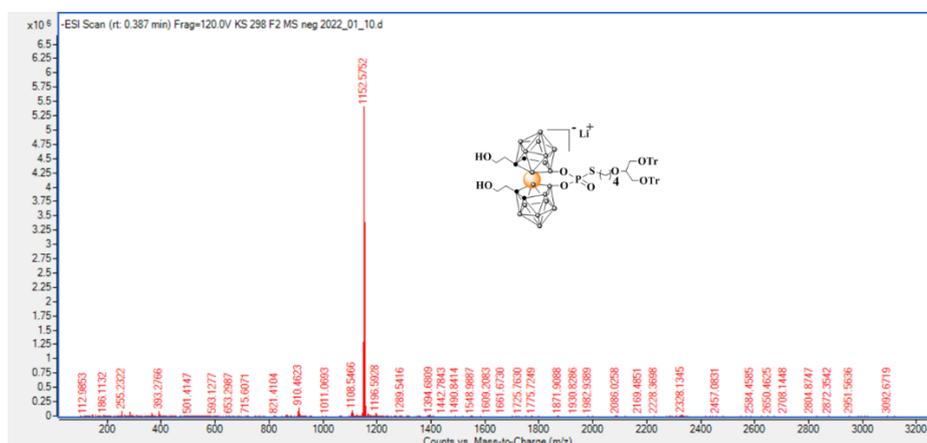


Figure S89. ESI (MS) spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-(CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1'\text{-(CH}_2)_2\text{OH-}1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\}$ $\text{HN}(\text{Et})_3$ (**23**)

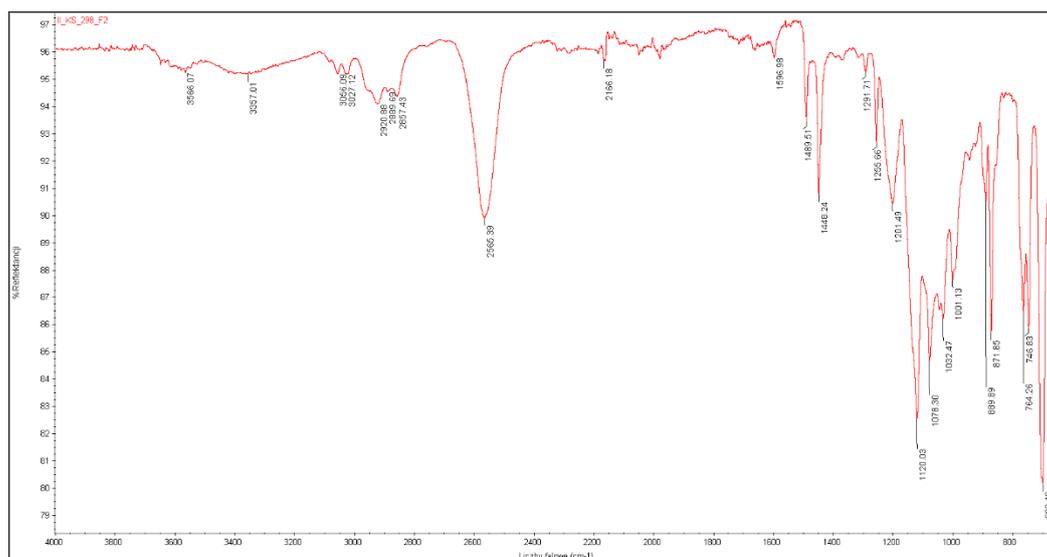


Figure S90. FT-IR spectrum of $\{8,8'\text{-O}_2\text{P}(\text{O})\text{S}[(\text{CH}_2)_4\text{OCH}(\text{CH}_2\text{OCPh}_3)_2]\text{-}3,3'\text{-Co}[1\text{-(CH}_2)_2\text{OH-}1,2\text{-C}_2\text{B}_9\text{H}_{10}][1'\text{-(CH}_2)_2\text{OH-}1',2'\text{-C}_2\text{B}_9\text{H}_{10}]\}$ $\text{HN}(\text{Et})_3$ (**23**)