J. Braz. Chem. Soc., Vol. 23, No. 2, S1-S17, 2012.
Printed in Brazil - ©2012 Sociedade Brasileira de Química
0103 - 5053 $6.00+0.00

Supplementary Information

Pd Complexes Based on Phosphine-Linked Cyclophosphazenes: Synthesis, Characterization and Application in Suzuki Coupling Reactions

Vanderlei I. de Paula, Cintia A. Sato and Regina Buffon*

Instituto de Química, Universidade Estadual de Campinas, CP 6154, 13084-971 Campinas-SP, Brazil

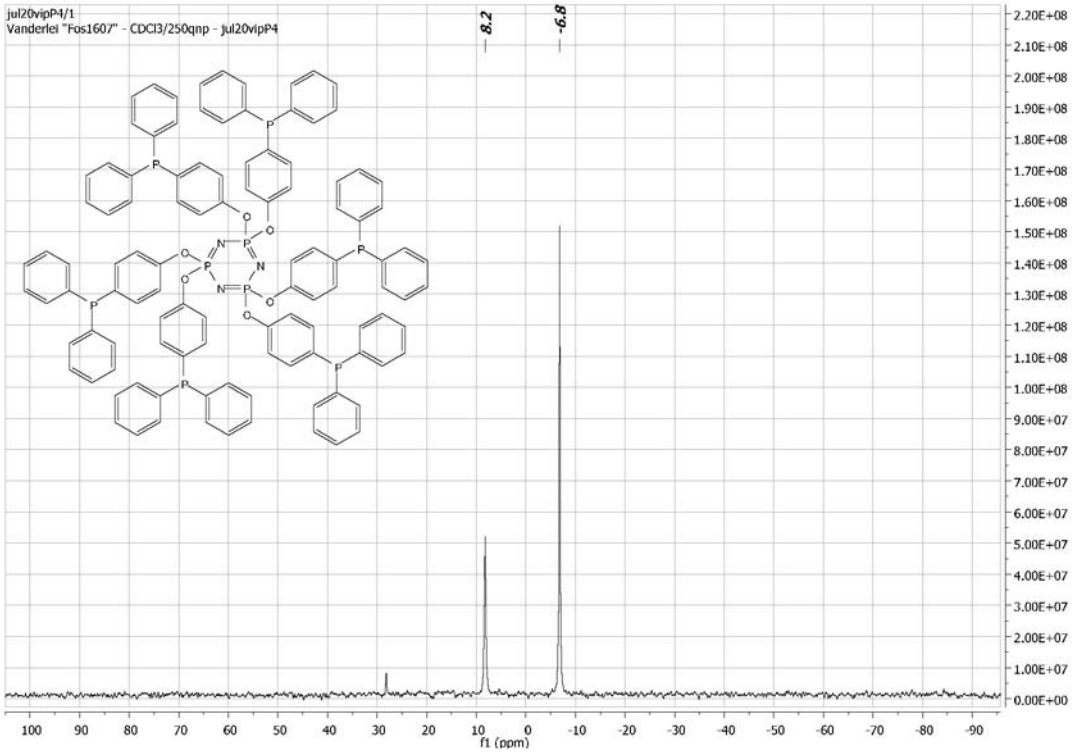

Figure S1. ^{31}P NMR spectrum of ligand **1a**.

*e-mail: rbuffon@iqm.unicamp.br

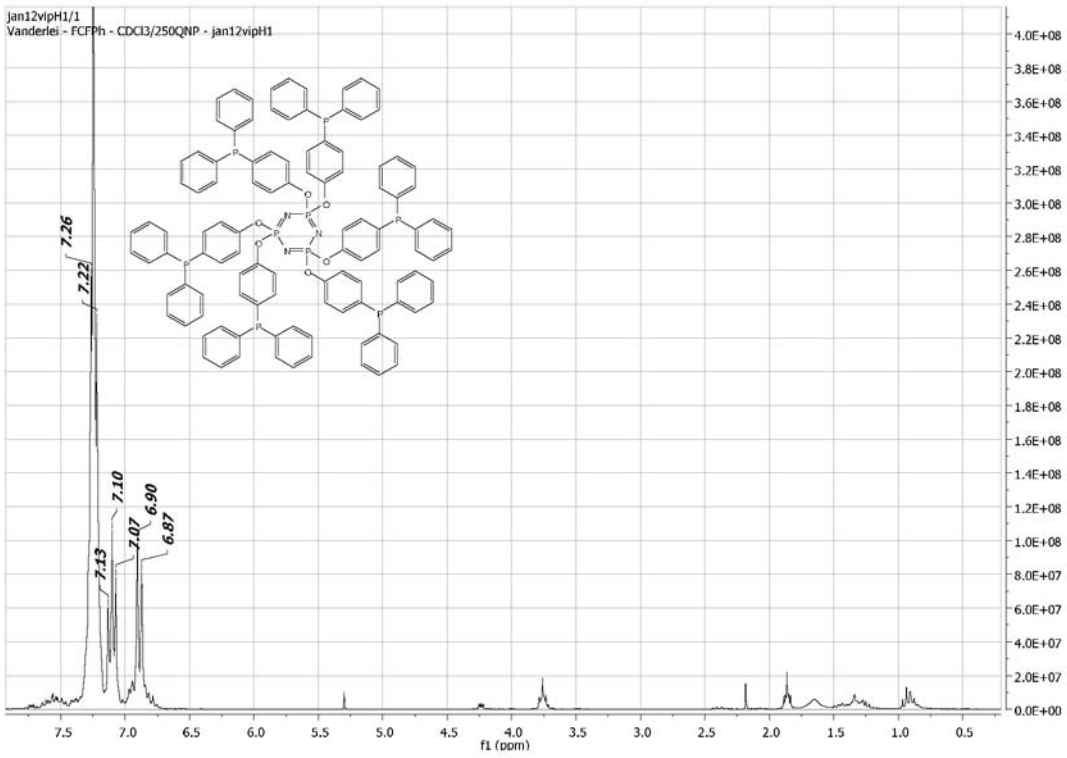

Figure S2. [1]H NMR spectrum of ligand **1a**.

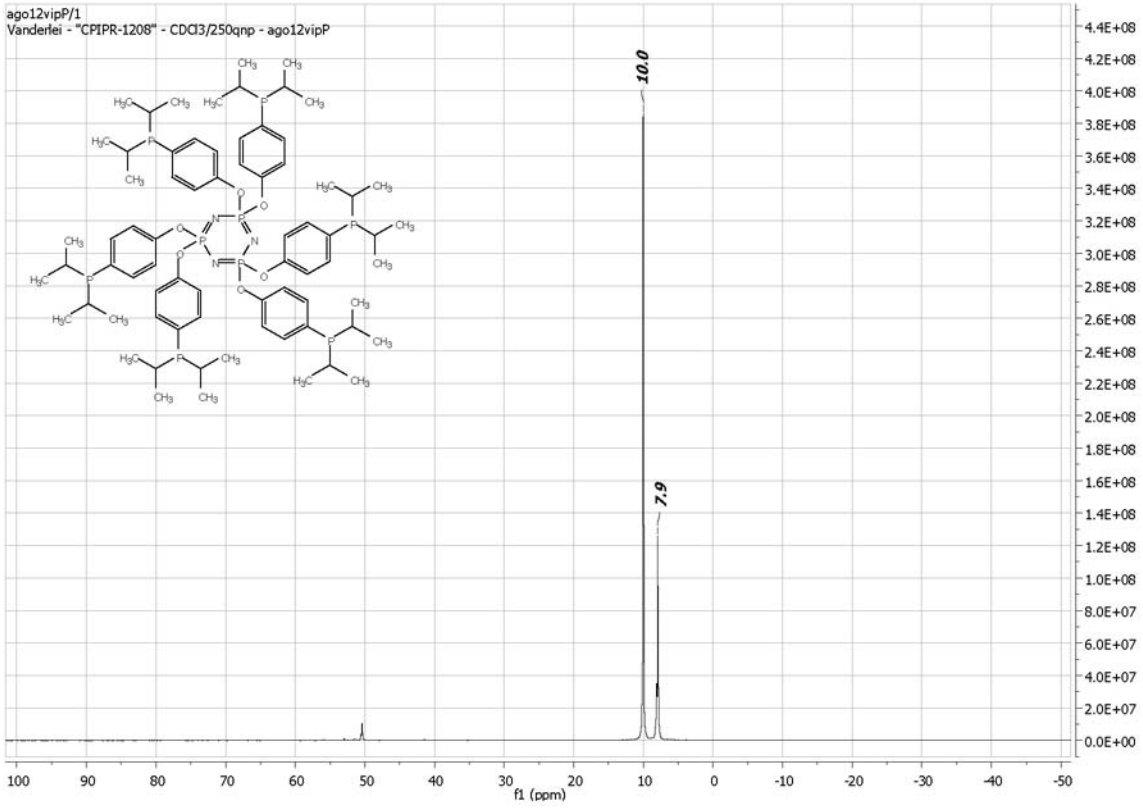

Figure S3. [31]P NMR spectrum of ligand **1b**.

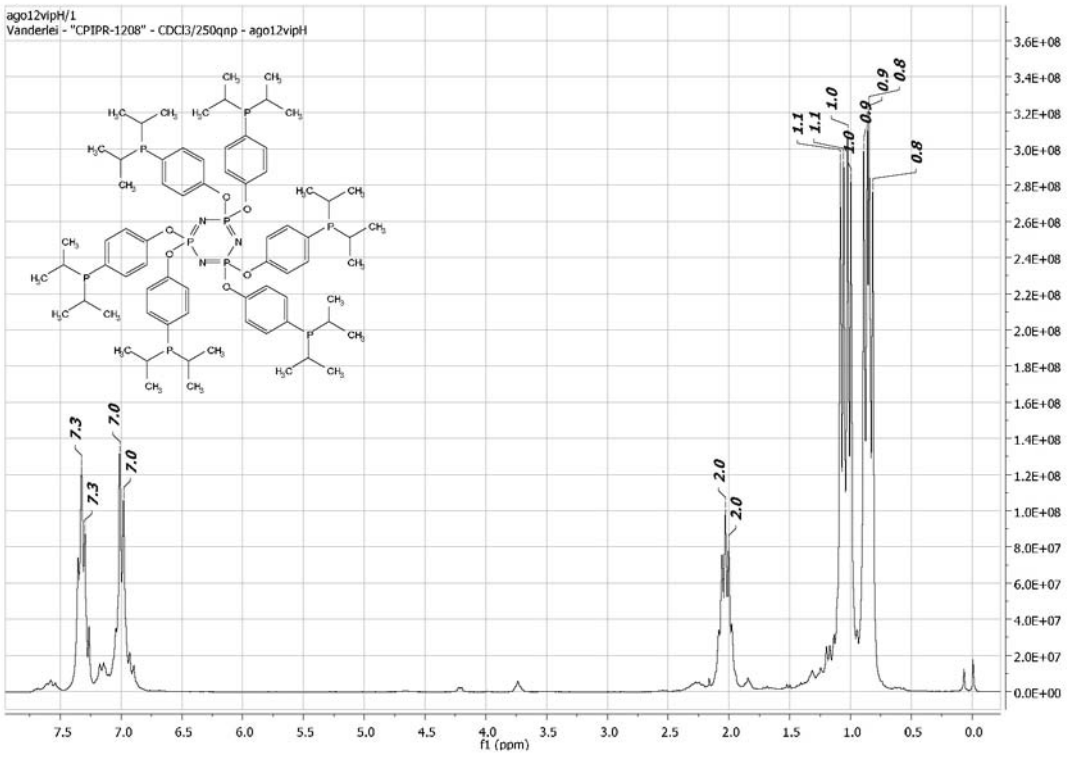

Figure S4. [1]H NMR spectrum of ligand **1b**.

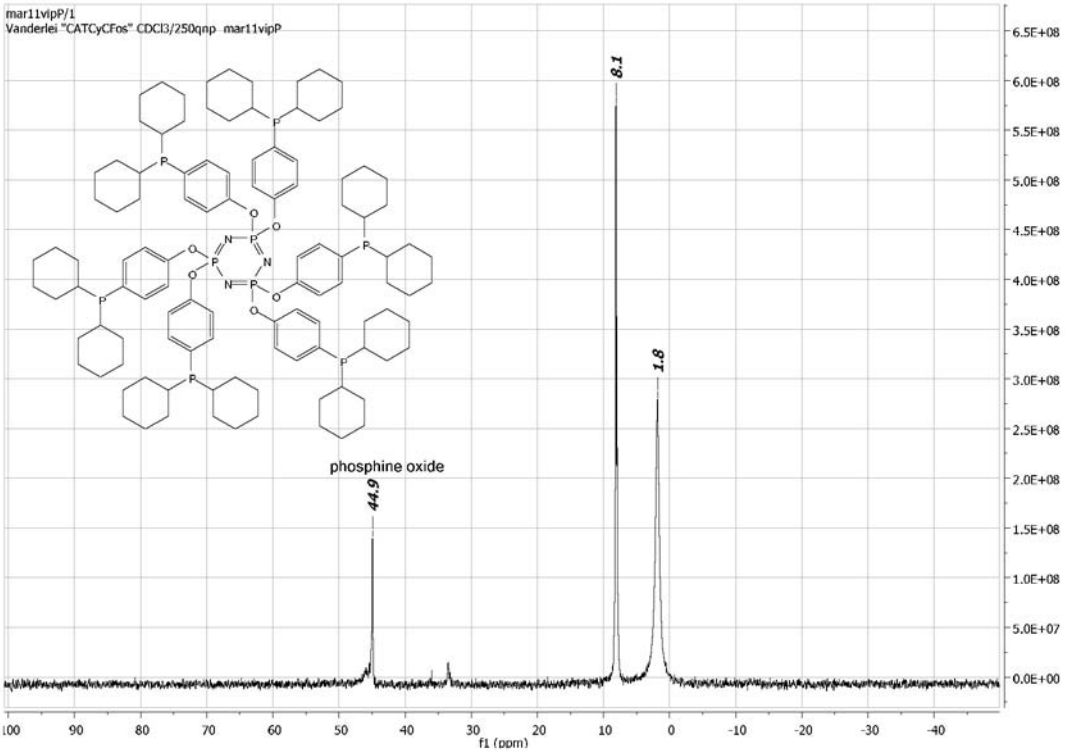

Figure S5. [31]P NMR spectrum of ligand **1c**.

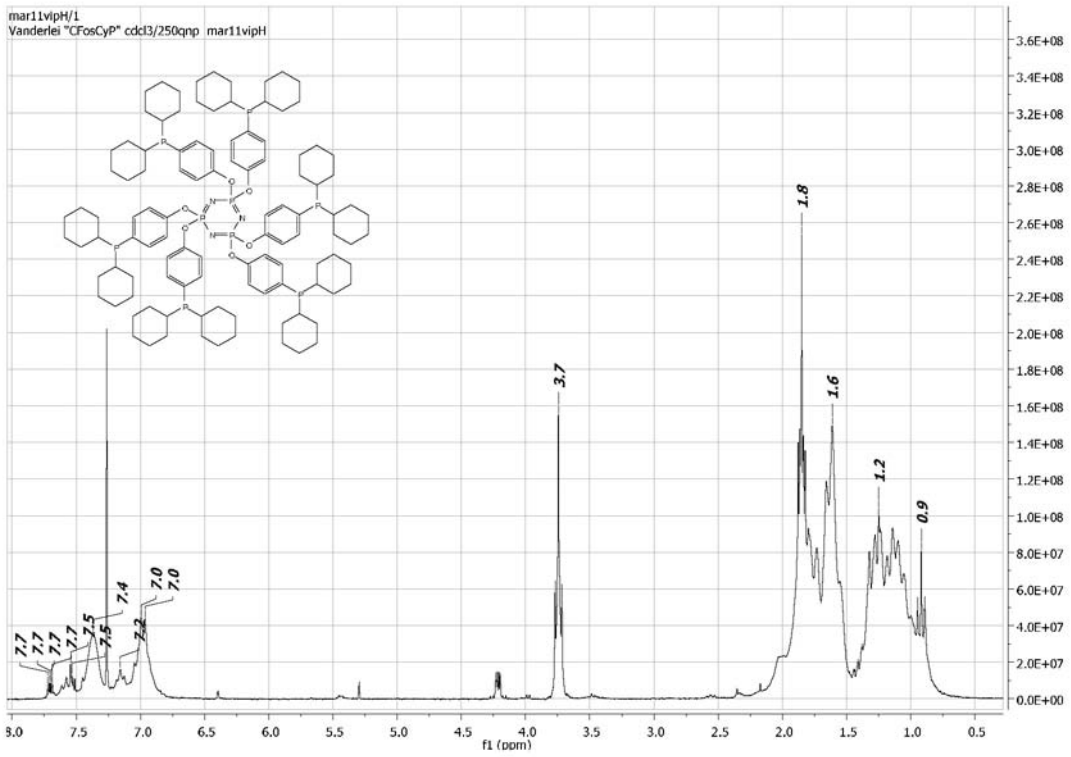

Figure S6. ^{1}H NMR spectrum of ligand **1c**.

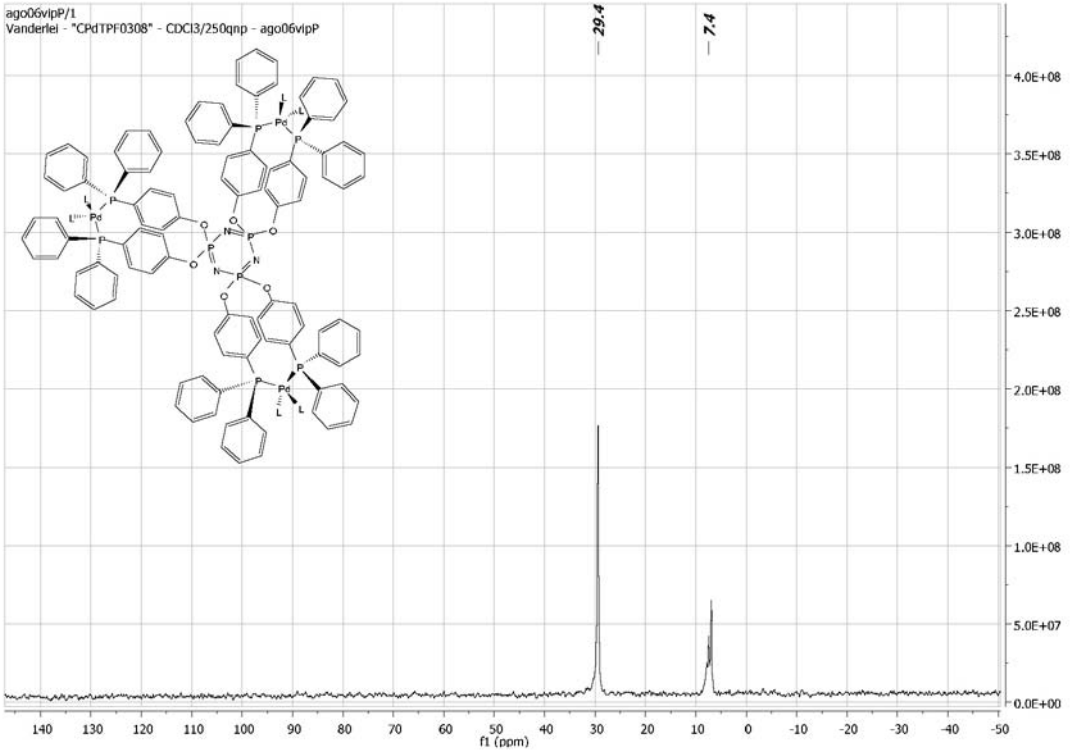

Figure S7. ^{31}P NMR spectrum of complex **1aPd$_3$(dba)**.

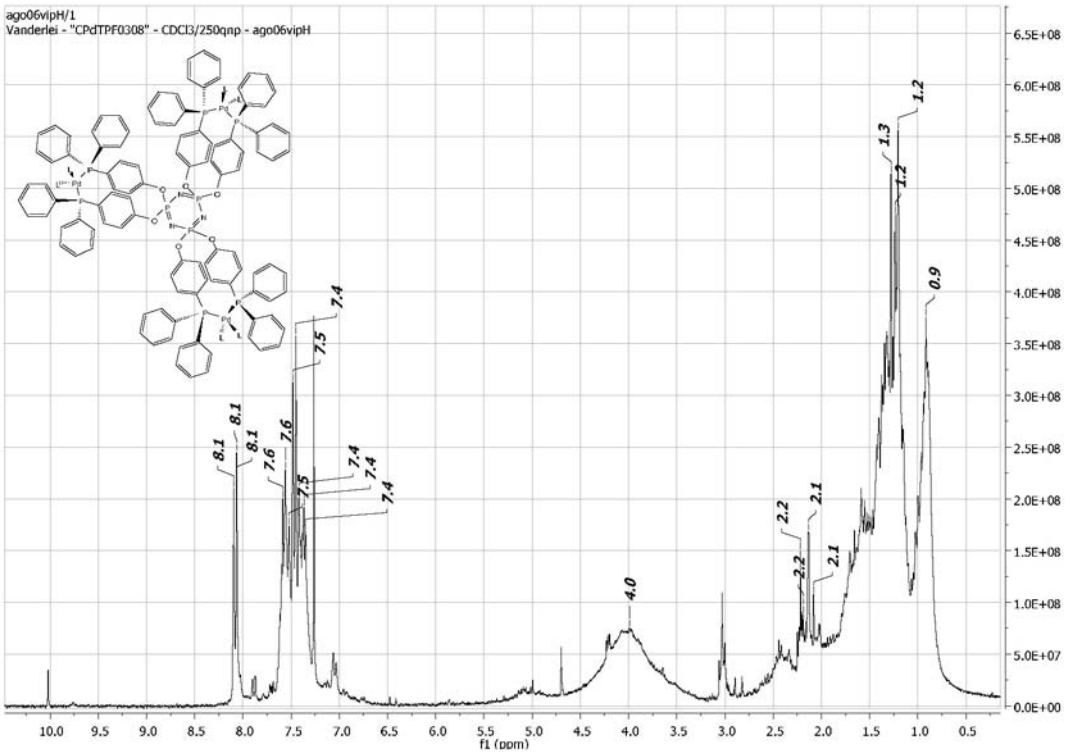

Figure S8. ^{1}H NMR spectrum of complex **1aPd$_3$(dba)**.

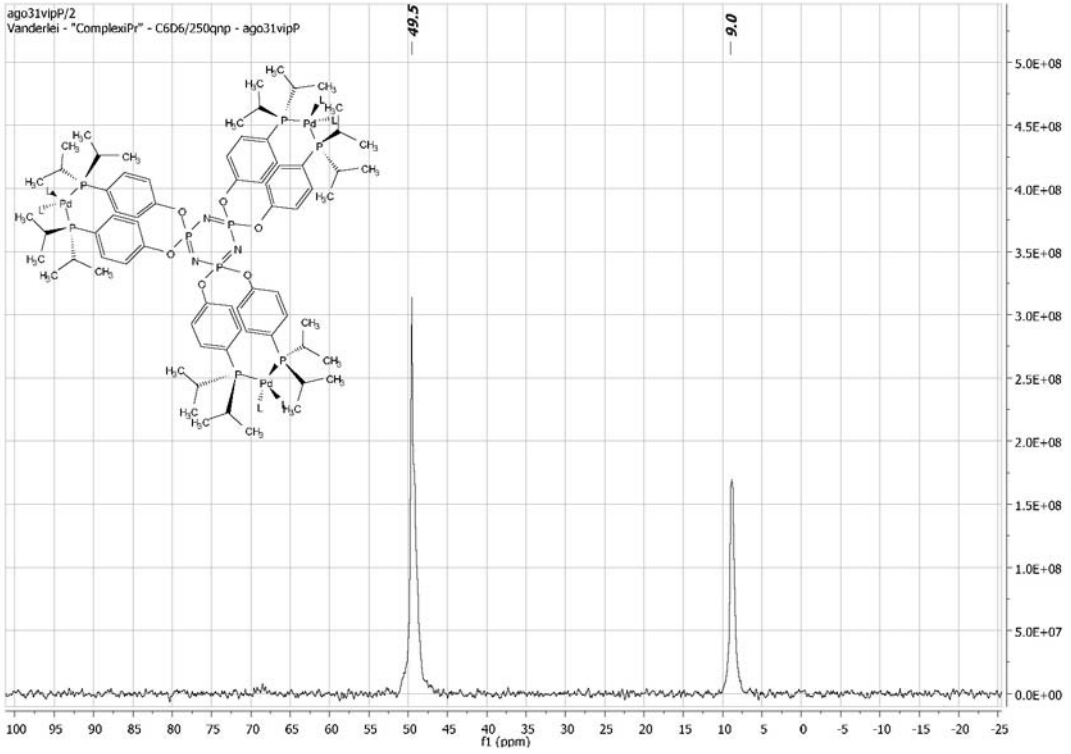

Figure S9. ^{31}P NMR spectrum of complex **1bPd$_3$(dba)$_3$**.

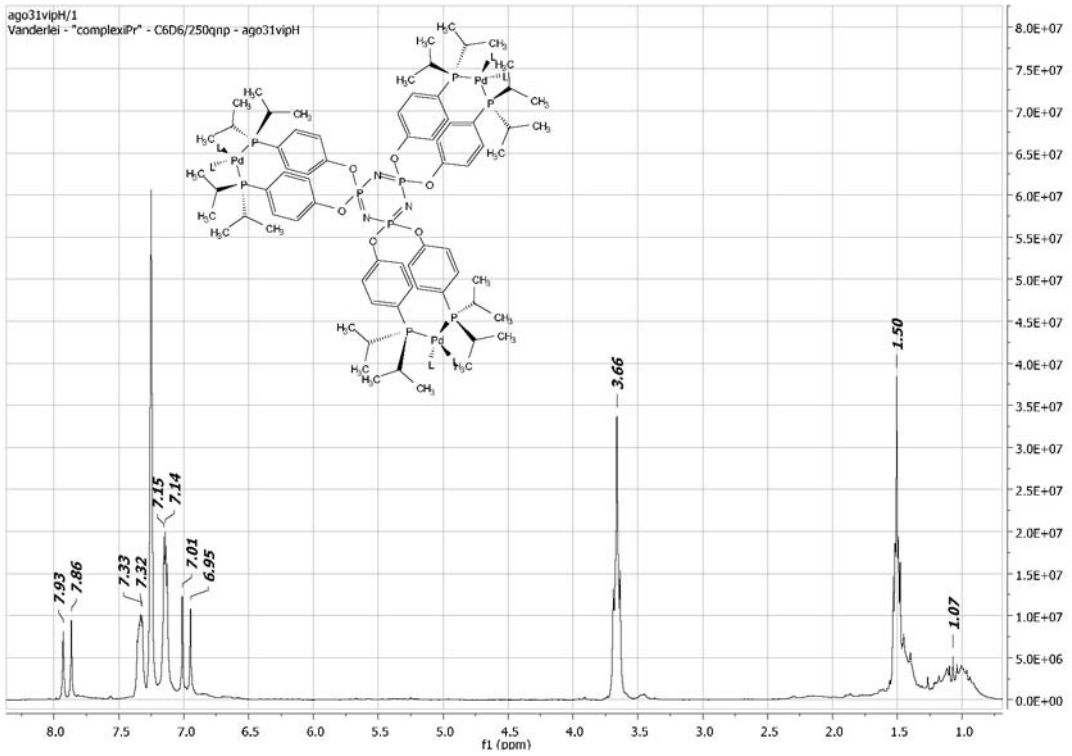

Figure S10. ^{1}H NMR spectrum of complex **1bPd$_3$(dba)$_3$**.

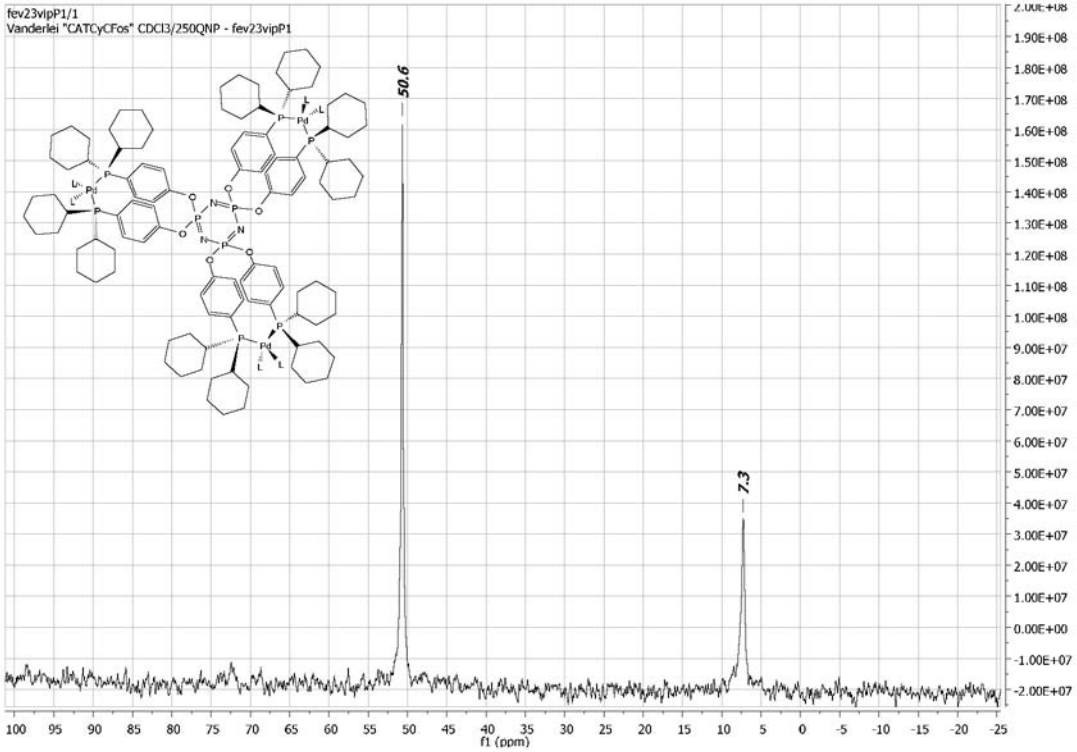

Figure S11. ^{31}P NMR spectrum of complex **1cPd$_3$(dba)**.

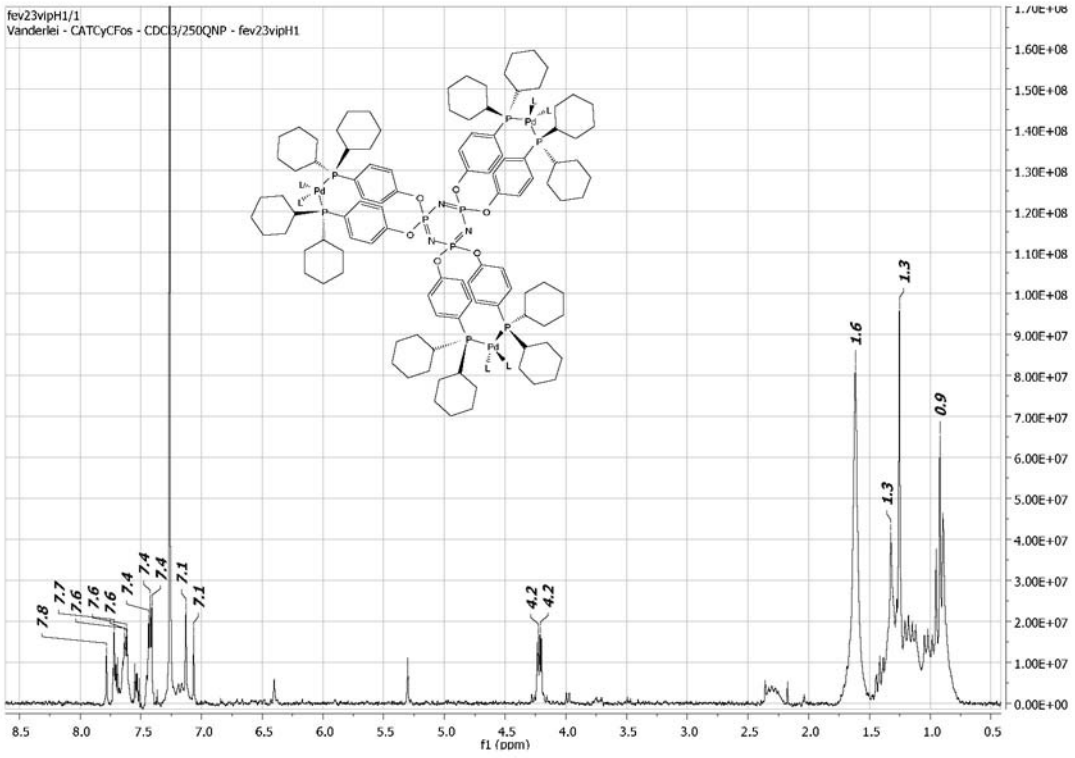

Figure S12. ^{1}H NMR spectrum of complex **1cPd$_3$(dba)**.

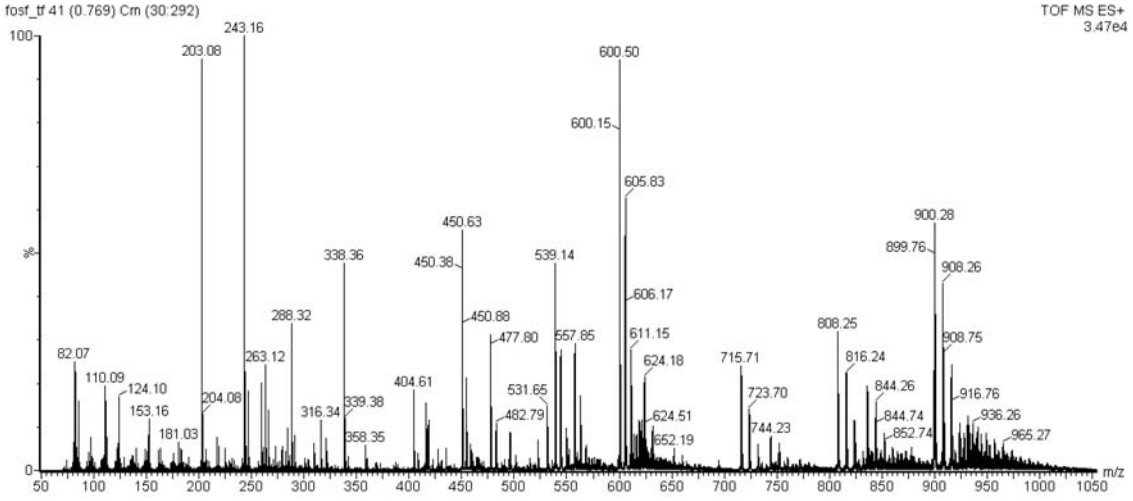

Figure S13. Mass spectrum of ligand **1a**.

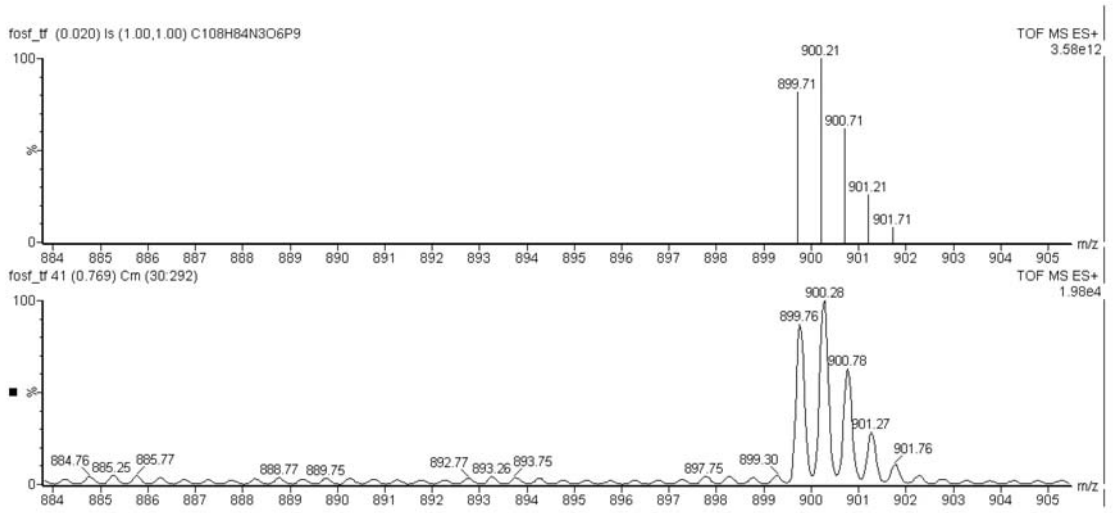

Figure S14. Mass spectrum of the ligand **1a** and isotopic pattern around 900.2.

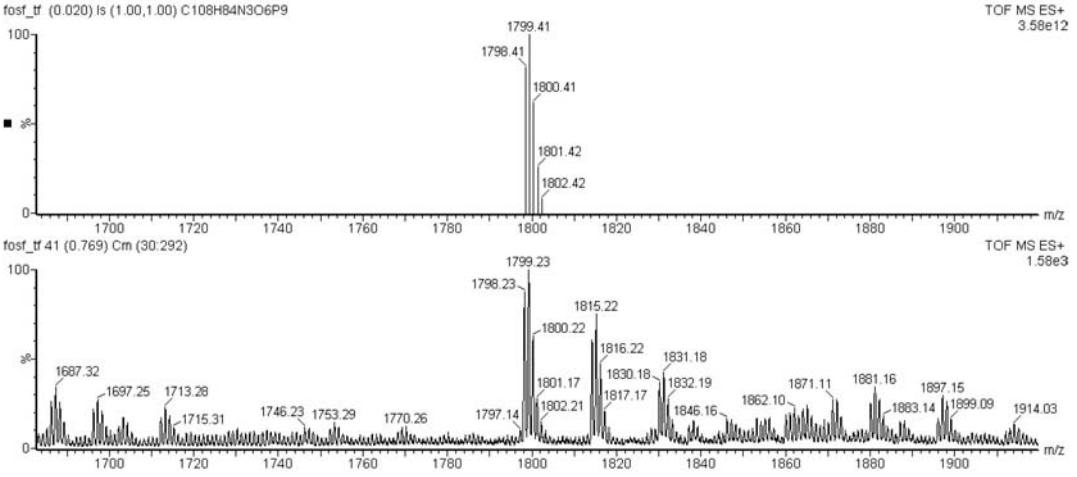

Figure S15. Mass spectrum of ligand **1a** and isotopic pattern around 1799.2.

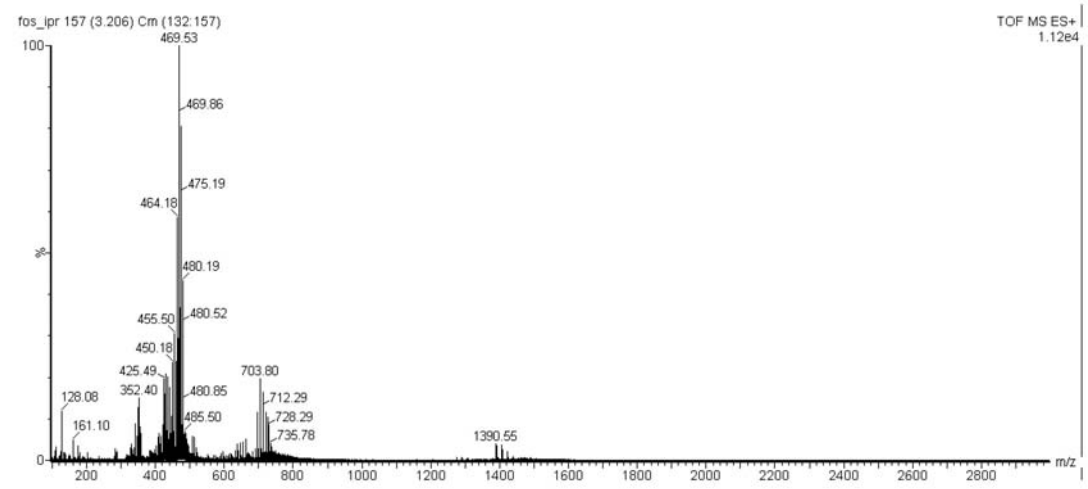

Figure S16. Mass spectrum of ligand **1b**.

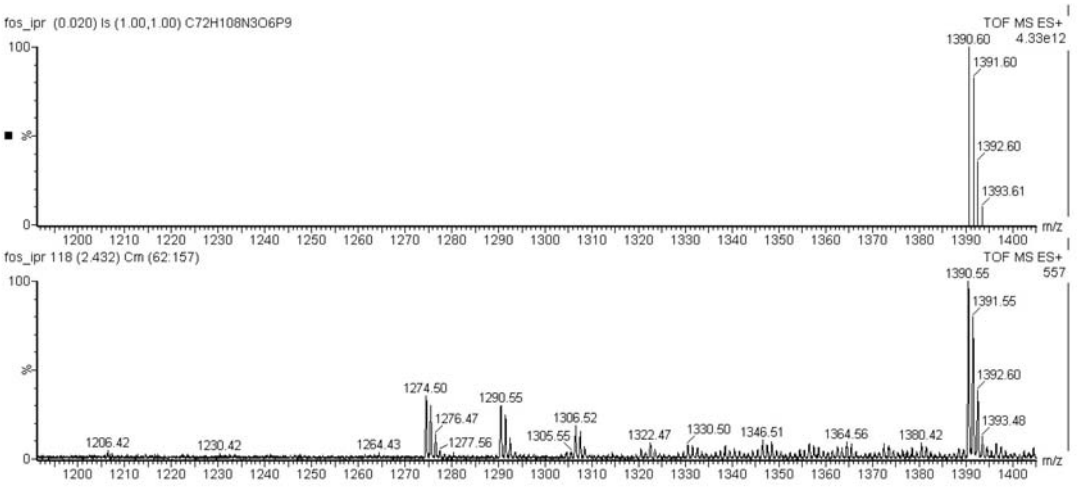

Figure S17. Mass spectrum of ligand **1b** and isotopic pattern around 1390.6.

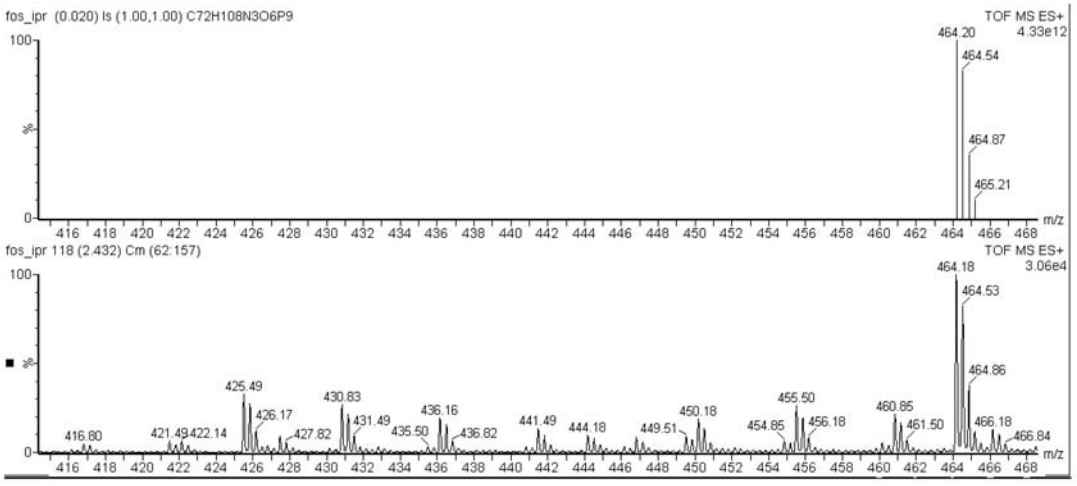

Figure S18. Mass spectrum of ligand **1b** and the isotopic pattern around 464.2 .

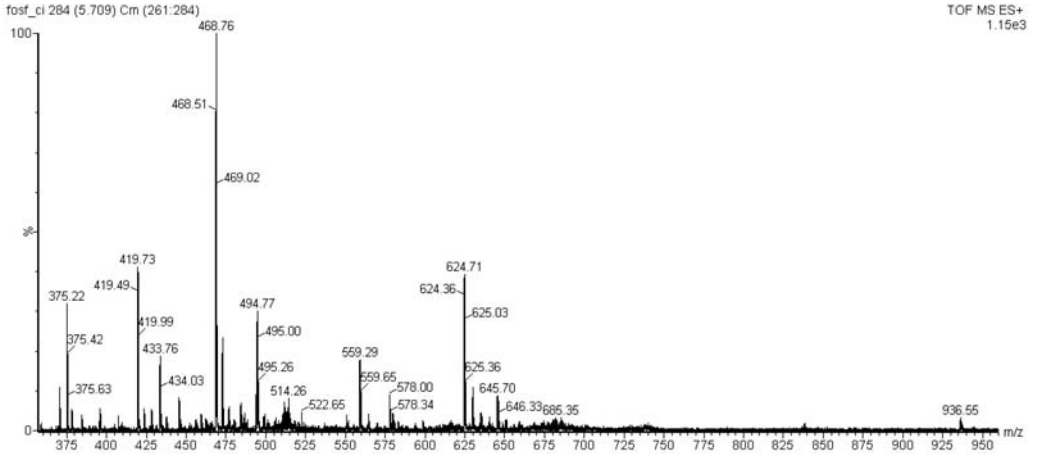

Figure S19. Mass spectrum of ligand **1c**.

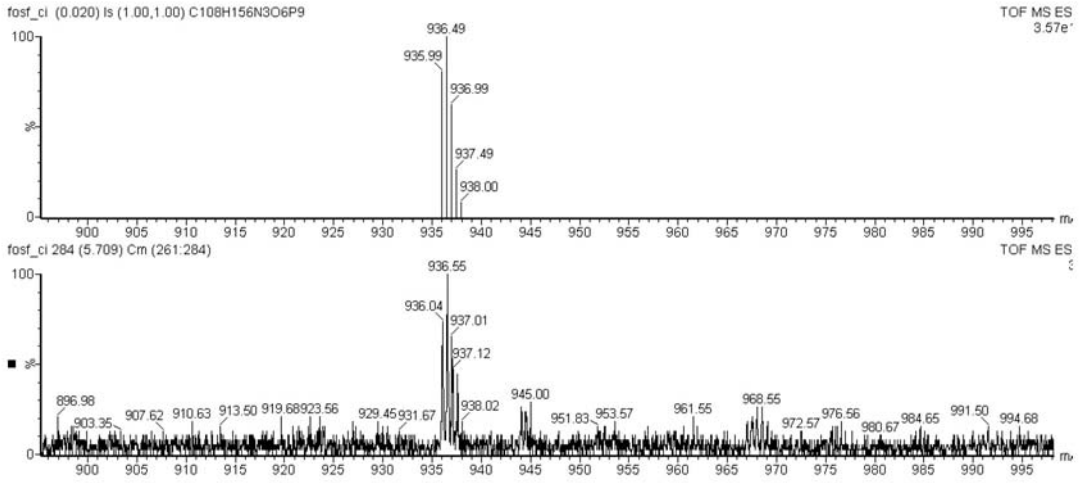

Figure S20. Mass spectrum of ligand **1c** and isotopic pattern around 936.5.

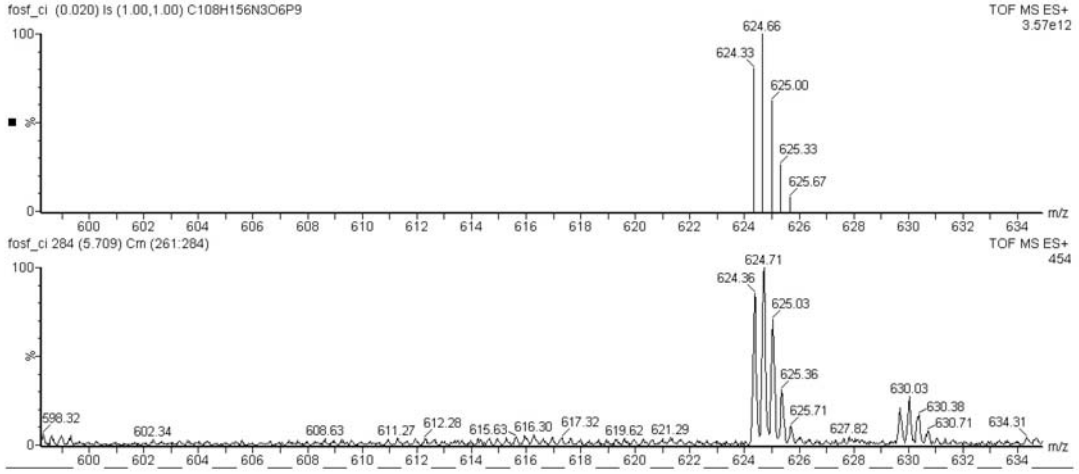

Figure S21. Mass spectrum of ligand **1c** and isotopic pattern around 624.7.

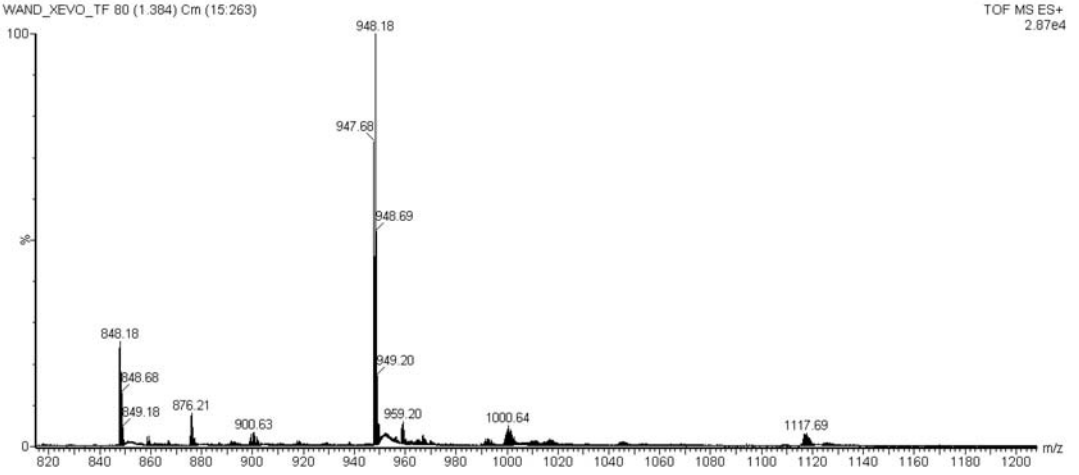

Figure S22. Mass spectrum of complex **1aPd$_3$(dba)**.

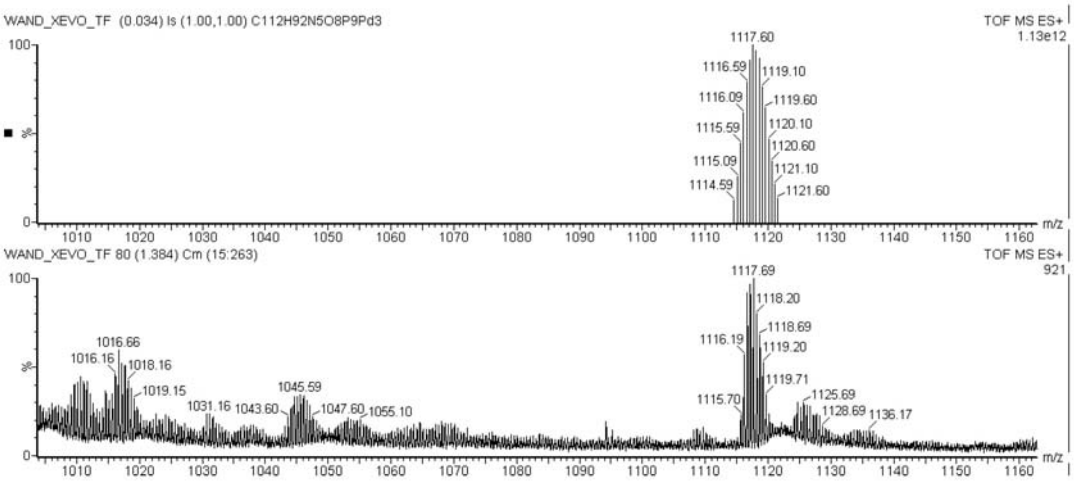

Figure S23. Mass spectrum of complex **1aPd$_3$(dba)** and isotopic pattern around 1117.6.

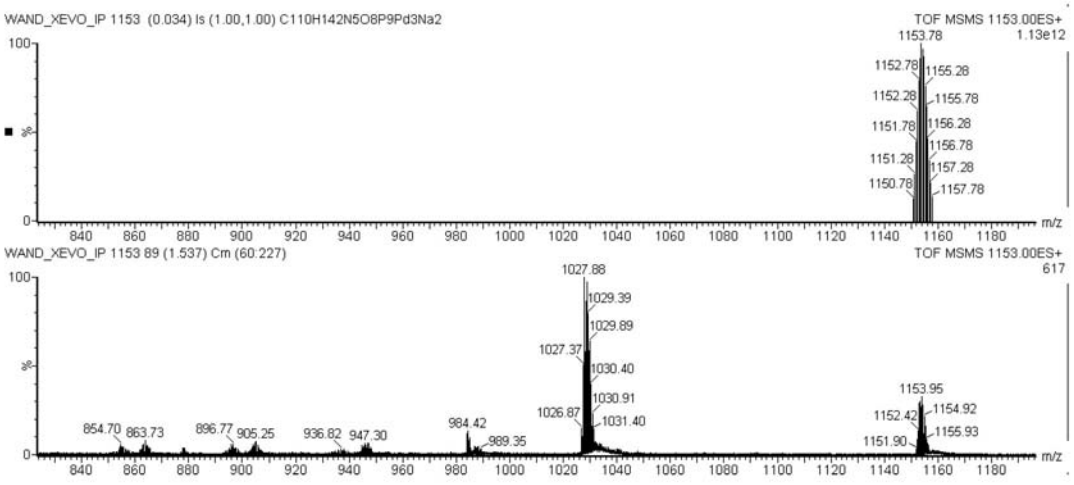

Figure S24. Mass spectrum of complex **1bPd$_3$(dba)$_3$** and isotopic pattern around 1153.

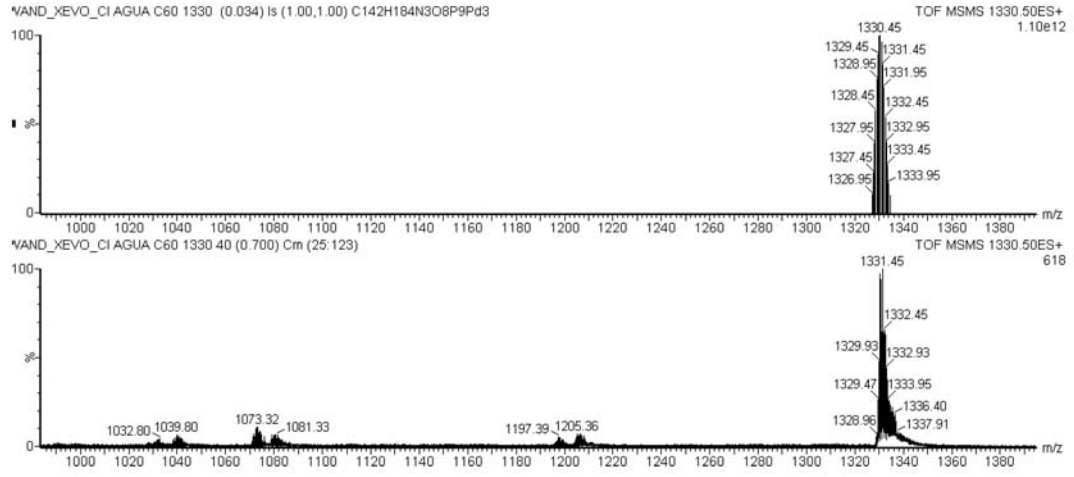

Figure S25. Mass spectrum of complex **1cPd$_3$(dba)** and isotopic pattern around 1330.4.

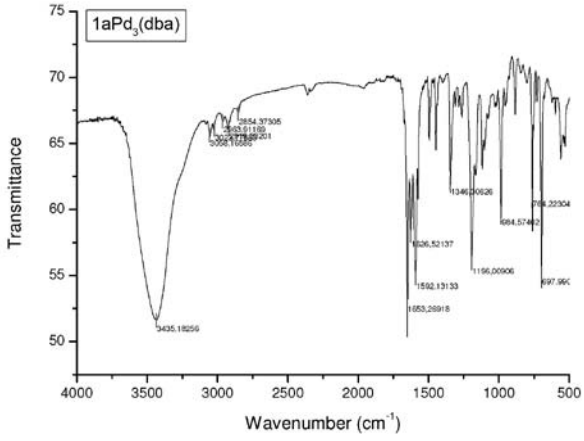

Figure S26. Infrared spectrum of complex **1aPd$_3$(dba)**.

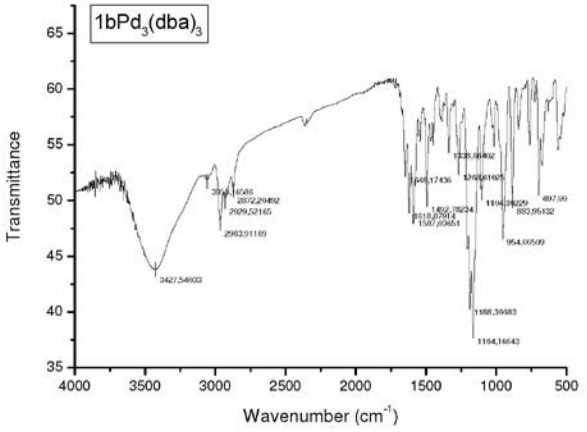

Figure S27. Infrared spectrum of complex **1bPd$_3$(dba)$_3$**.

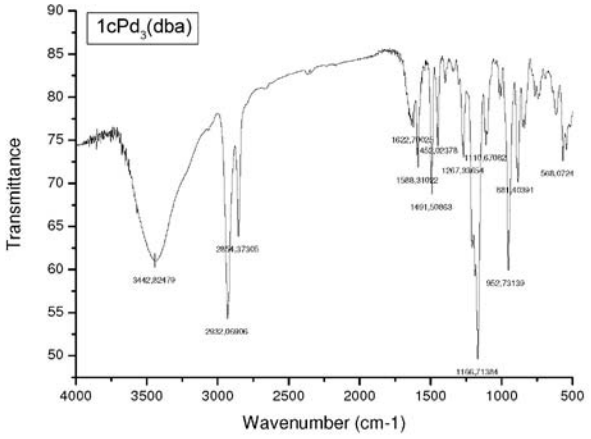

Figure S28. Infrared spectrum of complex **1cPd$_3$(dba)**.

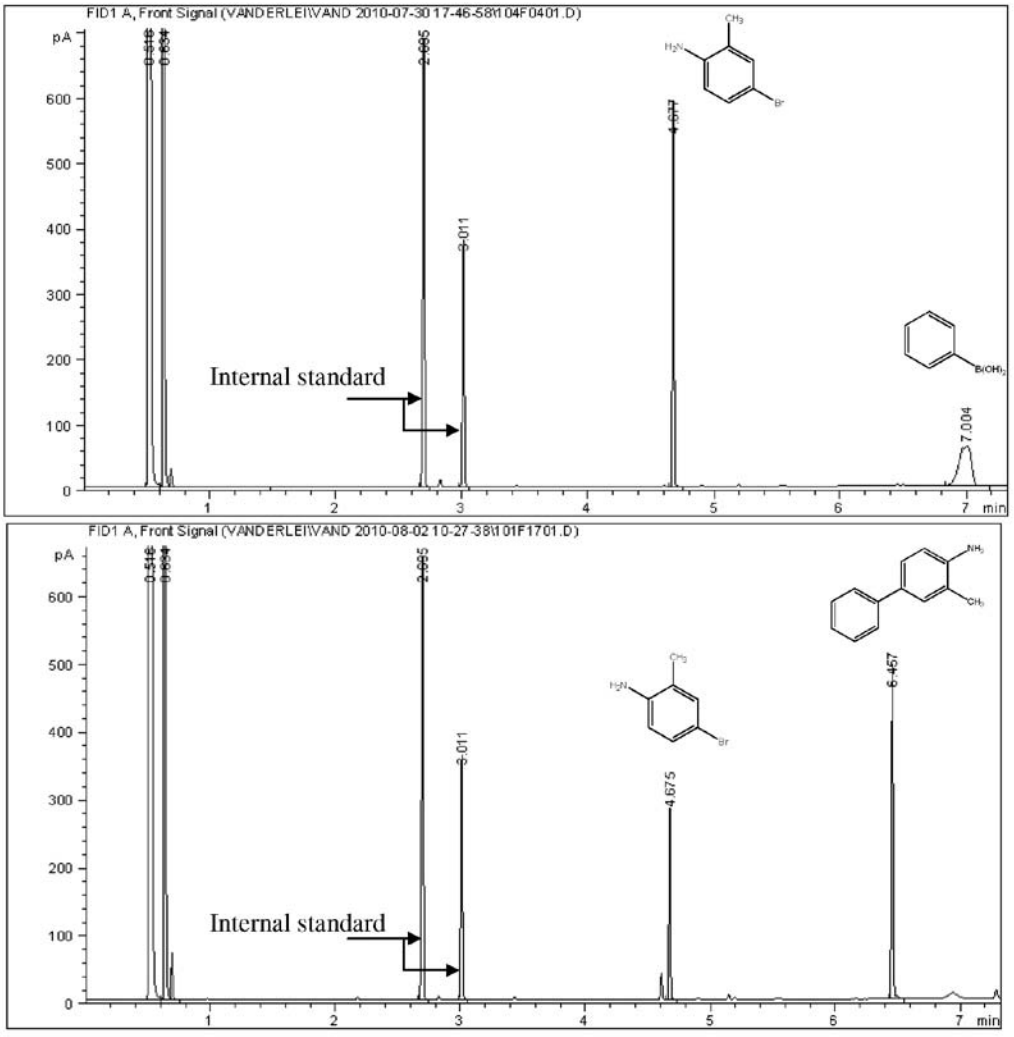

Figure S29. Gas phase chromatograms of the reaction described in Table 2, entry 24 (top: t = 0; bottom: t = 24 h). Peak at 4.60: biphenyl. The peak at 6.457 was assigned to 3-methyl-[1,1'-biphenyl]-4-amine based on its mass spectrum: *m/z* 183.0, 165.0, 151.9, 139.0, 127.9, 16.0, 91.6, 77.0, 63.0, 51.1, 39.9.

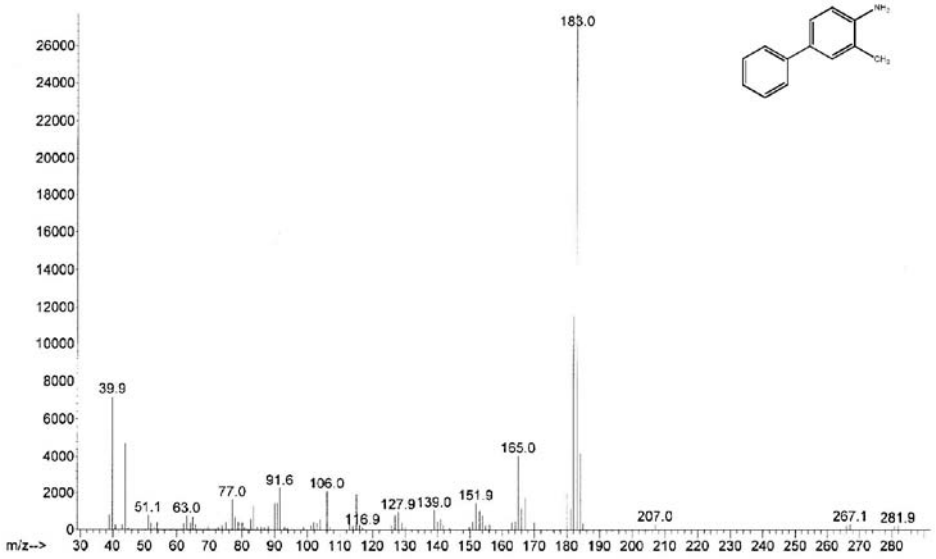

Figure S30. MS spectrum of the product of the reaction described in Table 2, entry 24.

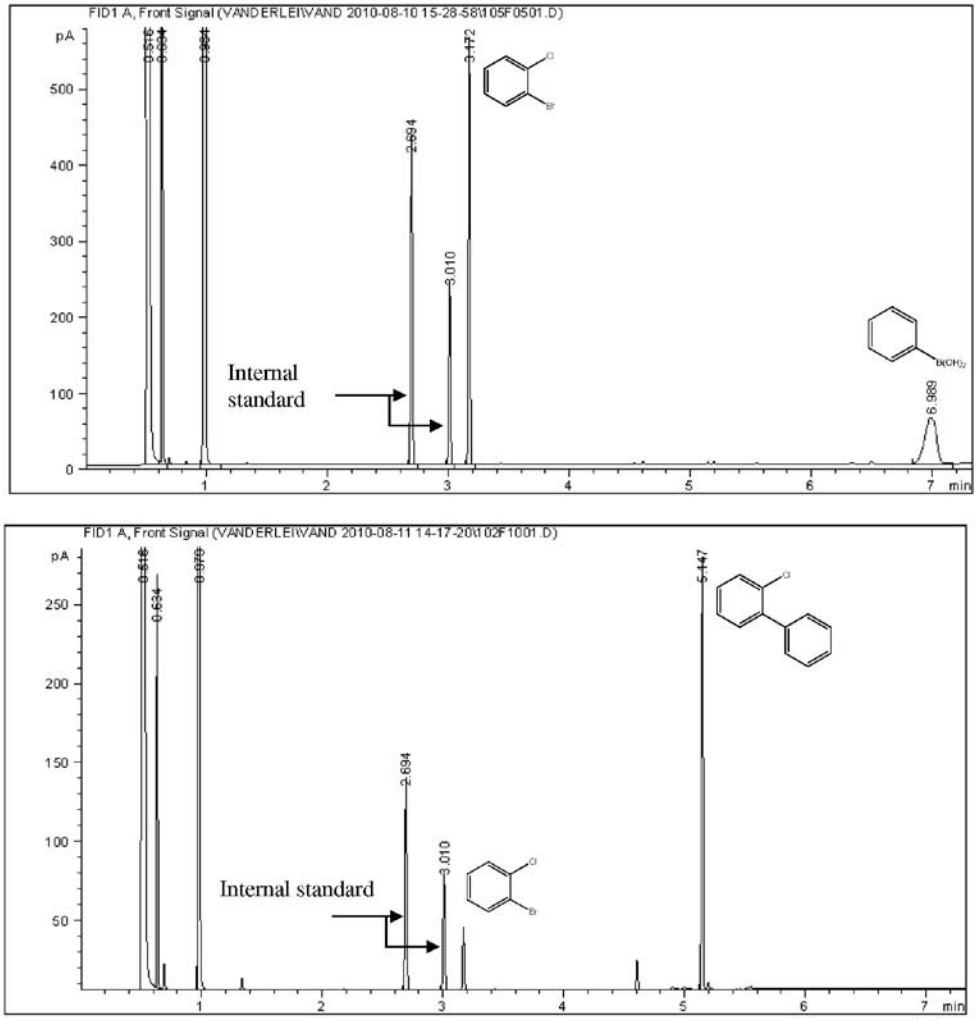

Figure S31. Gas phase chromatograms of the reaction described in Table 3, entry 5 (top: t = 0; bottom: t = 24 h). Peak at 4.60: biphenyl. The peak at 5.147 was assigned to 2-chloro-1,1'-biphenyl based on its mass spectrum (Figure S32). *m/z* 188.0, 152.0, 126.0, 113.0, 103.0, 94.0, 76.0, 63.0, 51.0, 39.1.

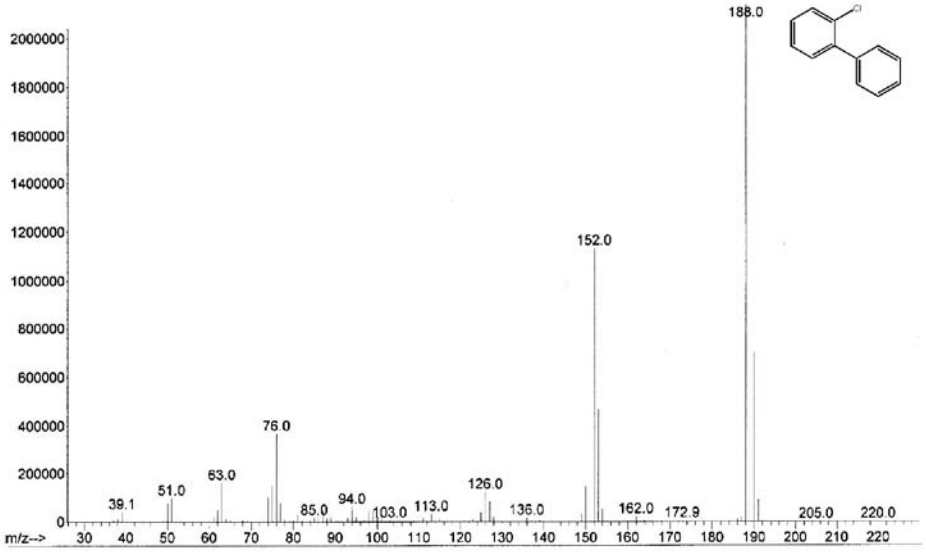

Figure S32. MS spectrum of the product of the reaction described in Table 3, entry 5

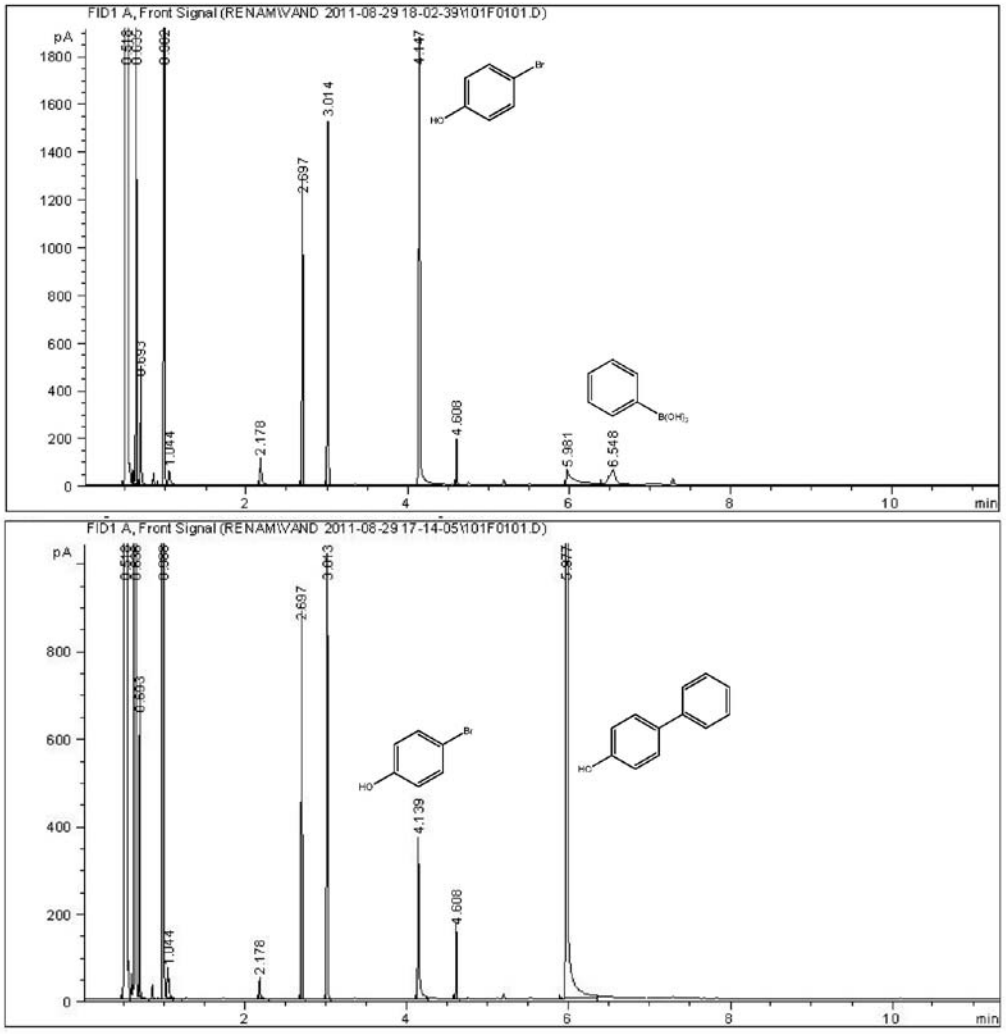

Figure S33. Gas phase chromatograms of the reaction described in Table 3, entry 1(top: t = 0; bottom: t = 24 h). Peak at 4.60: biphenyl.
The peak at 4.610 was assigned to [1,1'-biphenyl]-4-ol based on its mass spectrum (Figure S34): *m/z* 170.0, 151.9, 141.0, 130.9, 115.0, 102.1, 85.0, 77.0, 62.9, 51.1, 40.0.

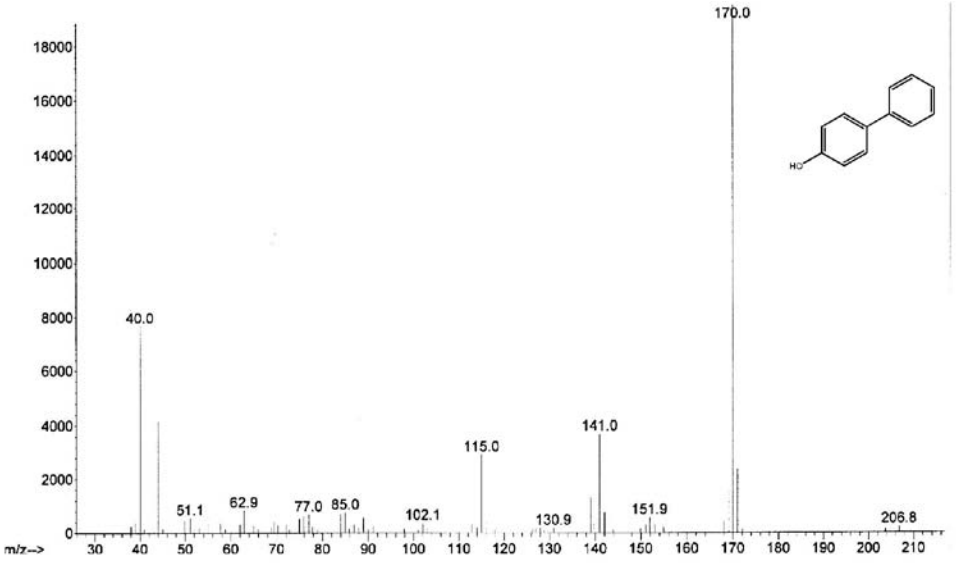

Figure S34. MS spectrum of the product of the reaction described in Table 3, entry 1.

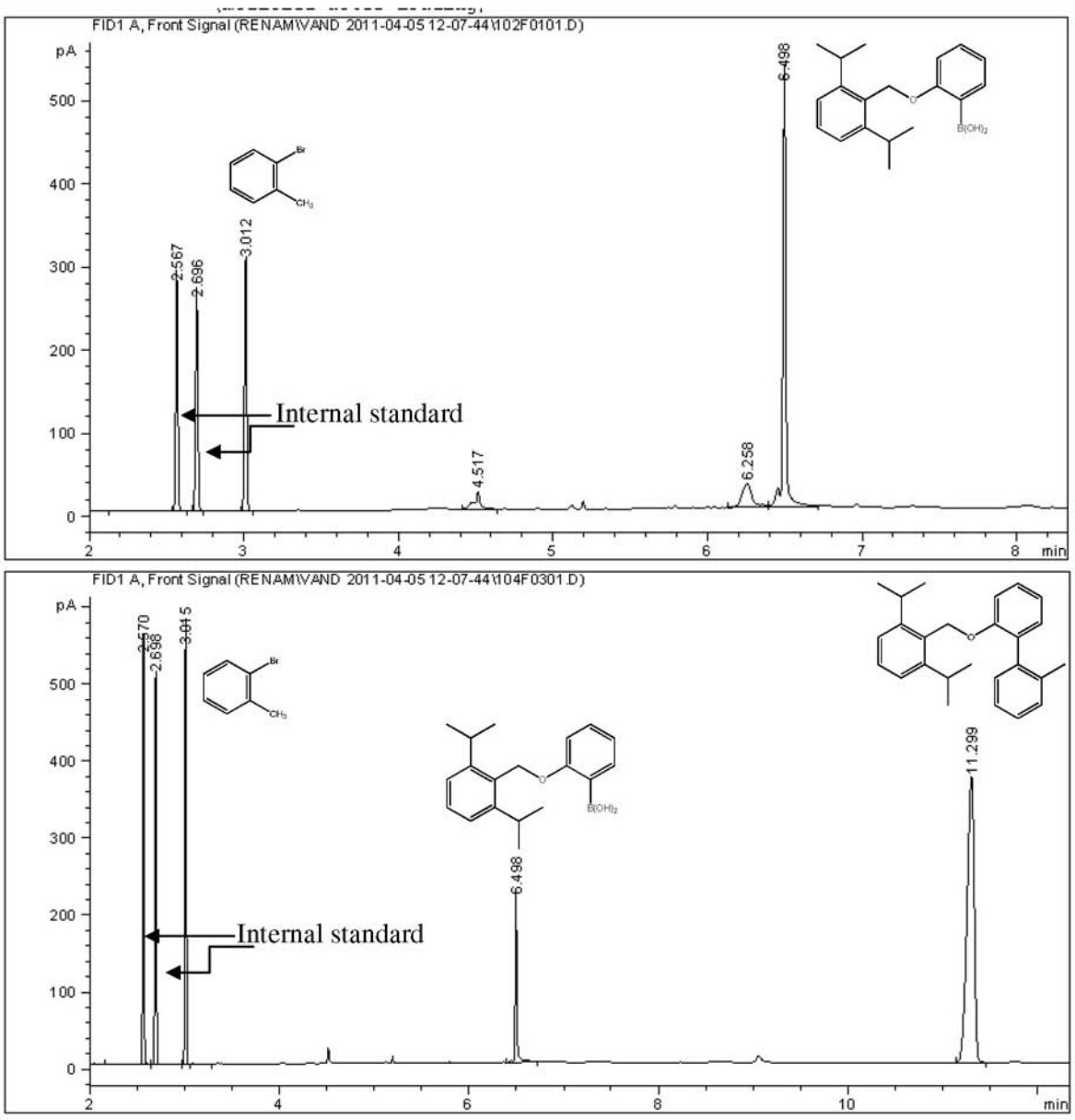

Figure S35. Gas phase chromatograms of the reaction described in Table 4, entry 4 (top: t = 0; bottom: t = 2 h).
The peak at 11.299 was assigned to 2-((2,6-diisopropylbenzyl)oxy)-2'-methyl-1,1'-biphenyl (tr = 11.299), based on its ^{1}H and ^{13}C NMR spectra (Figure S36). ^{1}H NMR (250 MHz, $CDCl_3$) δ 7.83 (d, *J* 7.5 Hz, 1H), 7.45 (dtd, *J* 20.4, 7.5, 1.5 Hz, 3H), 7.31-7.09 (m, 5H), 7.06 (s, 2H), 4.51 (s, 2H), 3.10 (p, *J* 6.9 Hz, 2H), 2.08 (s, 3H), 1.11 (d, *J* 6.9 Hz, 12H). ^{13}C NMR (63 MHz, $CDCl_3$) δ 152.97(s, Ar-O, 1C), 141.93 (s, Ar-Ar, 2C),140.83(s, Ar-Ar, 1C), 140.08 (s, Ar, 1C), 135.94(s, Ar, 1C), 135.46(s, Ar, 1C), 129.88(s, Ar, 1C), 129.54(s, Ar, 1C), 129.35(s, Ar, 1C), 128.37(s, Ar, 1C), 127.7-127.58(m, Ar, 3C), 125.51(s, Ar, 1C), 124.56(s, Ar, 1C), 123.95(s, Ar, 2C), 73.96(s,CH_2, 1C), 26.25(s, CH, 2C), 24.1(s, CH_3, 4C), 19.98(s, CH_3, 1C).

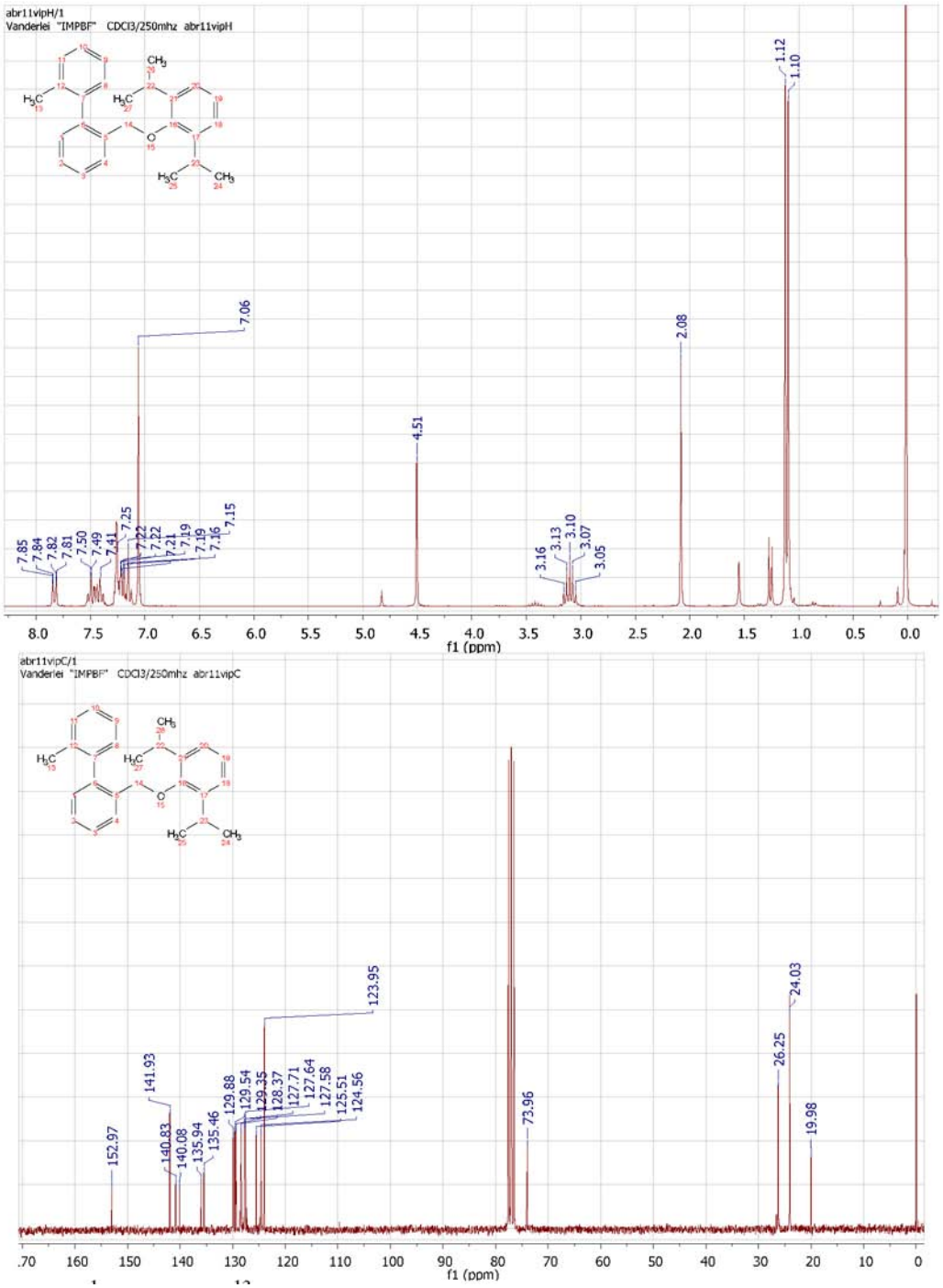

Figure S36. ^{1}H (top) and ^{13}C (bottom) NMR spectra of the product of the reaction described in Table 4, entry 4.