Electronic Supplementary Material (ESI) for RSC Advances.

An efficient, scaleable synthesis of ferrocenylphosphine and dichloroferrocenylphosphine

Brian A. Surgenor, Laurence J. Taylor, Andreas Nordheider, Alexandra M. Z. Slawin, Kasun S. Athukorala Arachchige, J. Derek Woollins and Petr Kilian*

Electronic Supplementary Information

General Synthetic Methods

All experiments were carried out in standard Schlenk glassware under an inert atmosphere or in a glove box unless otherwise stated. Solvents were dried on an MBraun solvent purification system and stored over molecular sieves prior to use. All reagents were purchased from commercial suppliers. All NMR spectra were recorded using a JEOL GSX Delta 270, a Bruker Avance 300 or Bruker Avance 400 spectrometer at room temperature. Chemical shifts are referenced to 85% H_3PO_4 (^{31}P) or TMS (^{1}H and ^{13}C). Measurements were performed at 25 °C unless otherwise indicated. All IR spectra were obtained in the range 4000–400 cm^{-1} on a Perkin-Elmer System 2000 NIR Fourier transform spectrometer. Elemental analysis (C, H and N) was performed by Mr Stephen Boyer at London Metropolitan University.

X-ray Experimental

Data were collected using a Rigaku Mercury70 diffractometer with Mo Kα radiation (λ = 0.71075 Å) at −180(2) °C ($FcPH_2$) and −100(1) °C ($FcPCl_2$). Intensities were corrected for Lorentz polarization and for absorption. The structures were solved by direct methods. Refinements were done by full-matrix least-squares based on F^2 using SHELXTL.[1] The PH_2 hydrogen atoms in $FcPH_2$ were refined isotropically subject to distance constraint. The rest of hydrogen atoms were refined using a riding model with idealised positions. CCDC 812314 and 1437271 contain the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/getstructures.

NMR spectra of $FcPH_2$ and $FcPCl_2$

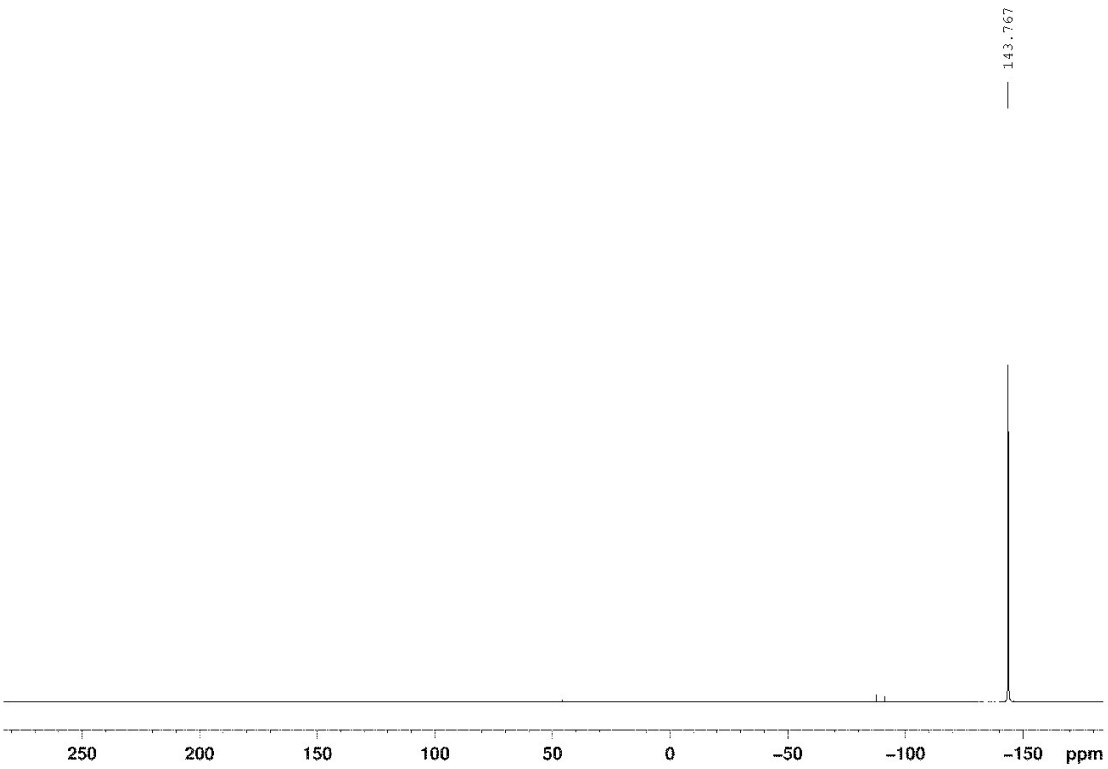

Figure 1: $^{31}P\{^{1}H\}$ NMR of $FcPH_2$ (109 MHz; $CDCl_3$)

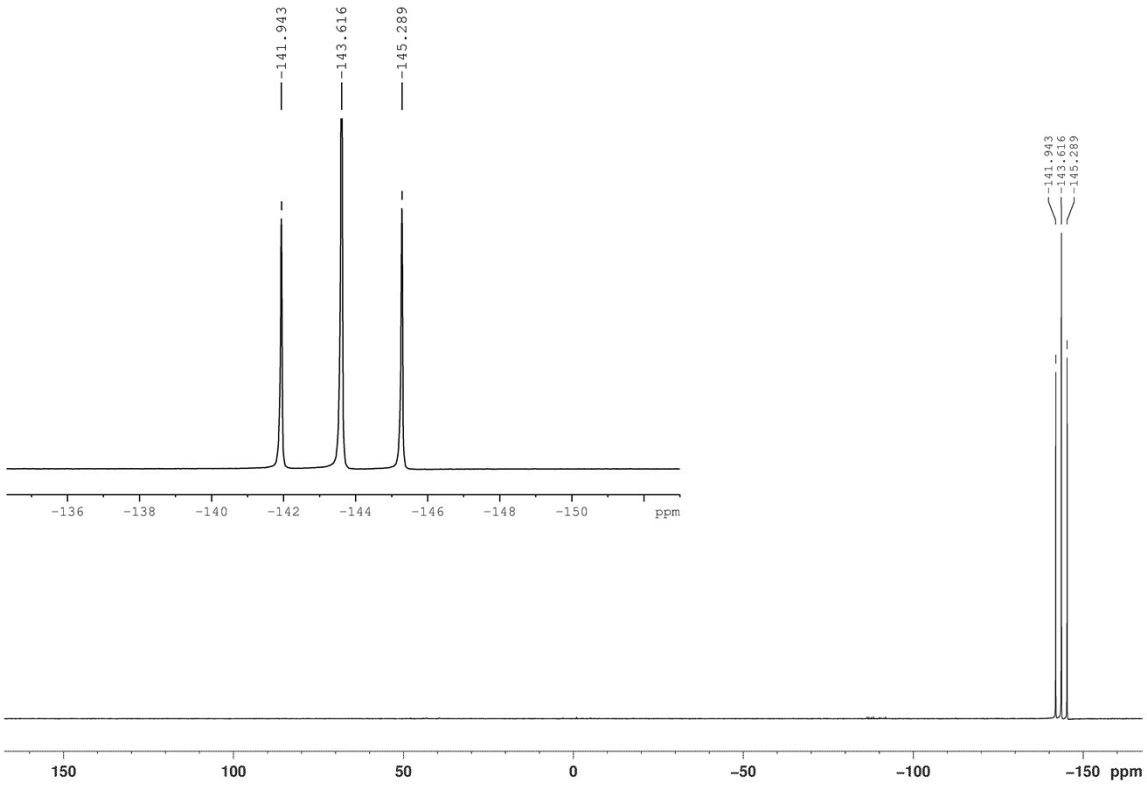

Figure 2: ^{31}P NMR of $FcPH_2$ (121 MHz; $CDCl_3$)

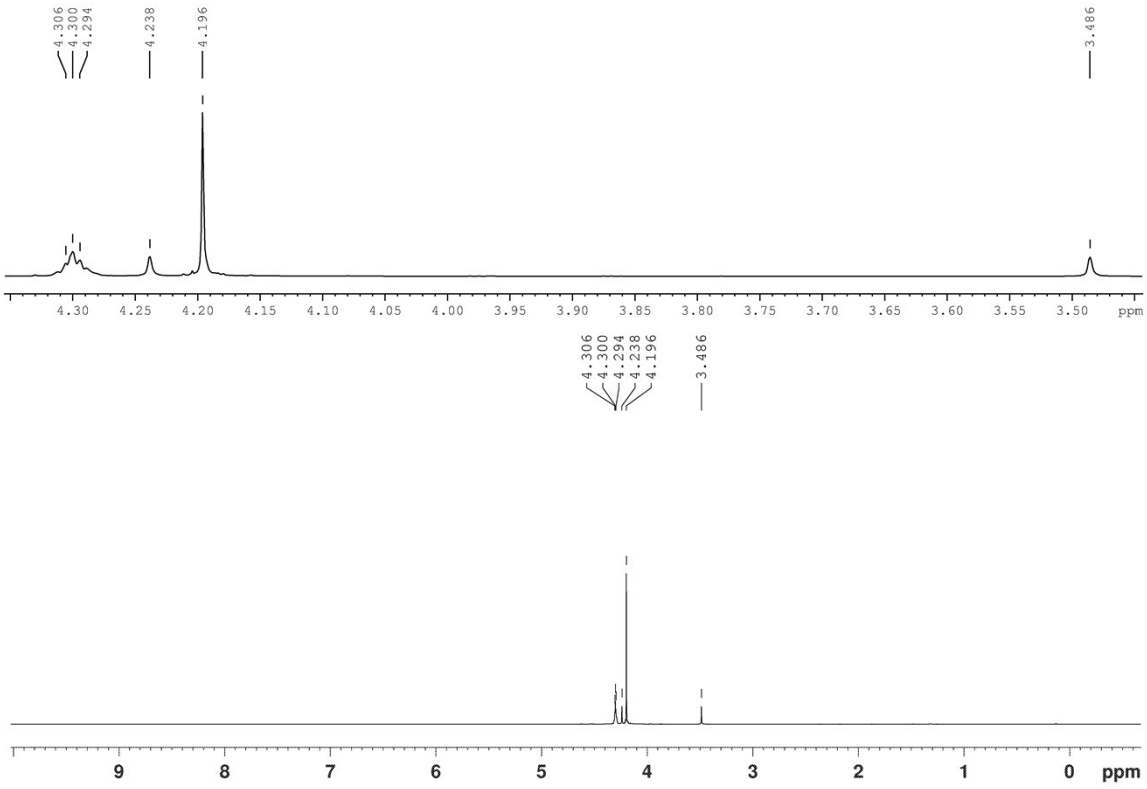

Figure 3: ^{1}H NMR of $FcPH_2$ (270 MHz; $CDCl_3$)

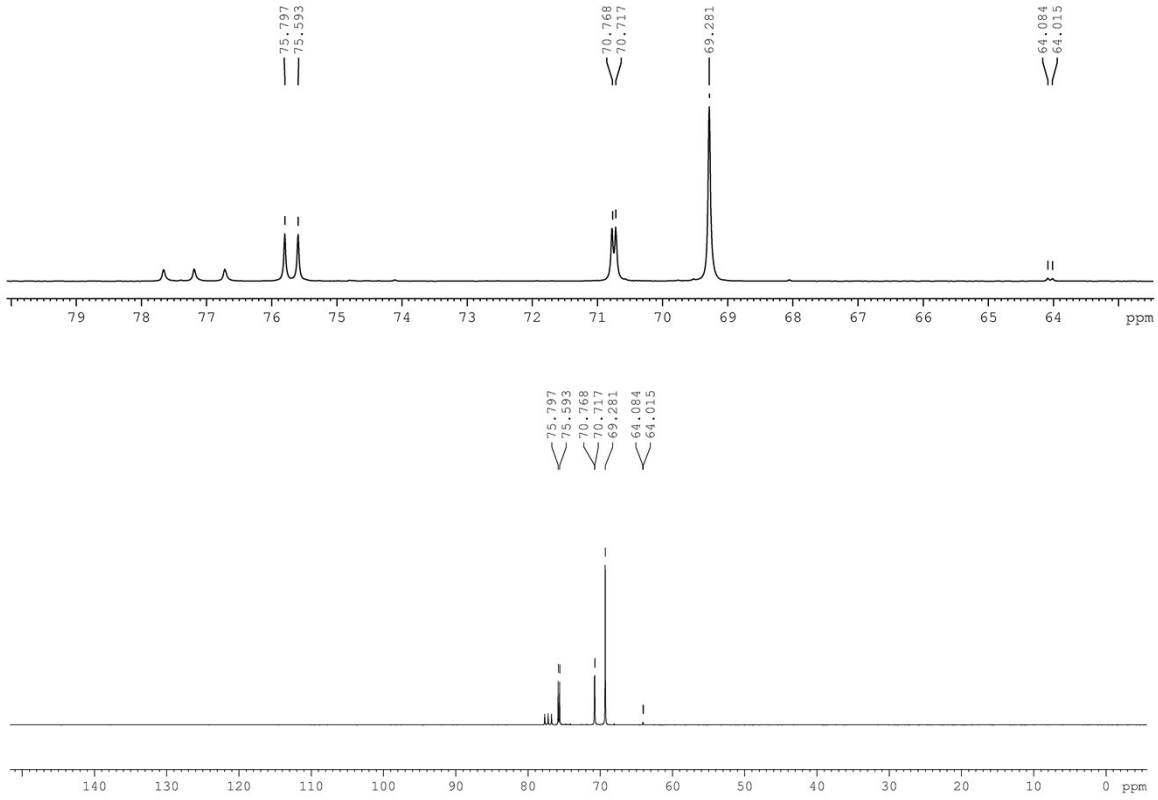

Figure 4: $^{13}C\{^{1}H\}$ NMR of $FcPH_2$ (75 MHz; $CDCl_3$)

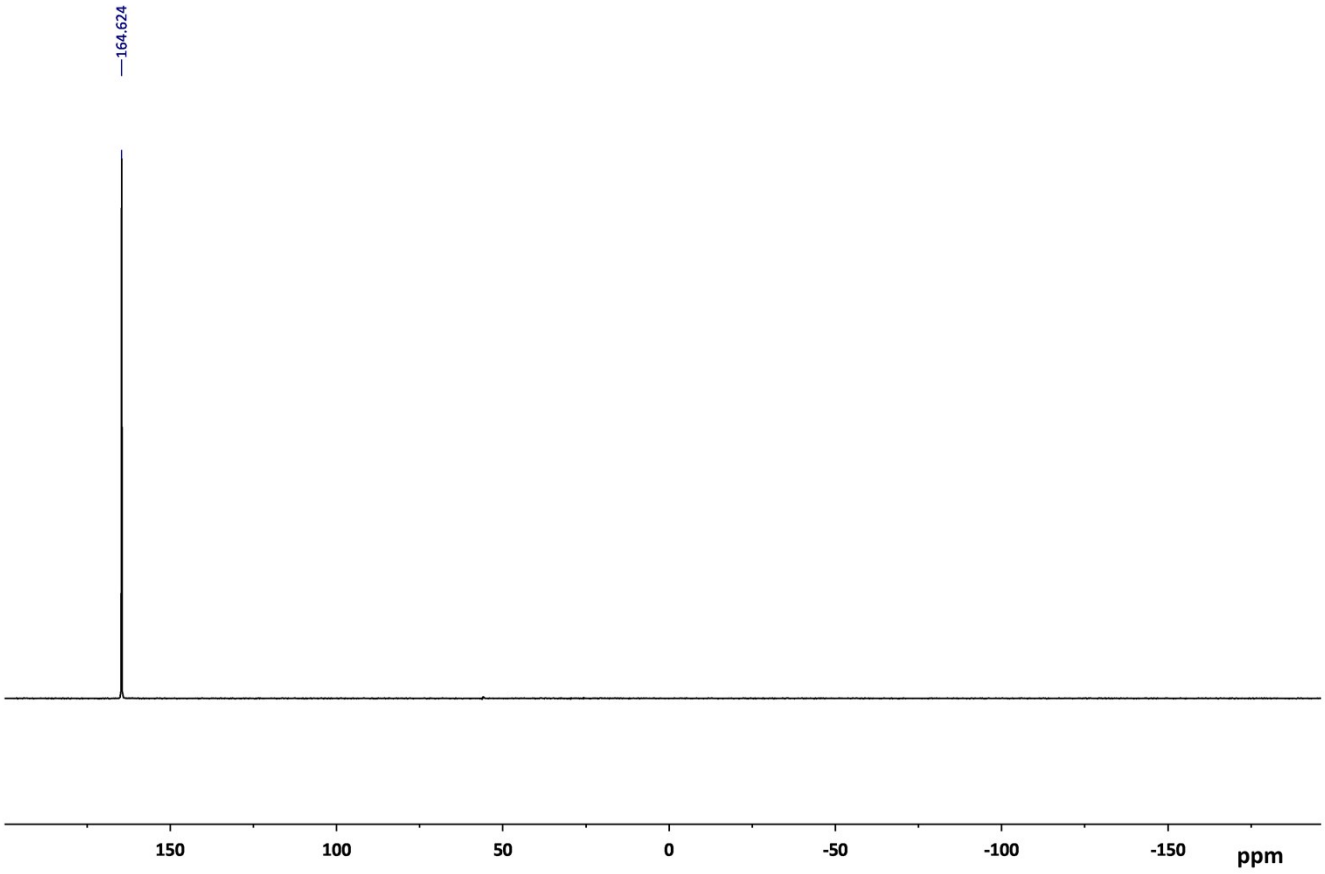

Figure 5: ^{31}P NMR of $FcPCl_2$ (162MHz; $CDCl_3$)

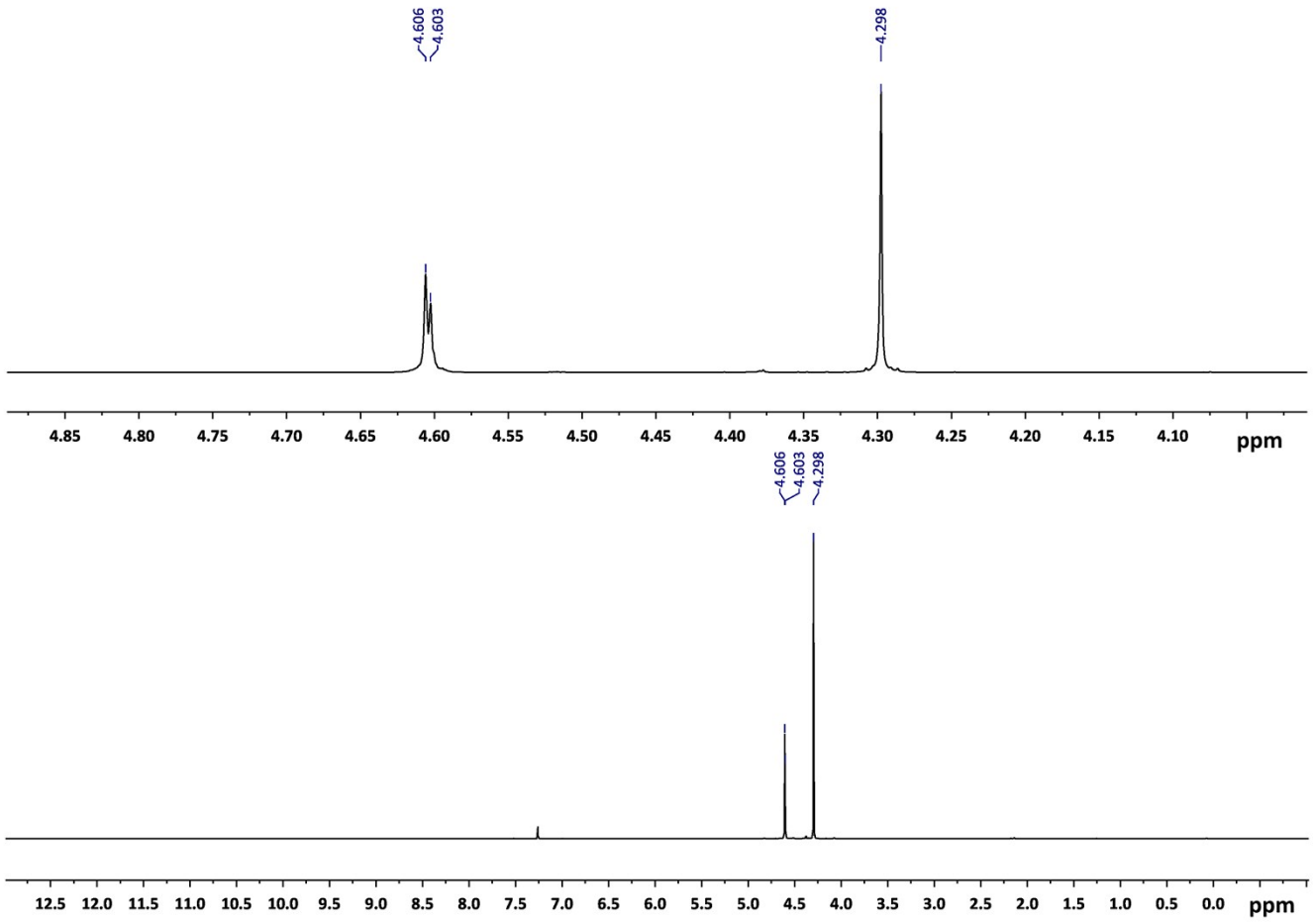

Figure 6: ^{1}H NMR of $FcPCl_2$ (400 MHz; $CDCl_3$)

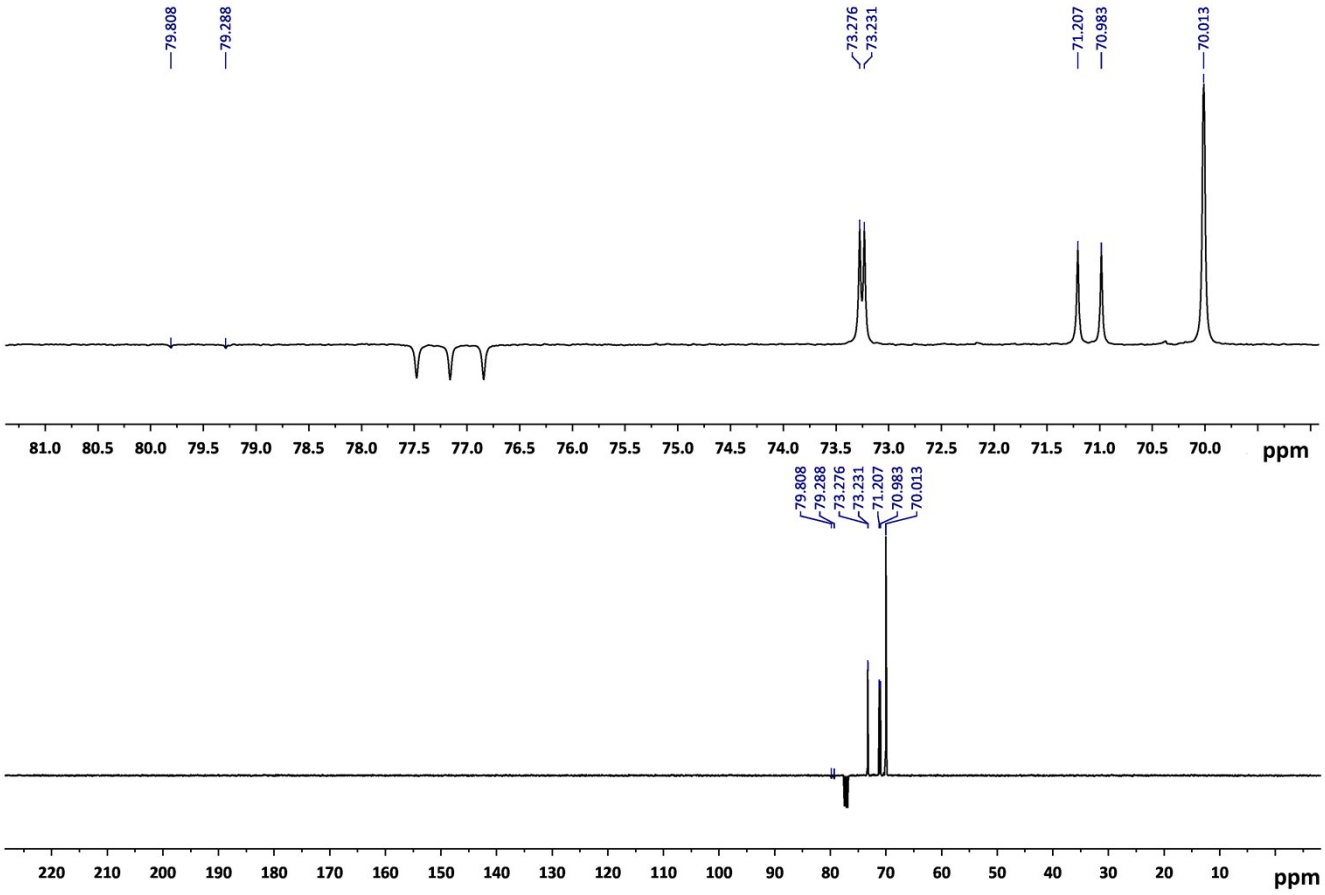

Figure 7: $^{13}C\{^{1}H\}$ NMR of $FcPCl_2$ (101 MHz; $CDCl_3$)

References

1. G. M. Sheldrick, *Acta Crystallogr., Sect. A*, 2008, **A64**, 112–122.