Electronic Supplementary Information

Ag-mediated cascade decarboxylative coupling and annulation: A convenient route to 2-Phosphinobenzo[*b*]phosphole Oxides

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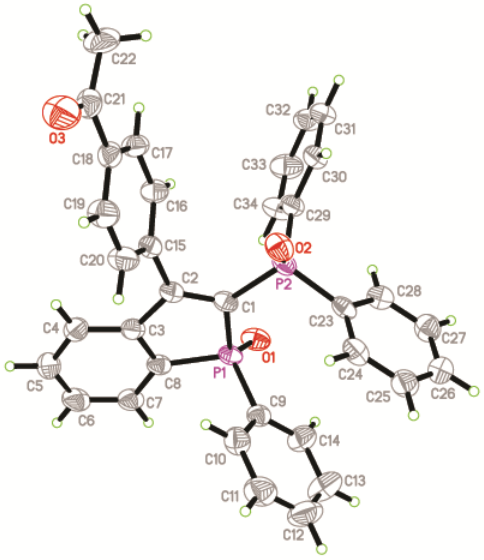
Crystallographic detail of 3m:

1-(4-(2-(diphenylphosphoryl)-1-oxido-1-phenyl-1H-phosphindol-3-yl)phenyl)ethanone

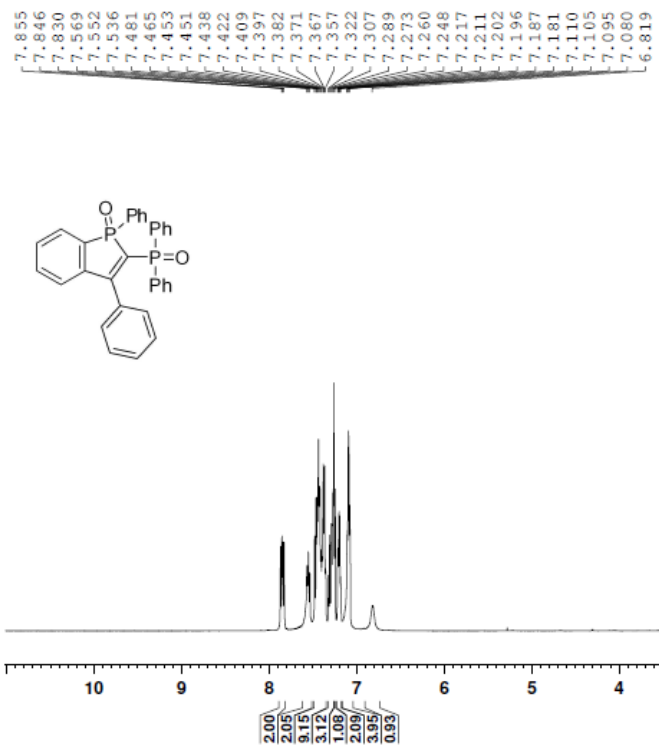


Single crystal X-ray structure of compound **3m**, showing 50% probability displacement ellipsoids (arbitrary spheres for H atoms). The compound **3m** crystallizes with two molecules in the asymmetric unit. Compound **3m** crystallizes in the triclinic P -1 space group with two molecules per unit cell. There is one independent molecule in the asymmetric unit of **3m**.

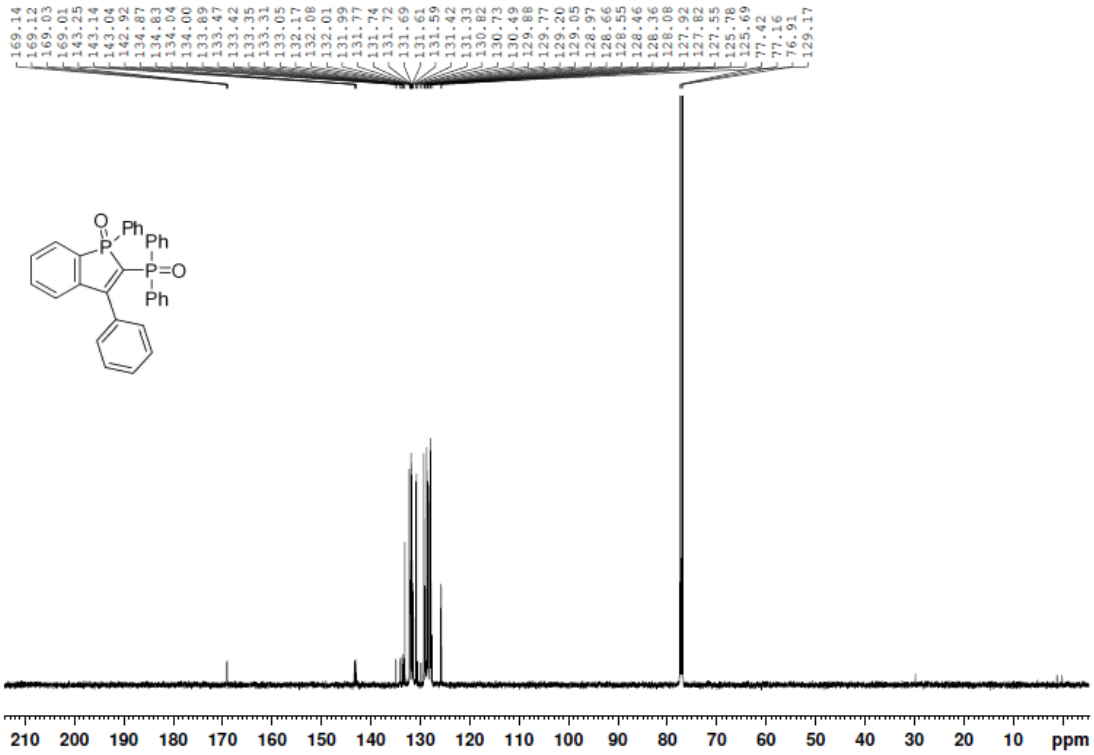
Detector with graphite-monochromated MoKa radiation (λ = 0.71073 Å) at 173 K. All of the Data were corrected for absorption effects using the multi-scan technique. The structures were solved by direct methods, expanded by difference Fourier syntheses and refined by full matrix least-squares on F2 using Bruker SHELXTL (Version 6.10) program package. Non-H atoms were refined anisotropically unless otherwise stated. Hydrogen atoms were introduced at their geometric positions and refined as riding atoms unless otherwise stated. SQUEEZE removed two disordered methanol molecules per formula unit. Some crystal data for **3m**: $C_{34}H_{26}O_3P_2$, M = 544.49, triclinic, space group P -1, pale yellow plate, a = 10.554(2) Å, b = 12.782(3) Å, c = 12.959(3) Å, α = 75.259(4)°, β = 67.900(4)°, γ = 76.462(4)°, V = 1565.5(6) $Å^3$, Z = 2, Dc = 1.155 g/cm^{-3}, F(000) = 568, μ(Mo-Kα) = 0.142 mm^{-1}, T = 173(2) K. Final R [4812 with I > 2σ(I)] = 0.0519, wR2 (all data) = 0.1387. Further details on the crystal structure investigation have been deposited at the Cambridge Crystallographic Data Centre as the deposition number CCDC 1046595.

NMR Spectrum of 3a-3v, 5 and 6:

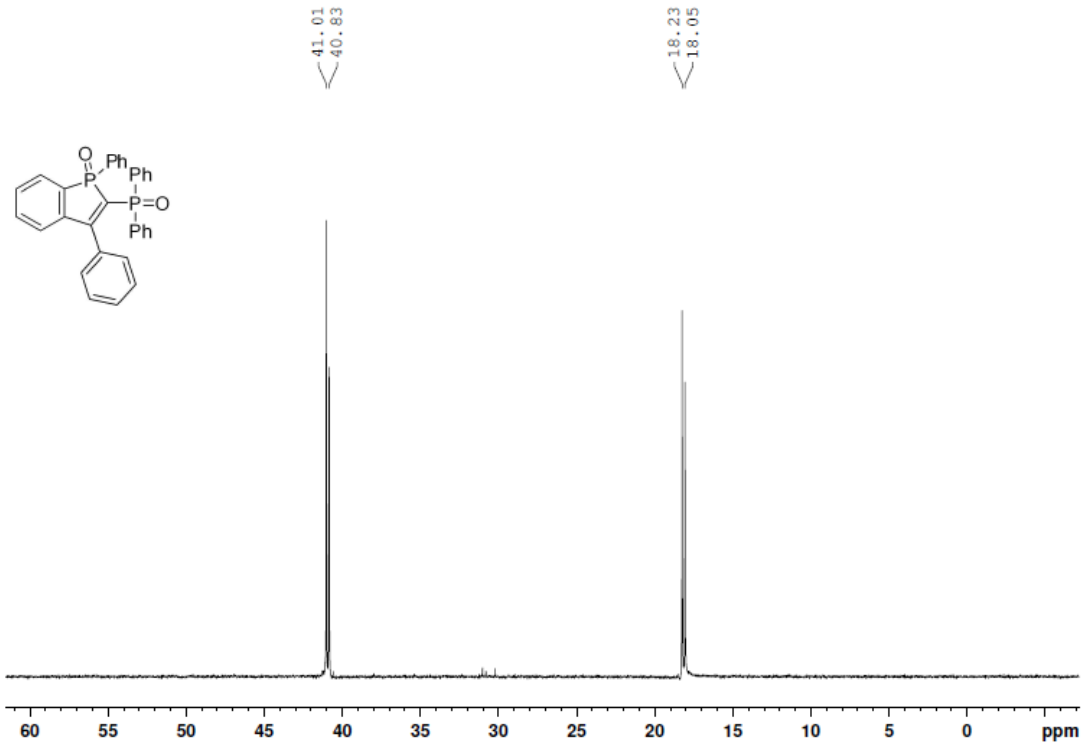
[1] H NMR of **3a**



[13] C NMR of **3a**



^{31} P NMR of **3a**



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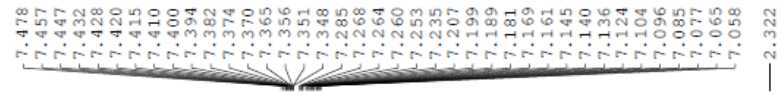

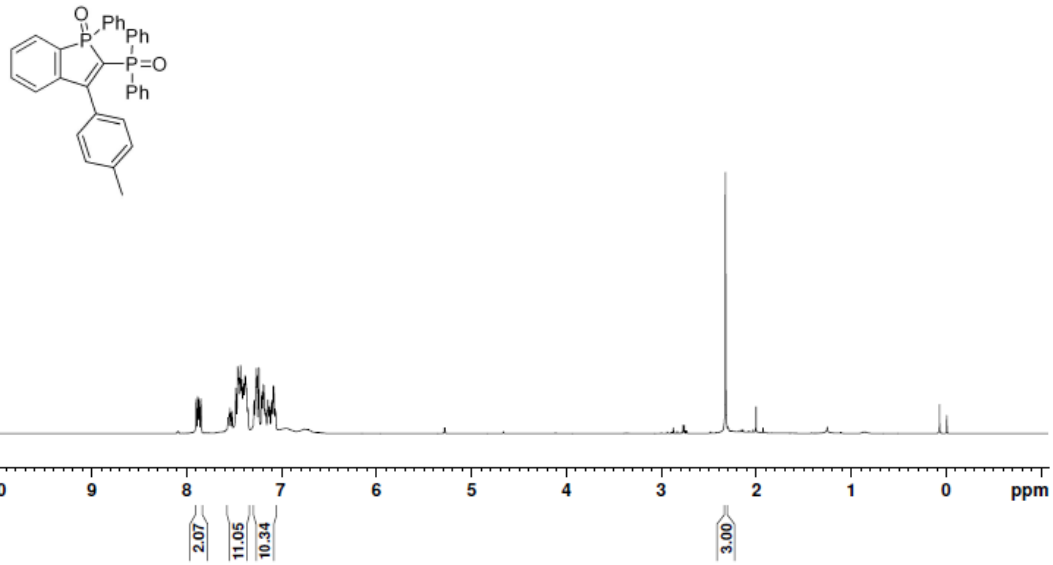

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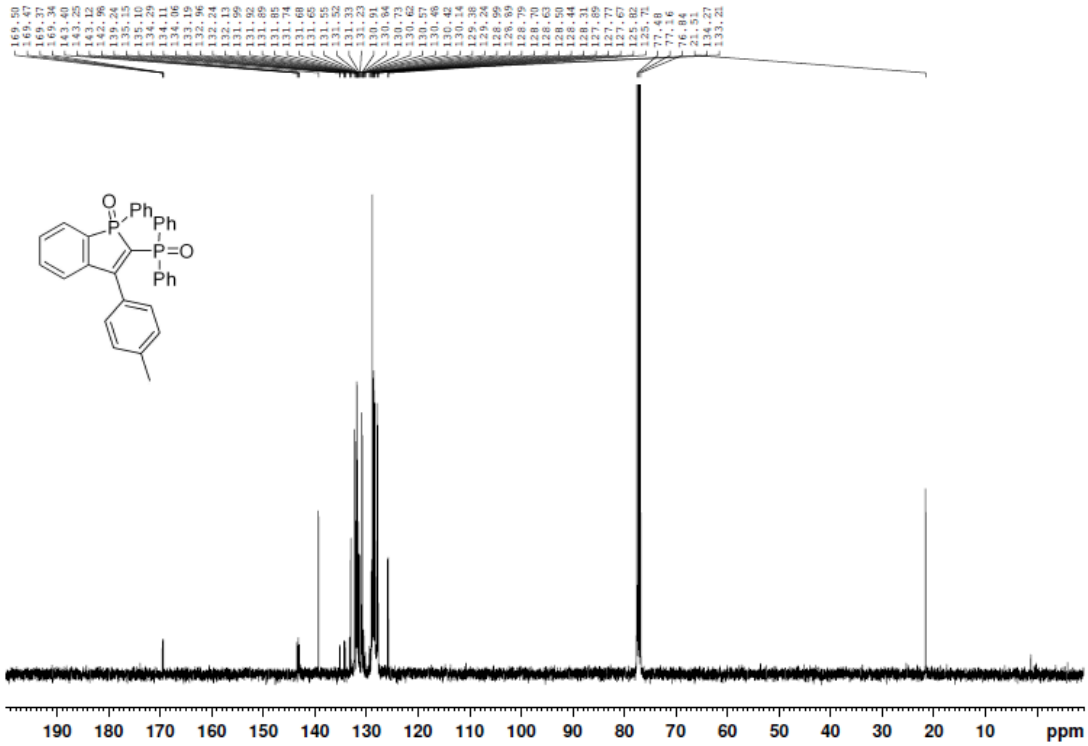

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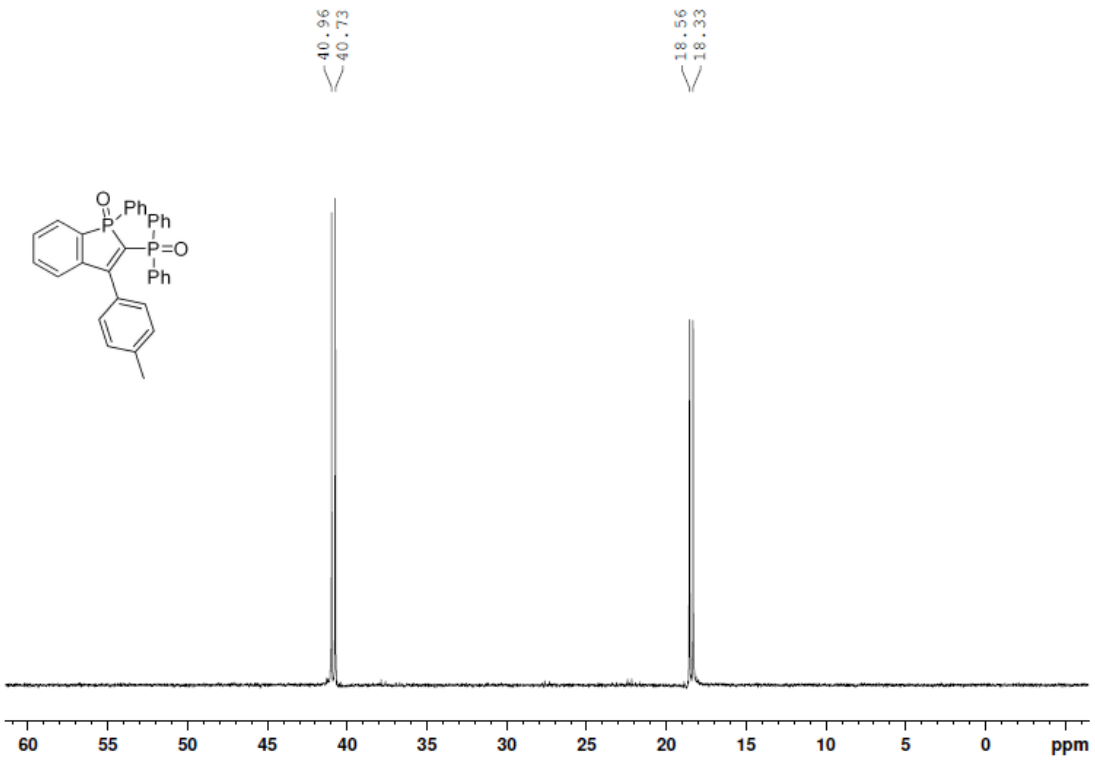

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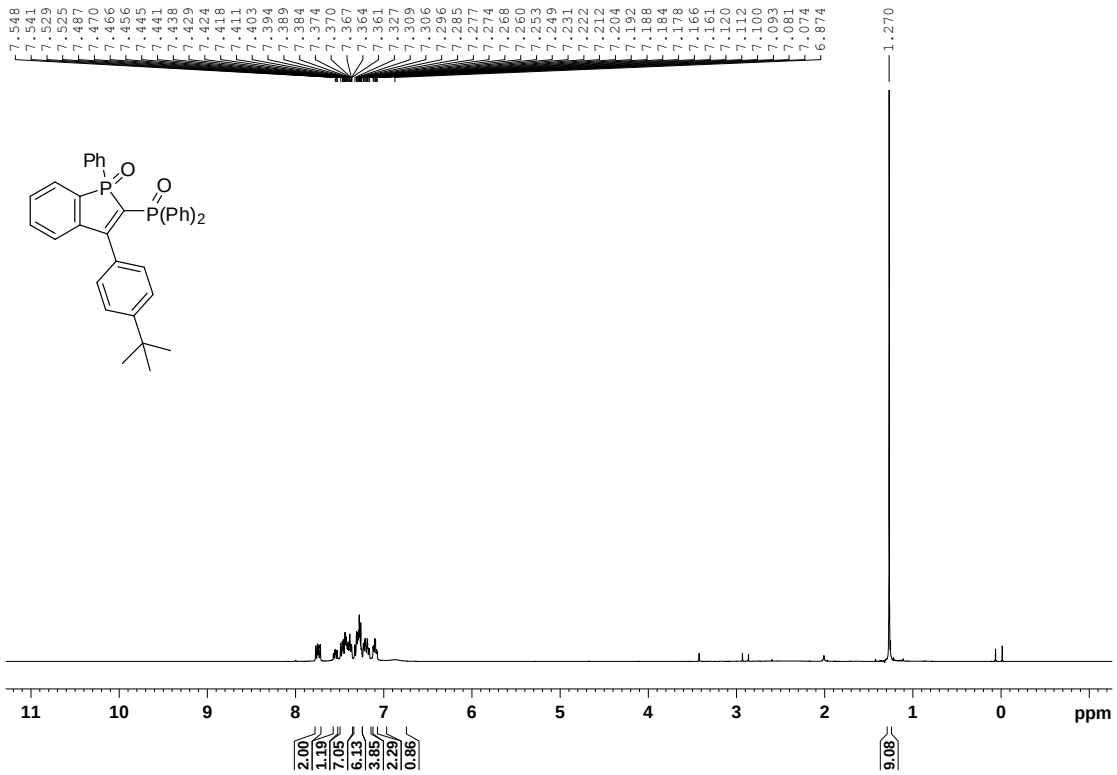

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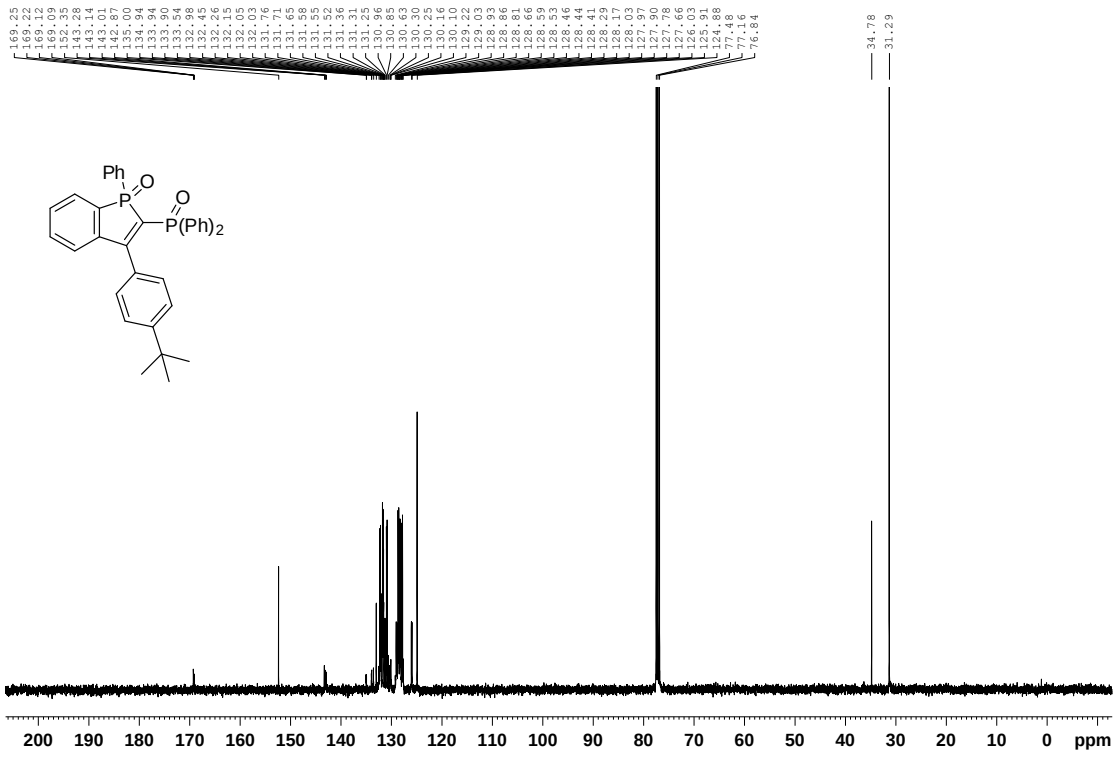

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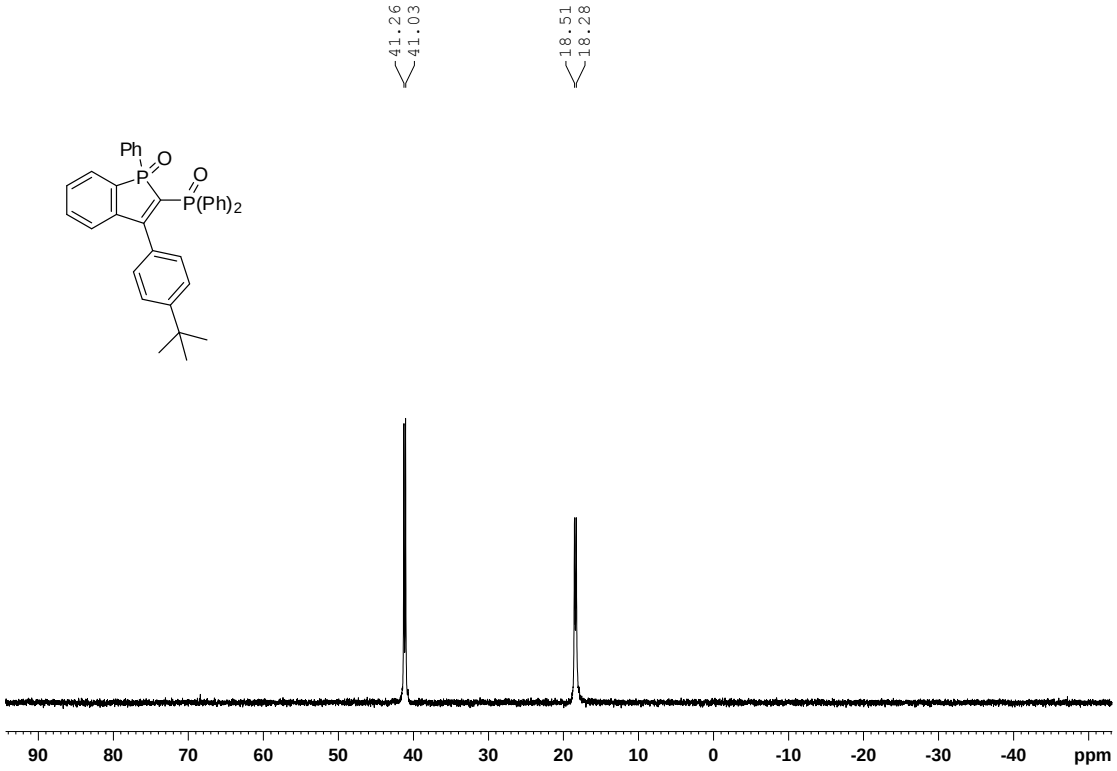

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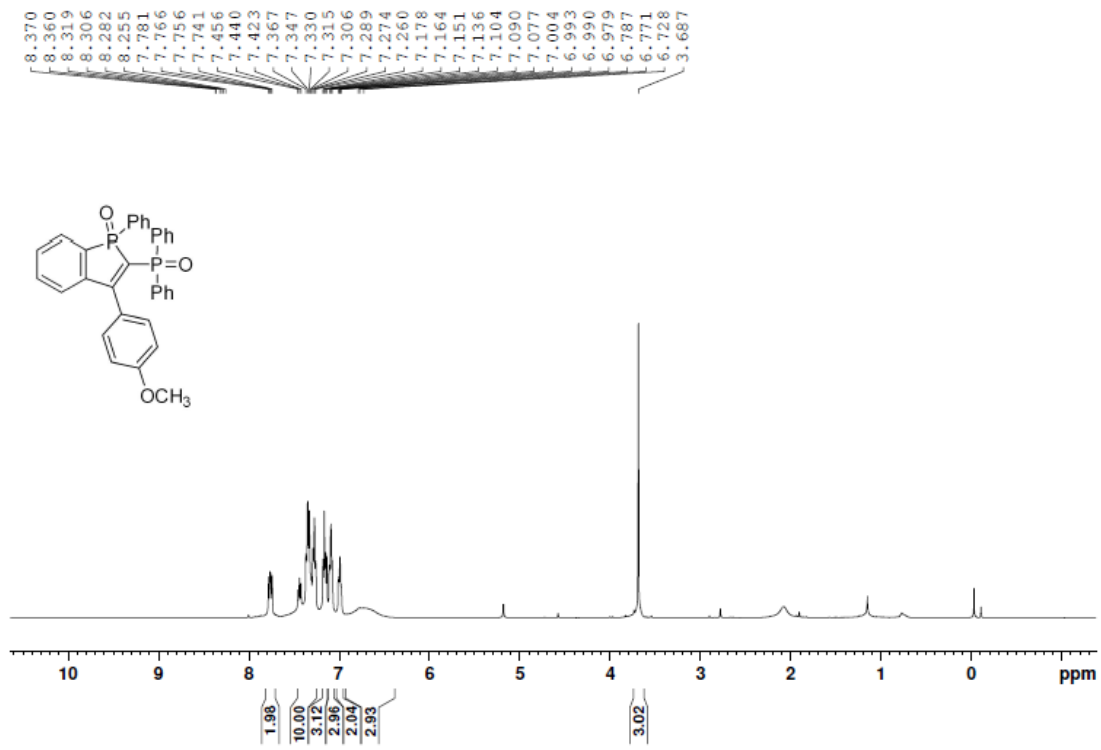

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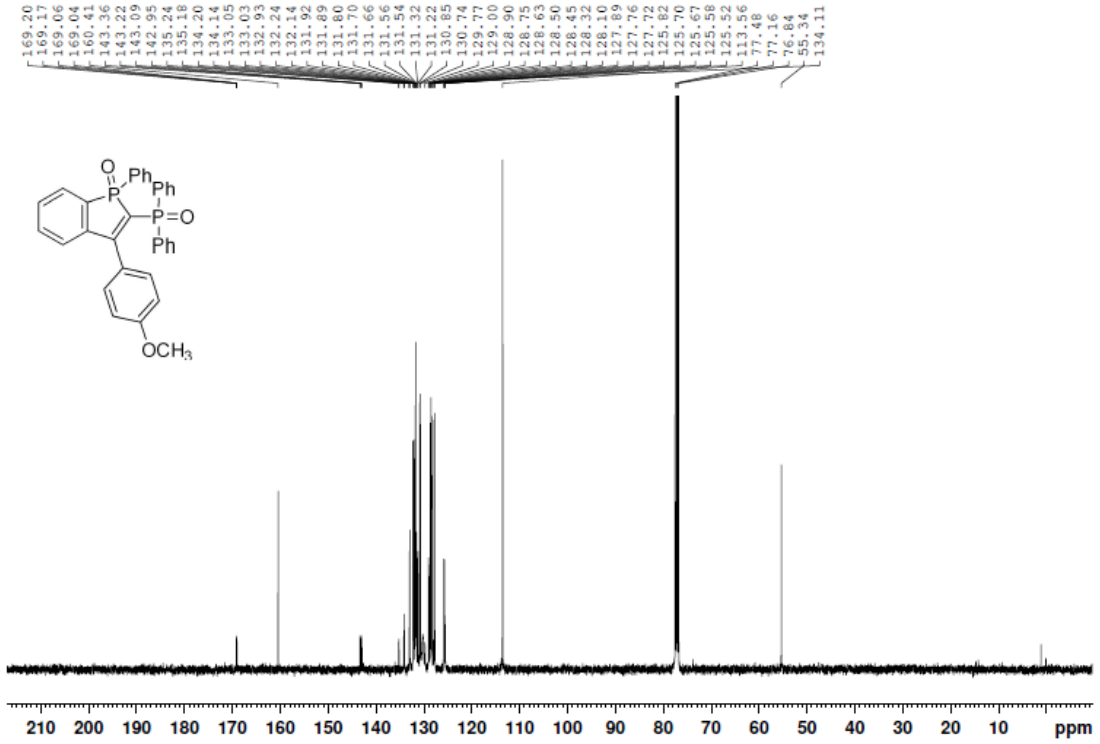

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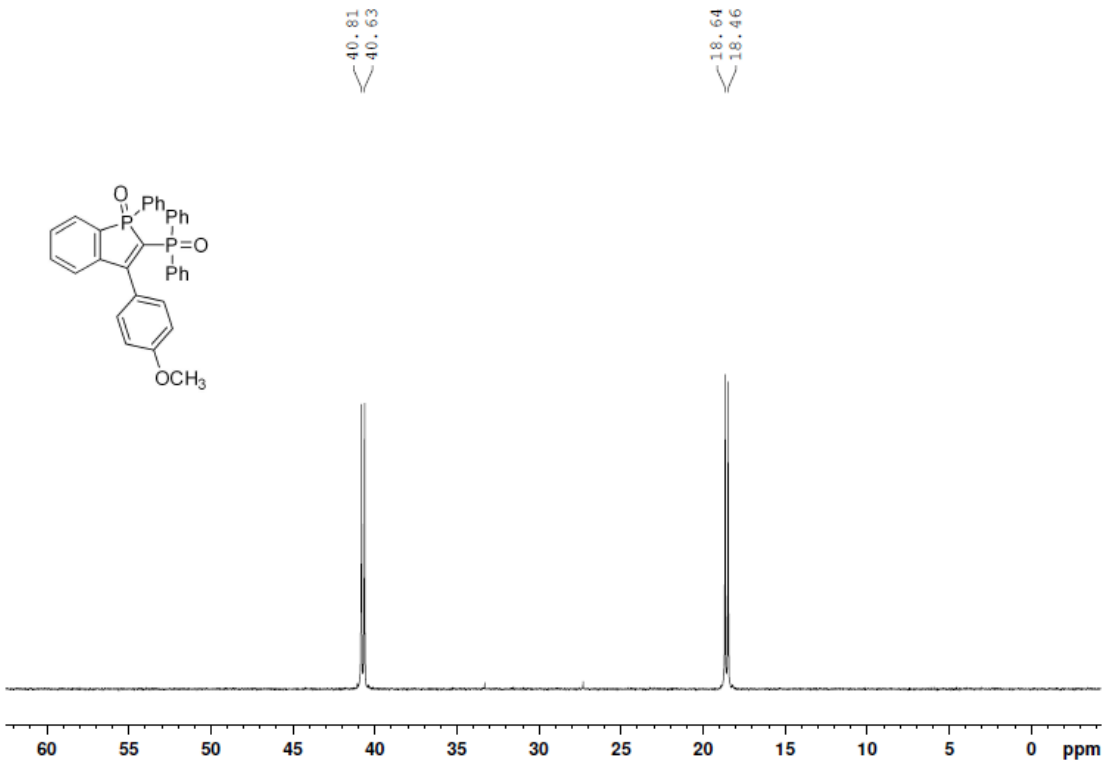

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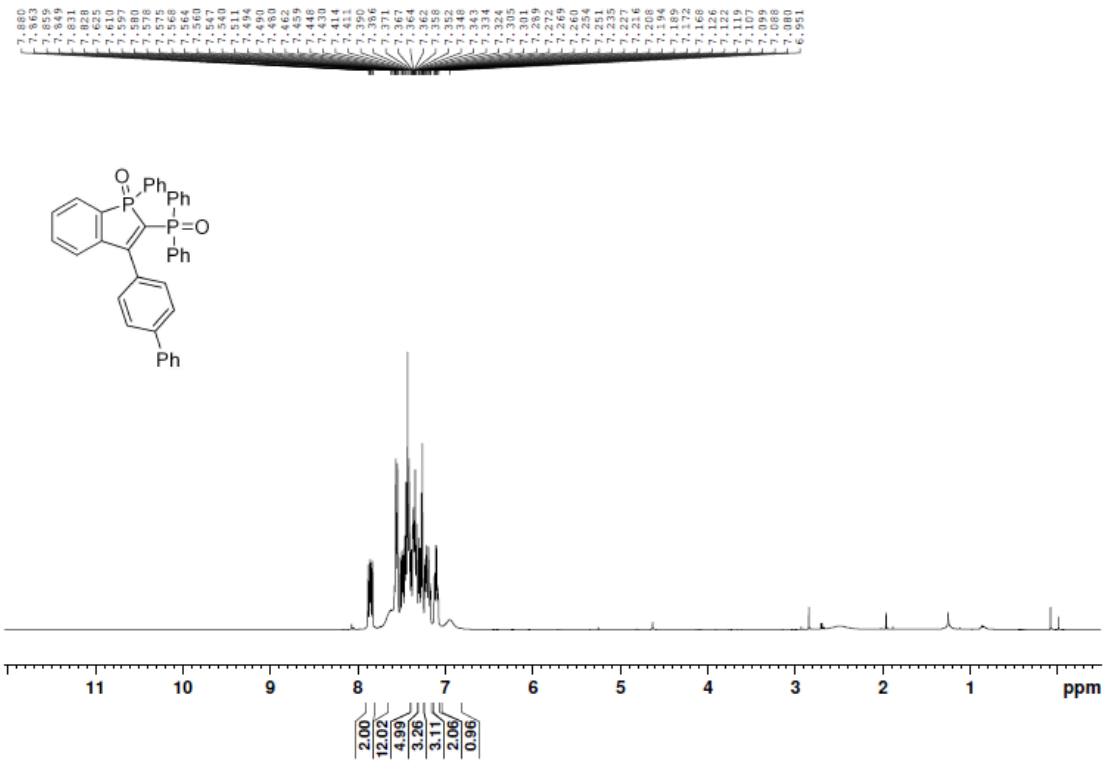

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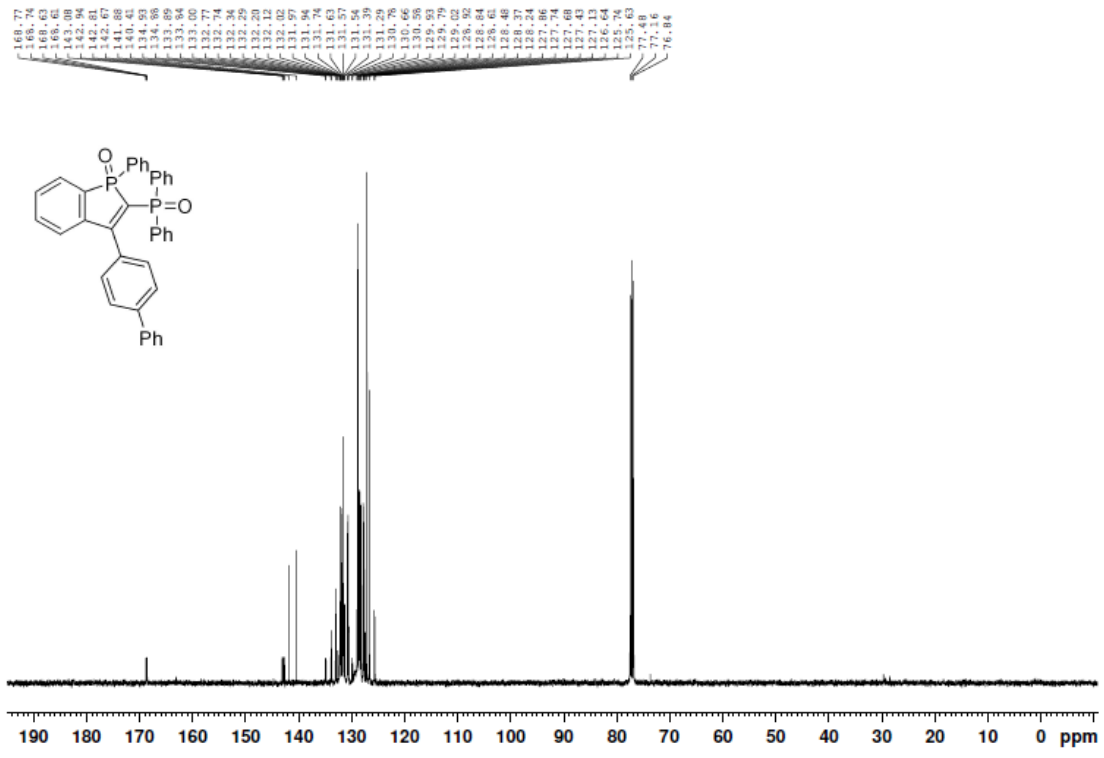

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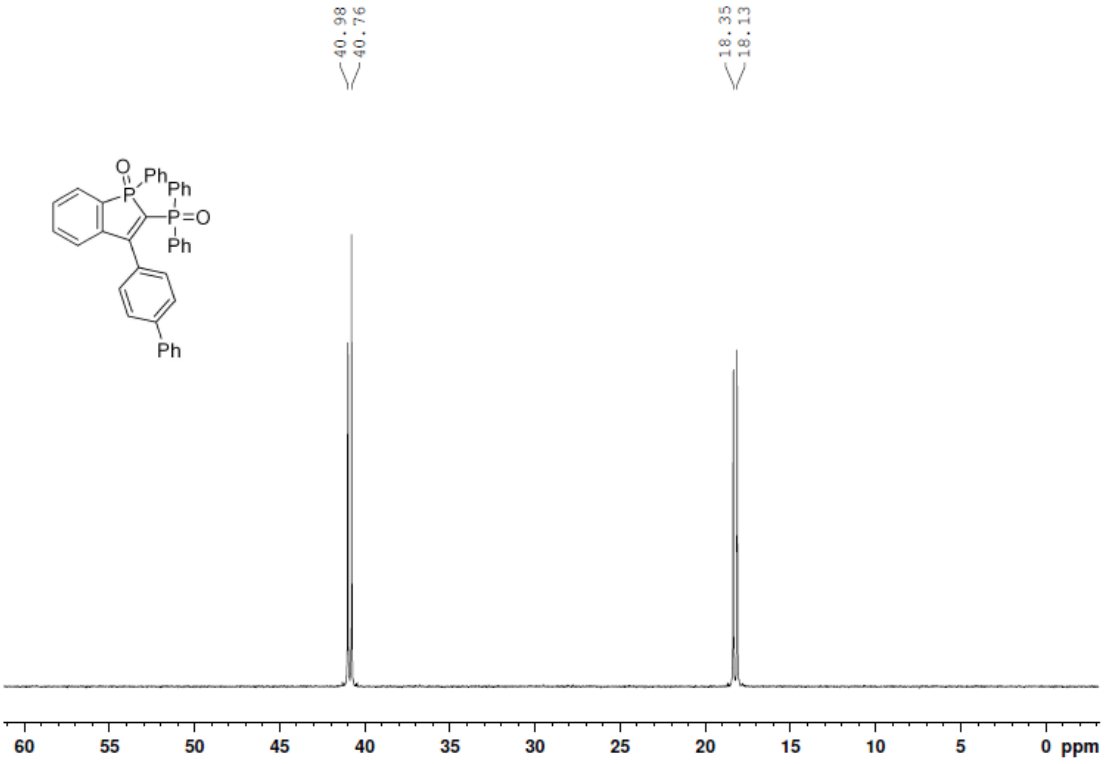

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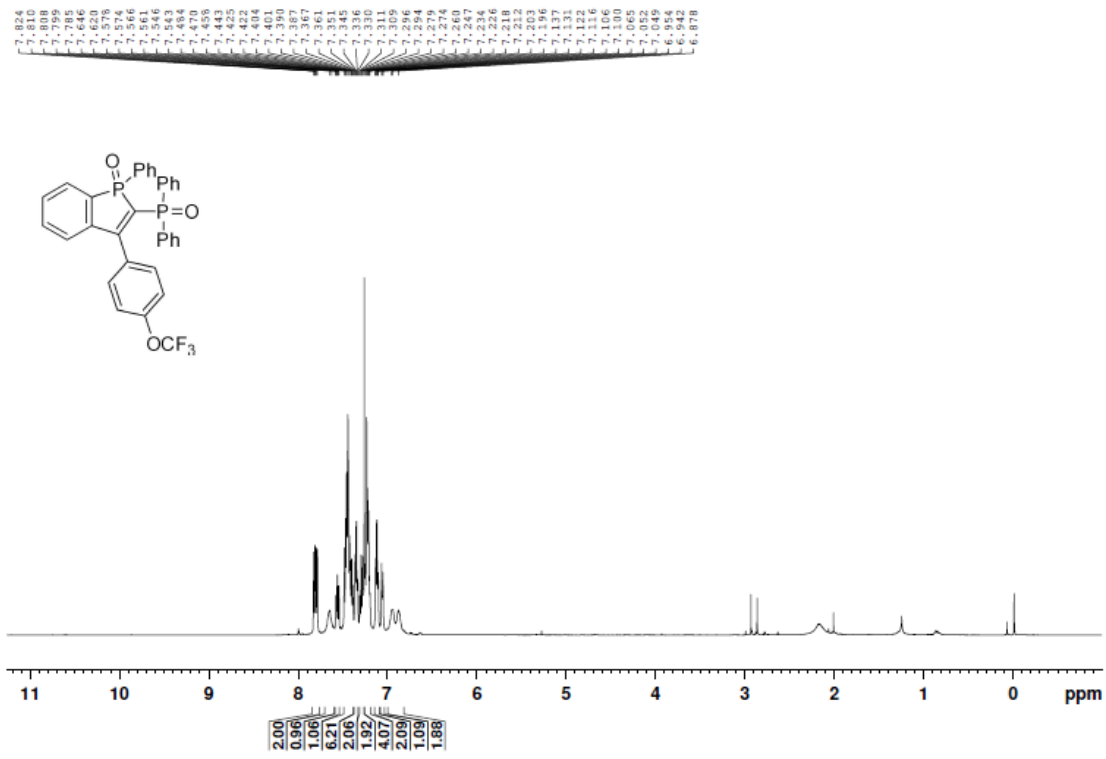

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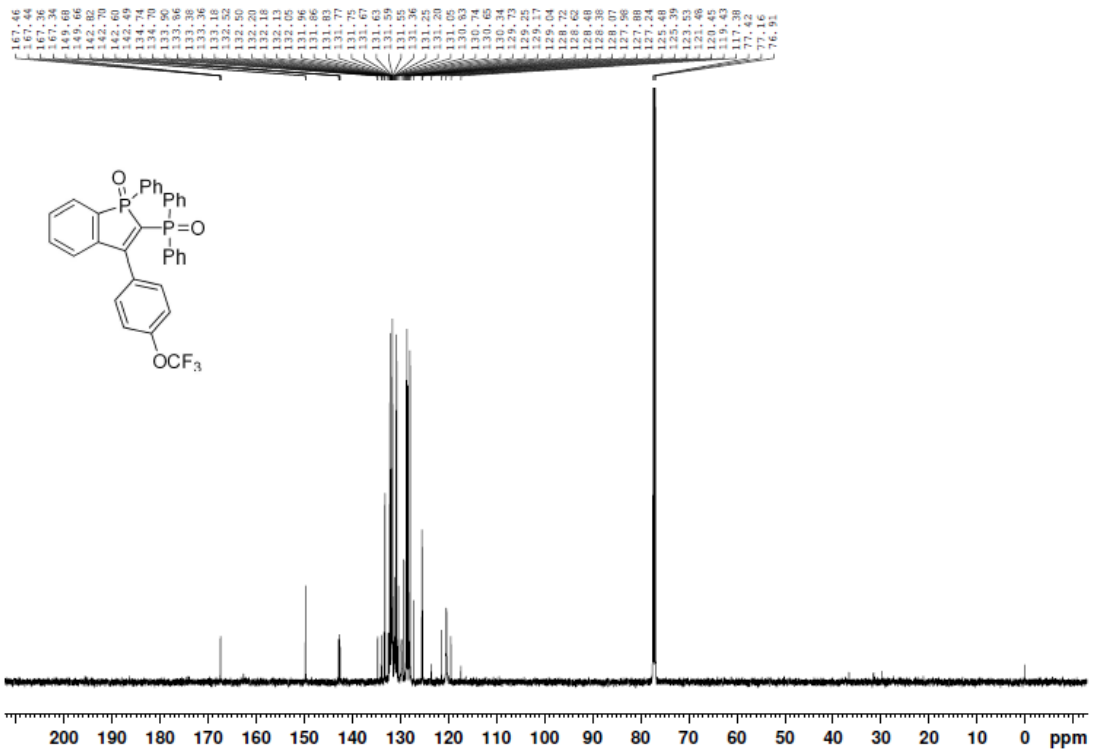

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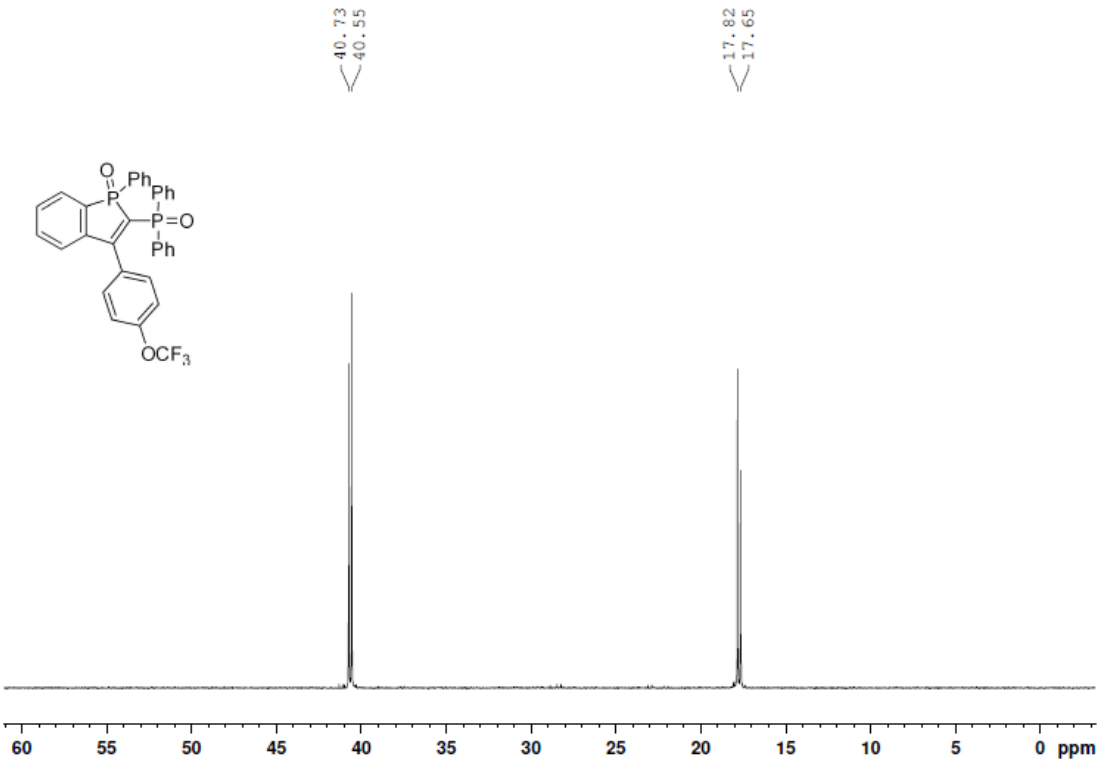

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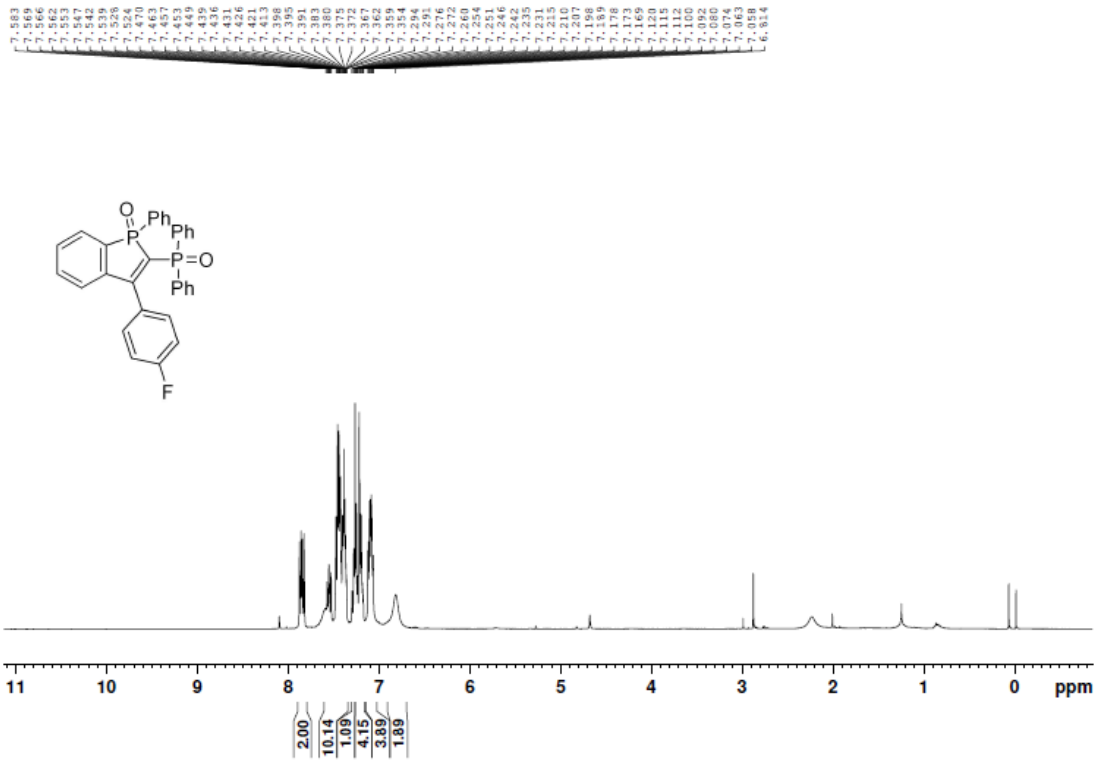

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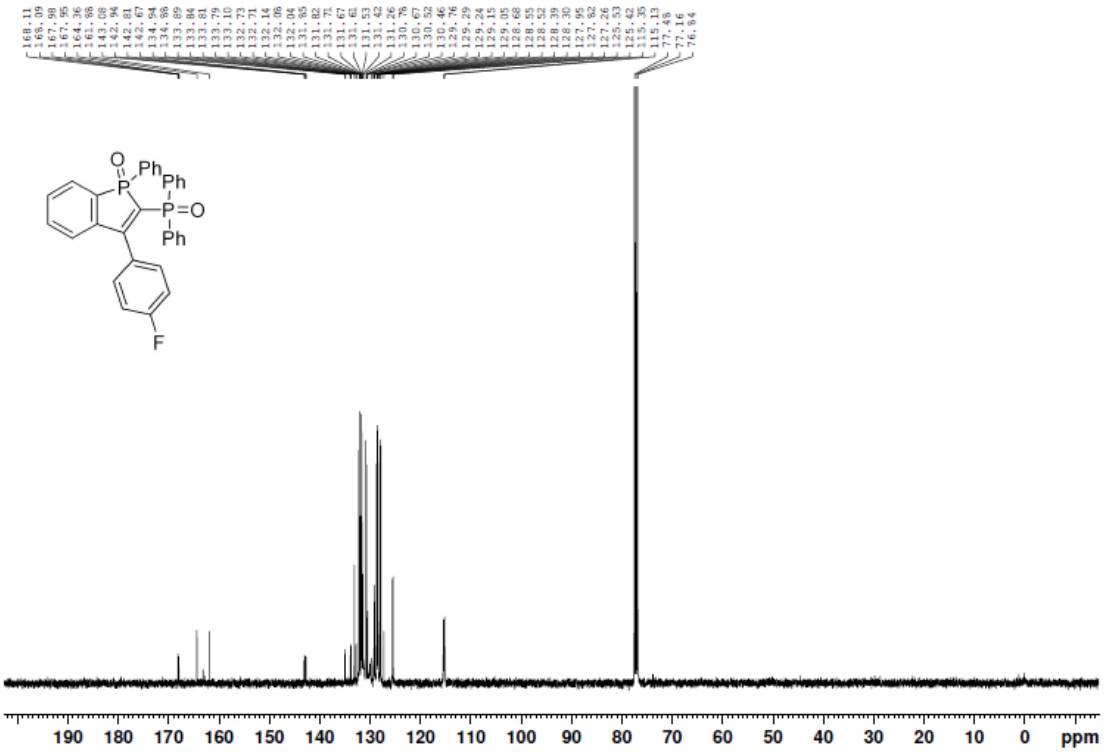

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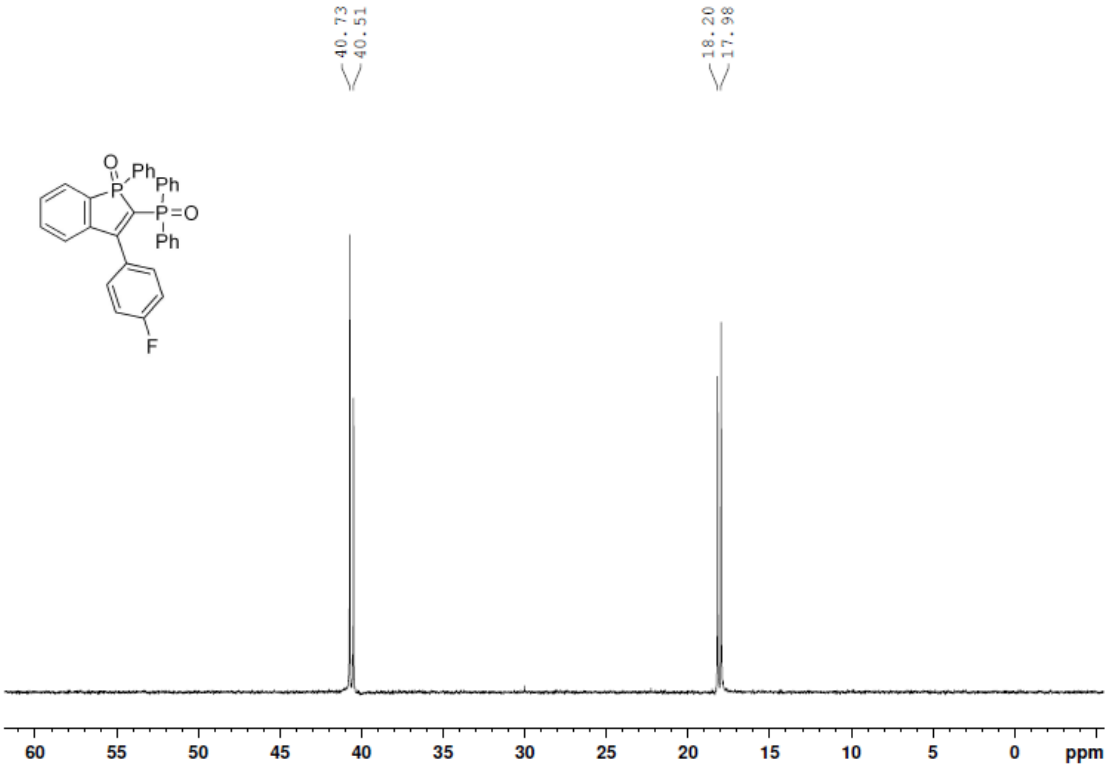

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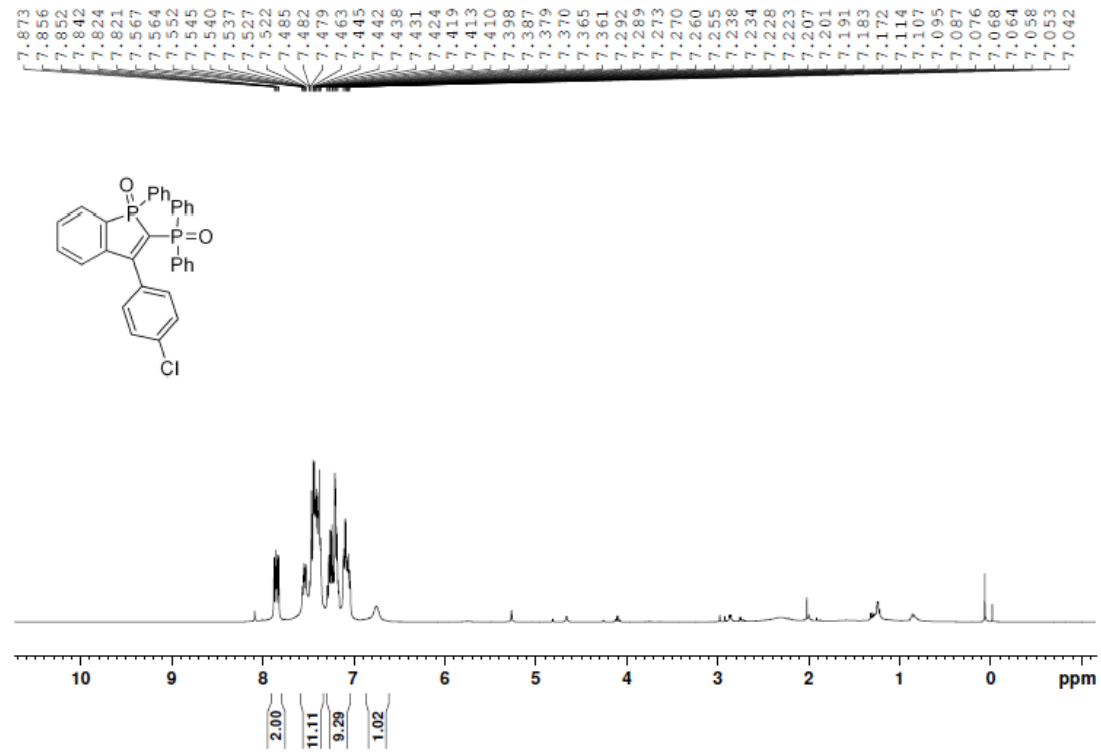

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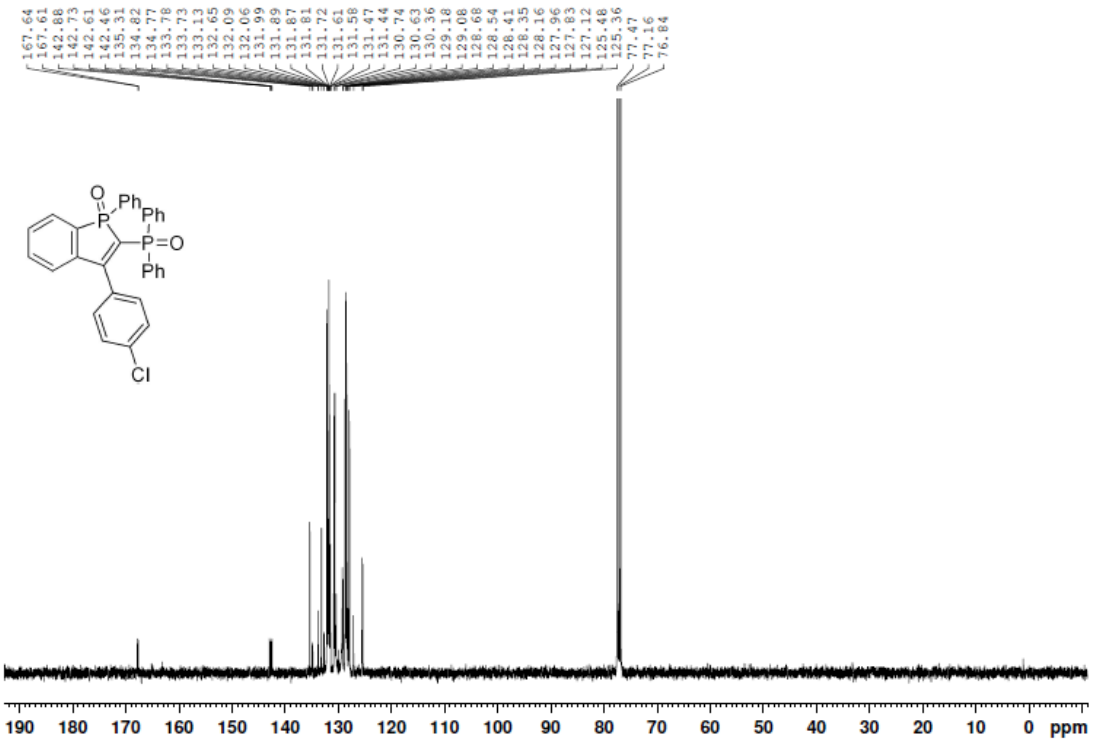

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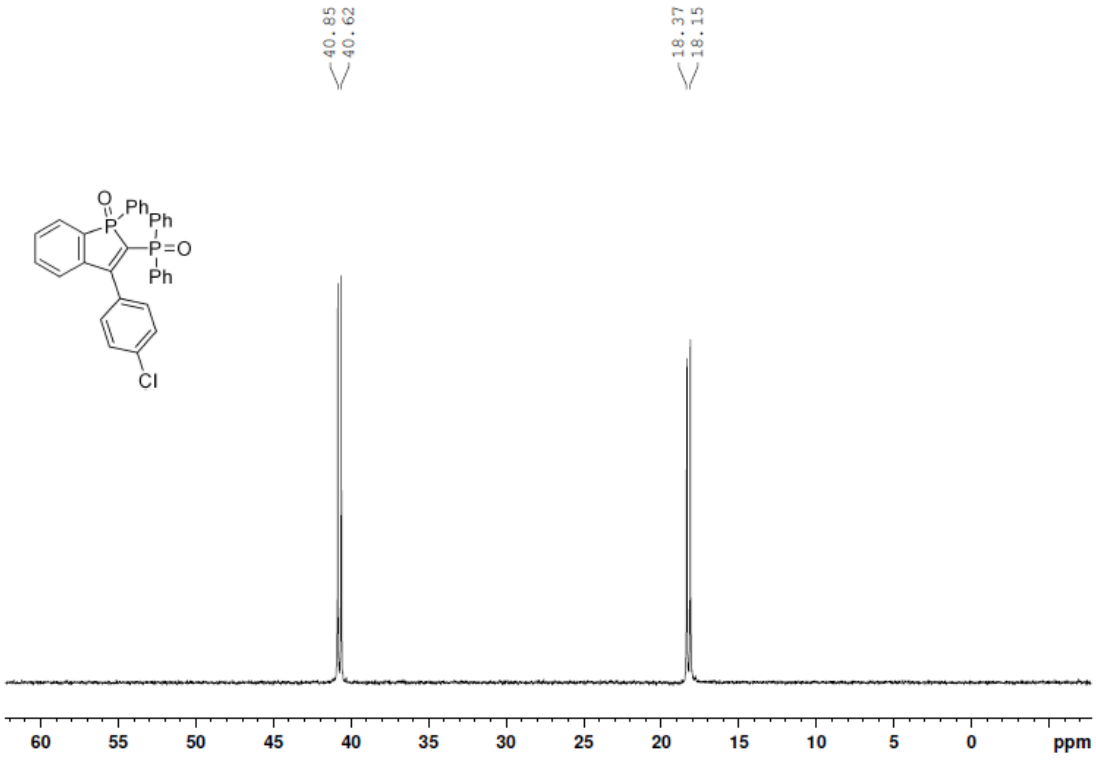

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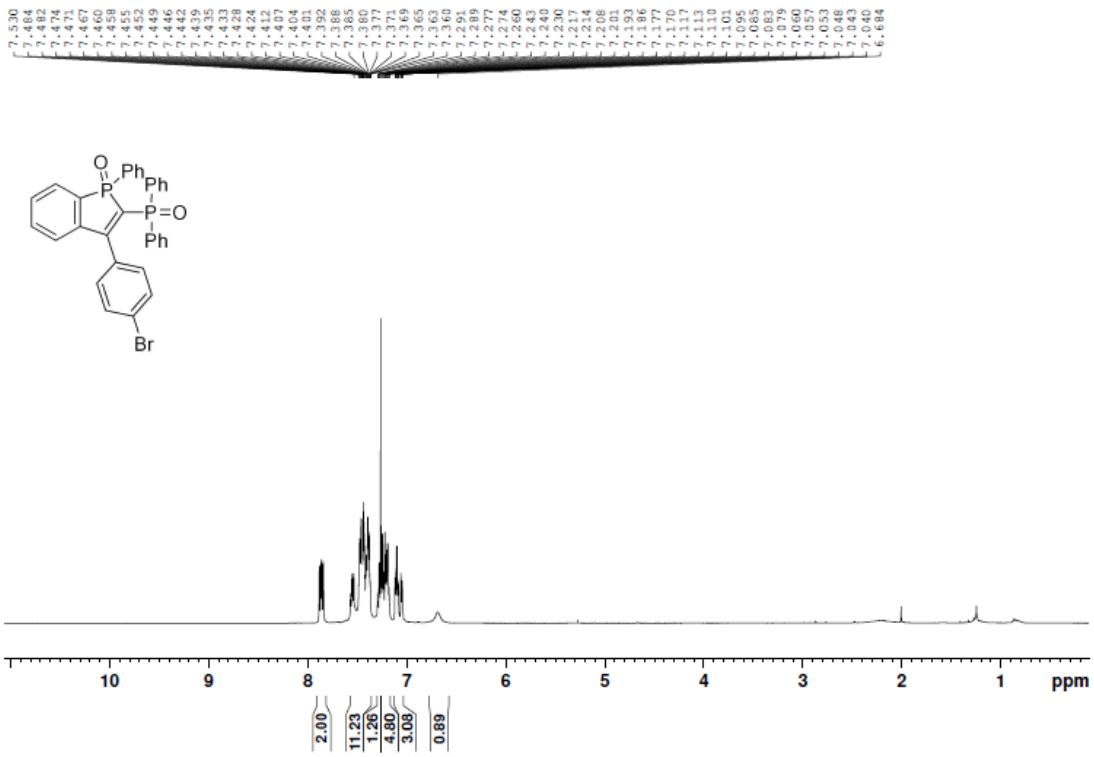

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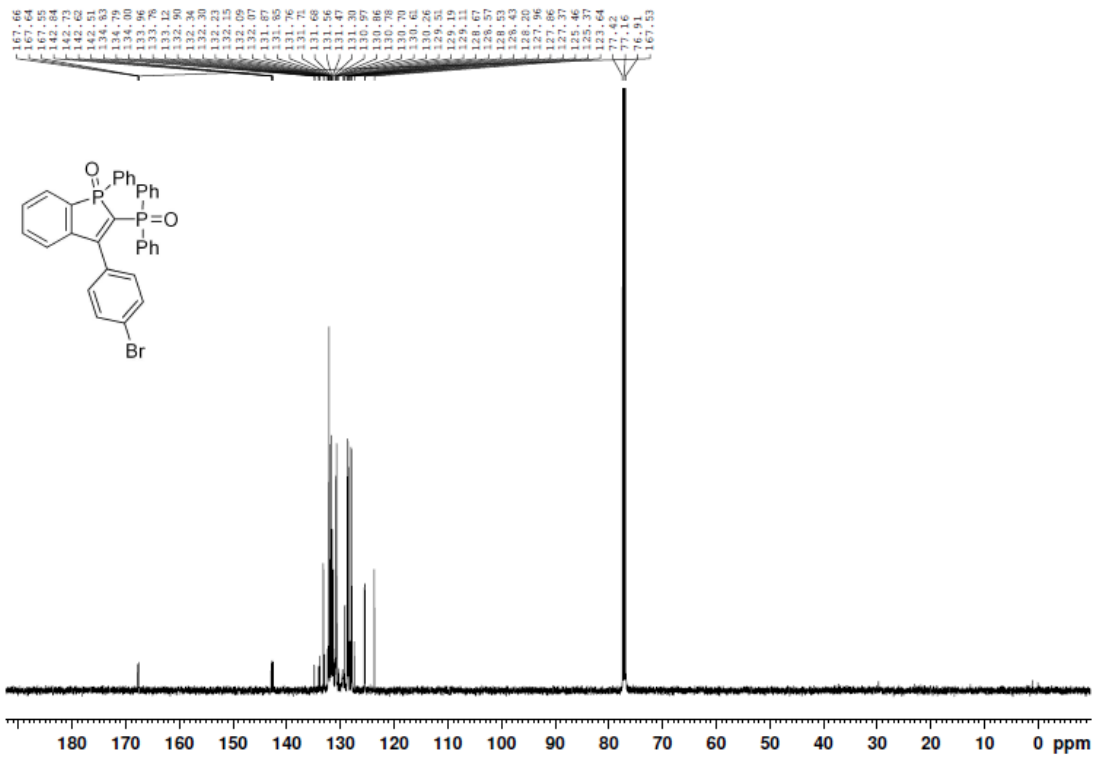

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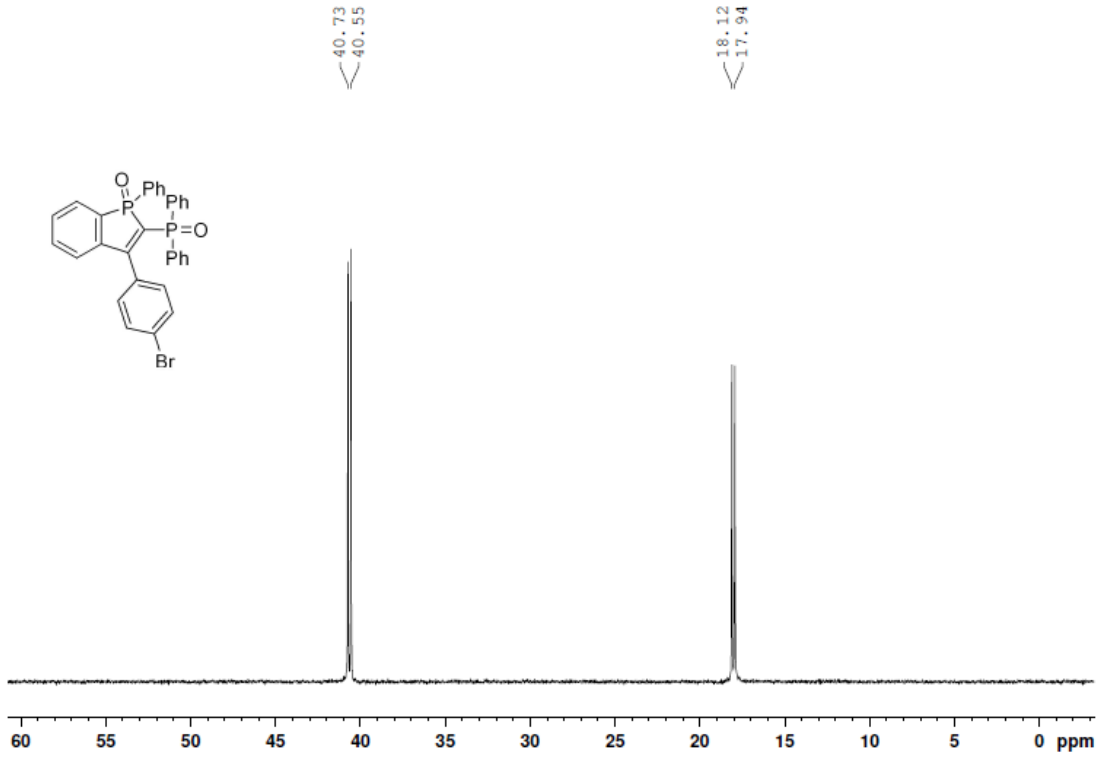

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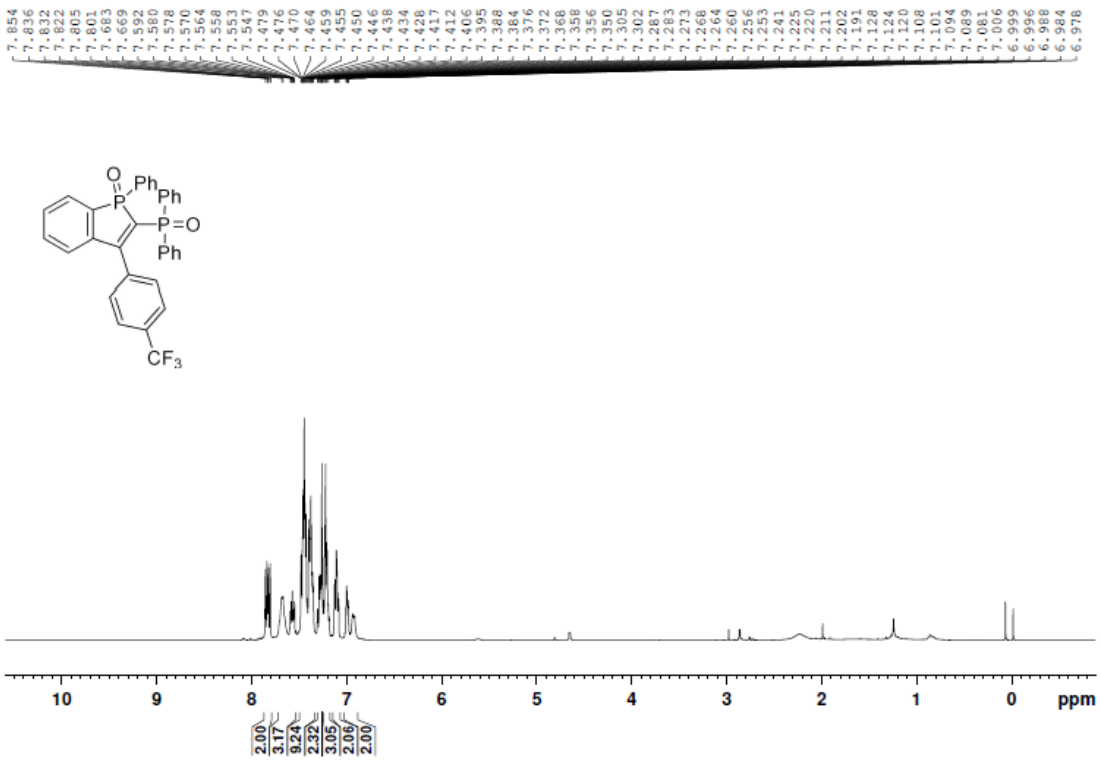

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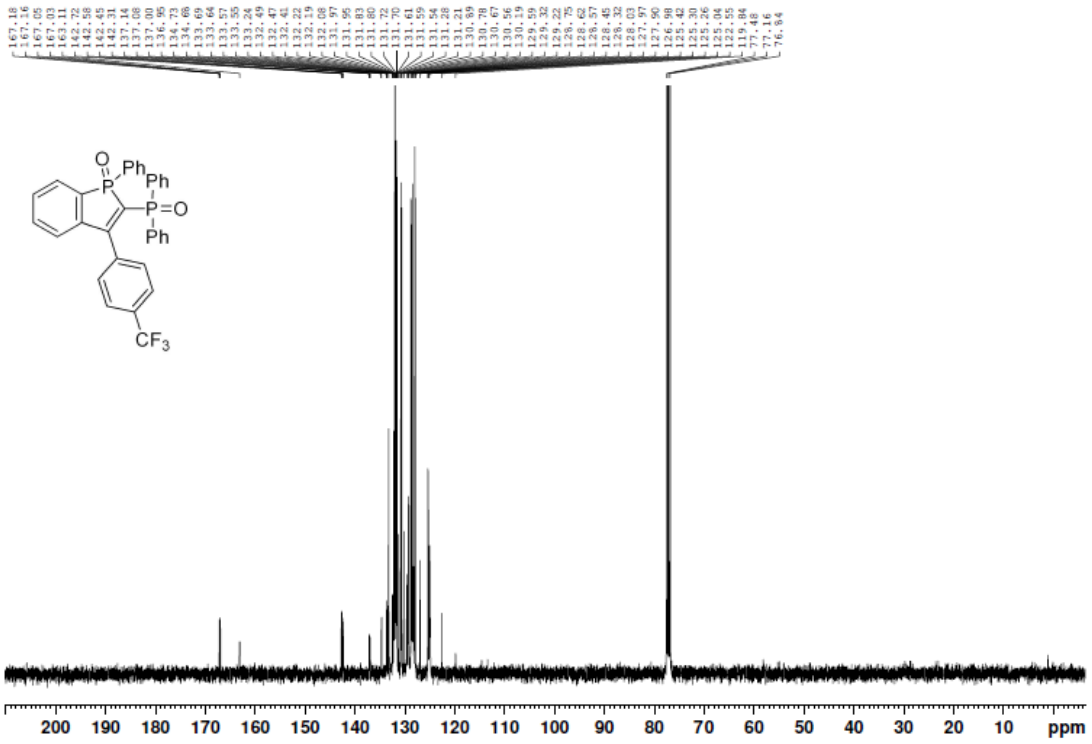

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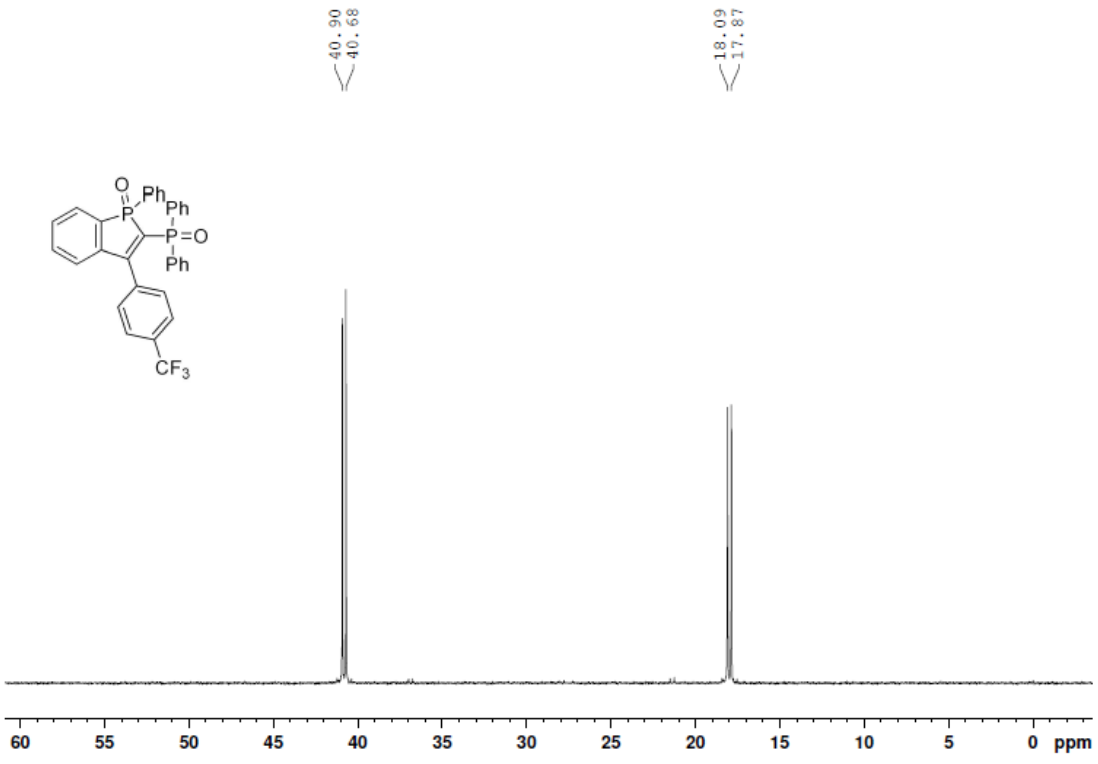

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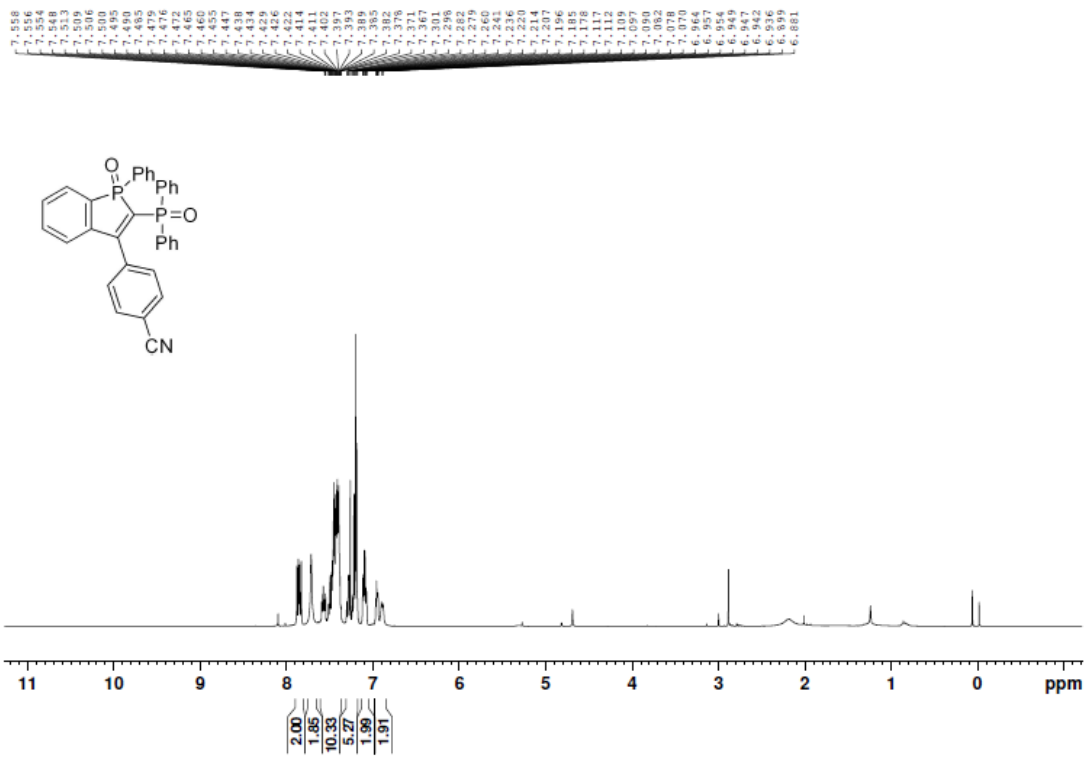

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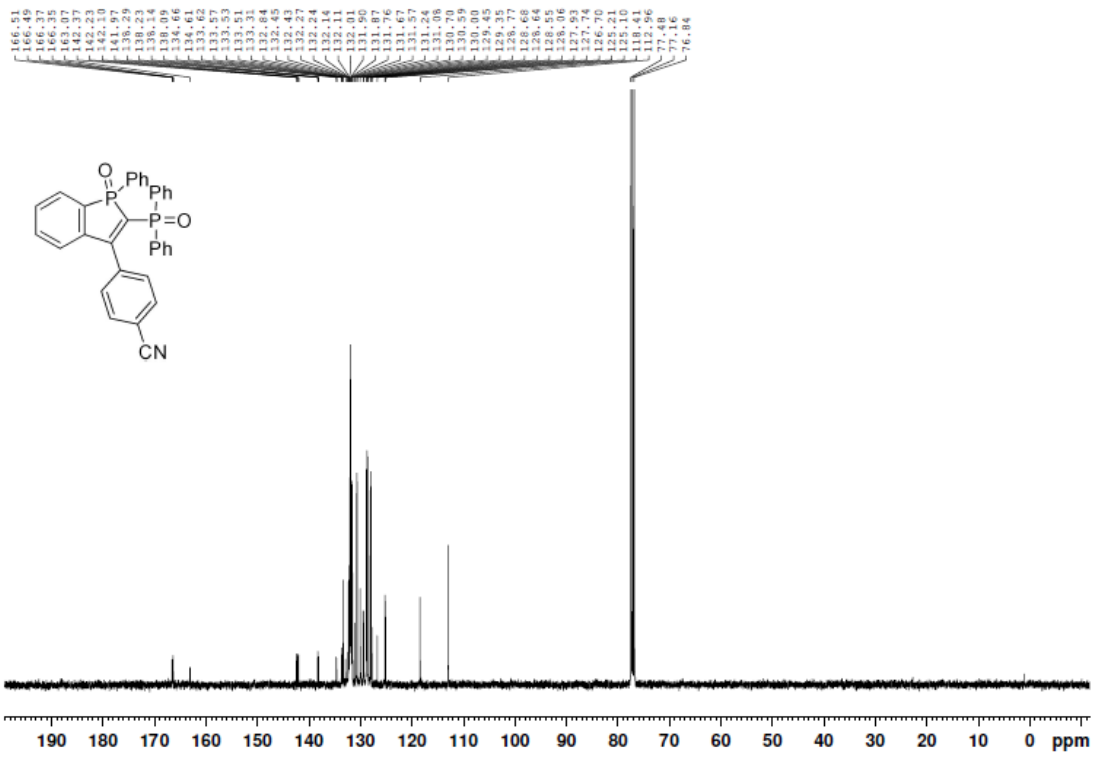

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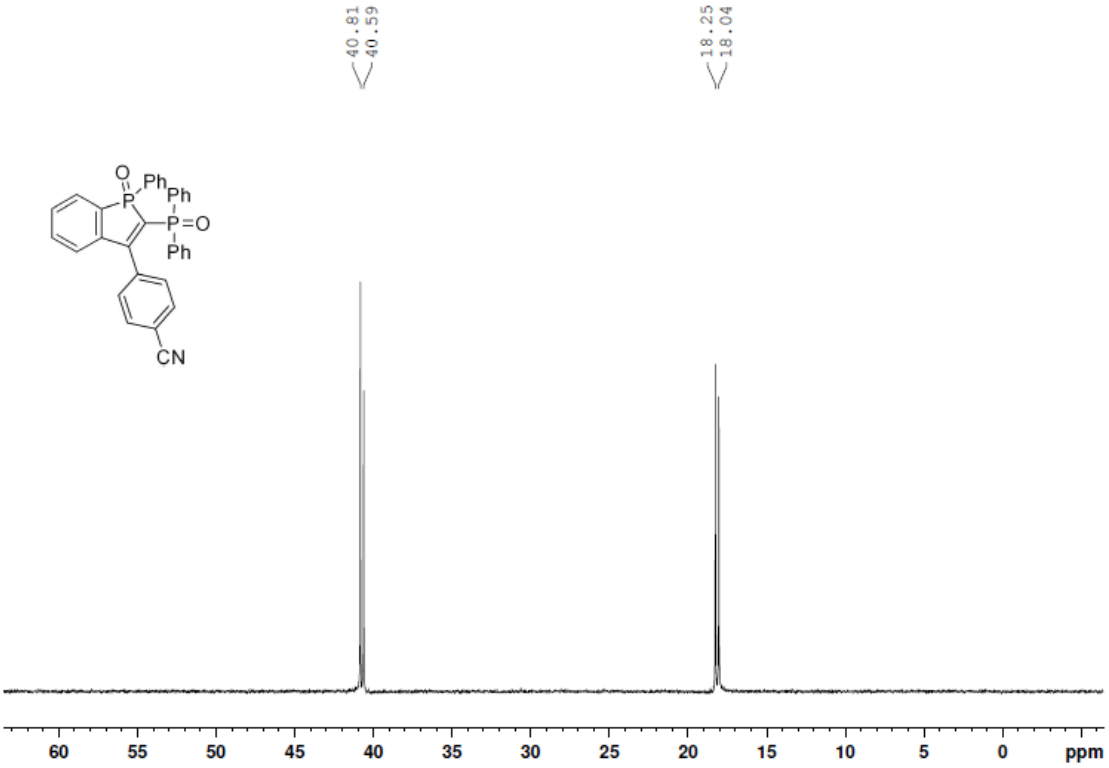

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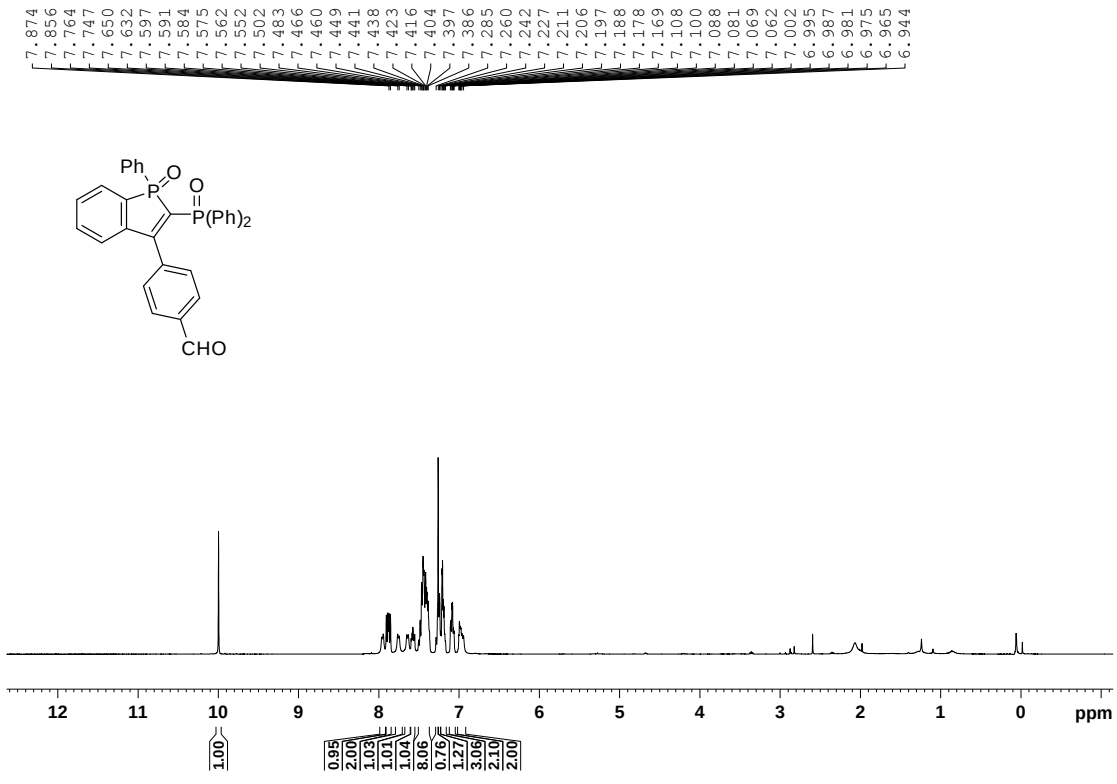

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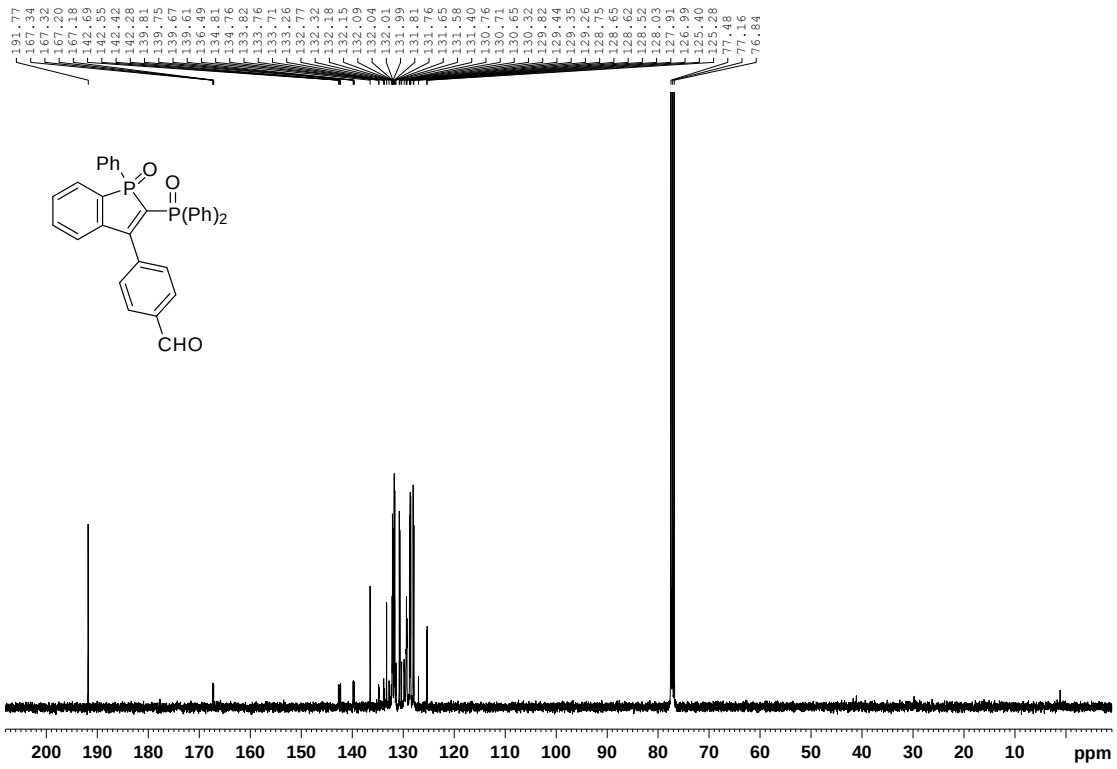

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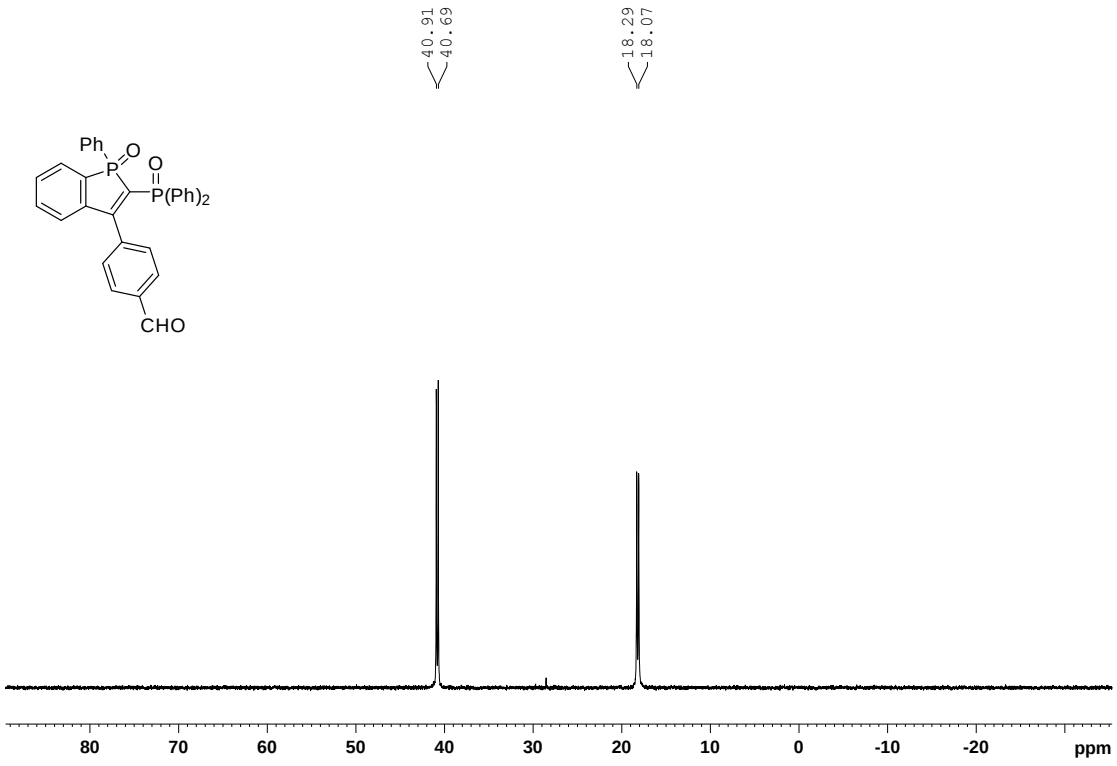

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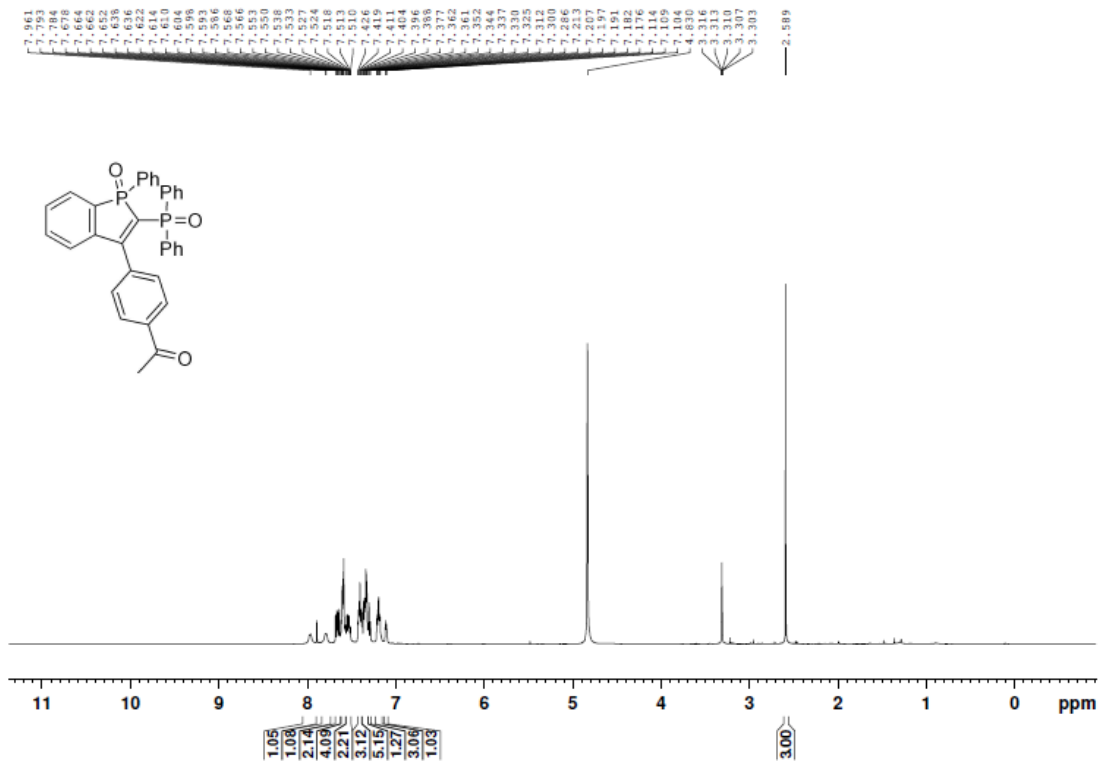

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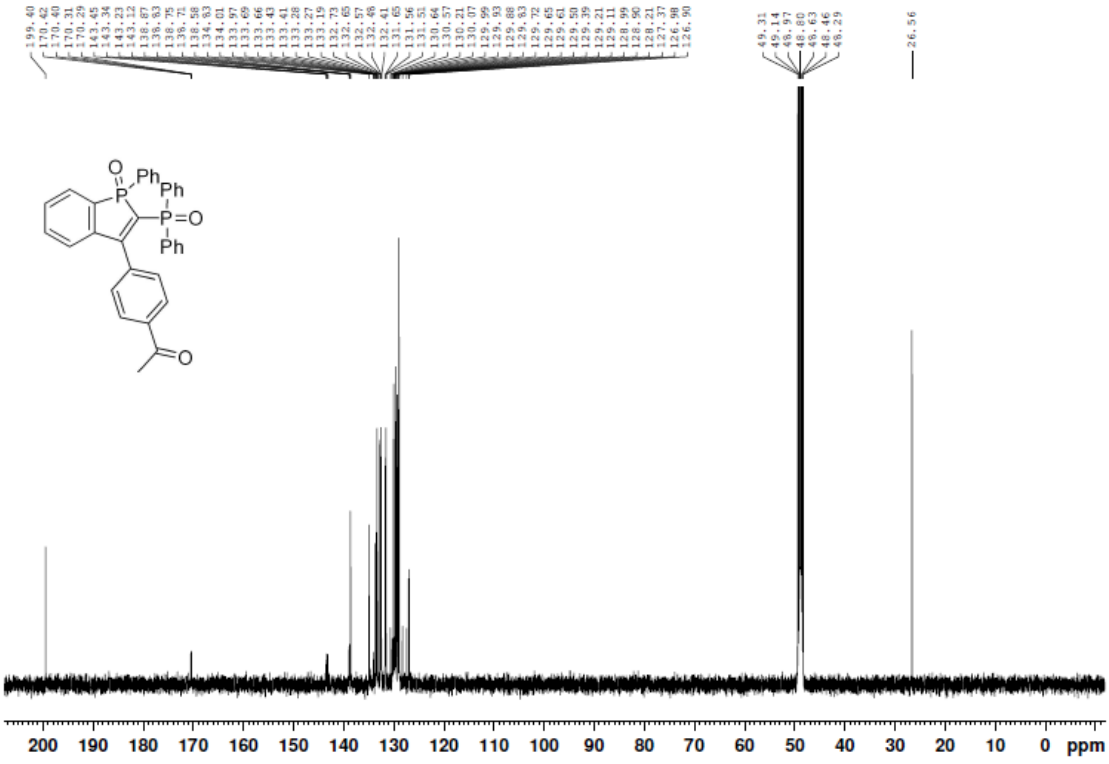

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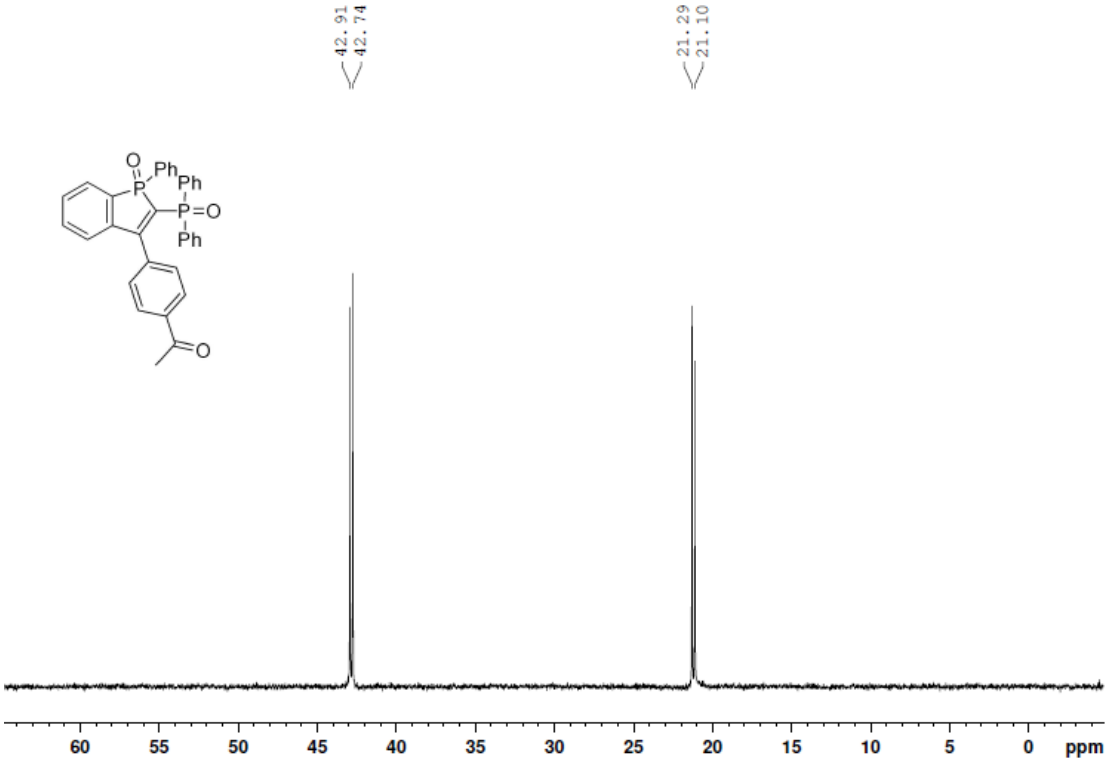

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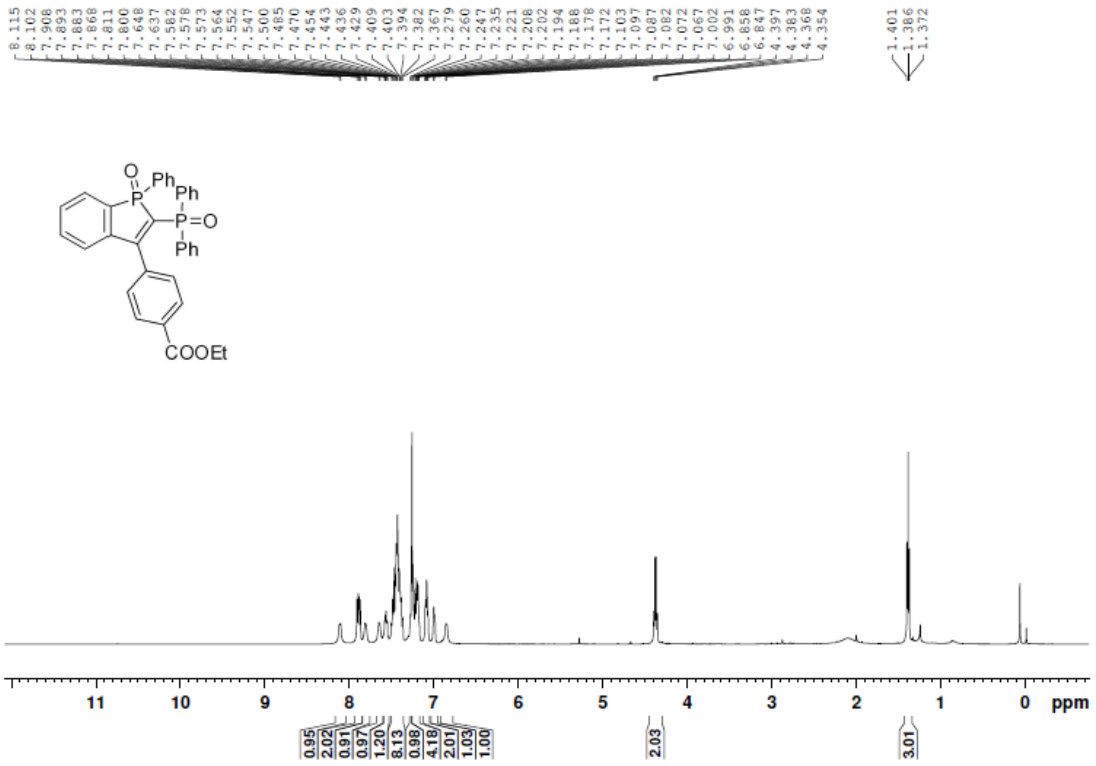

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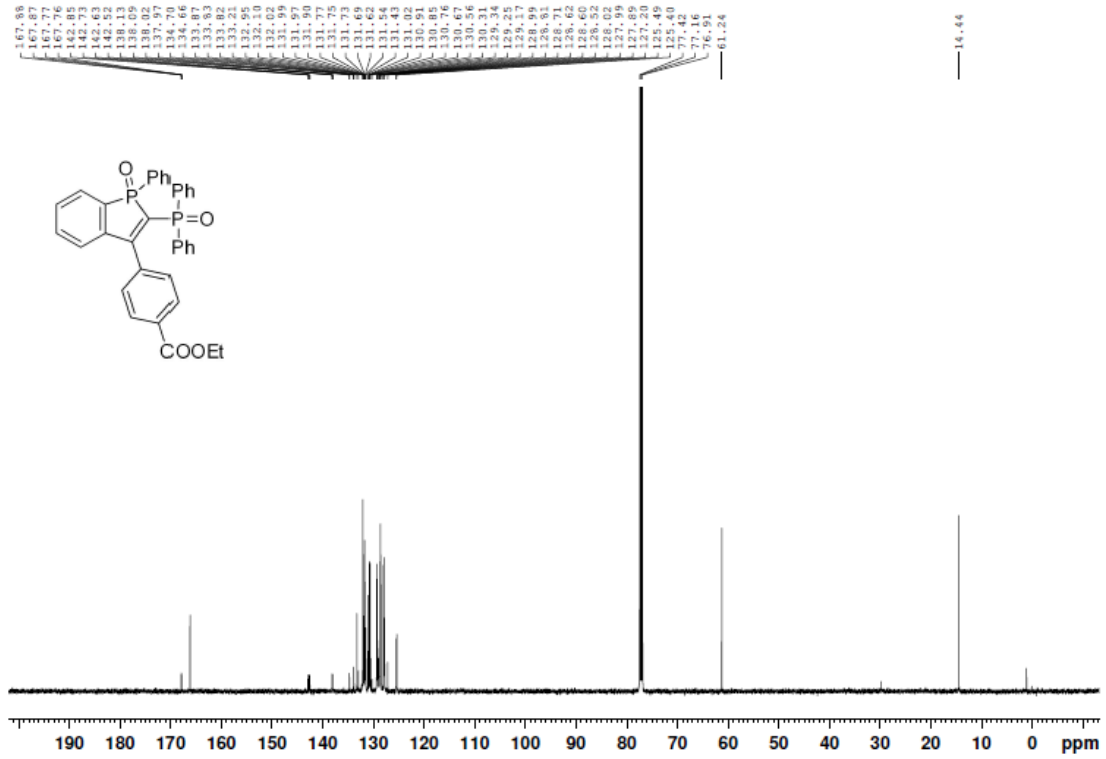

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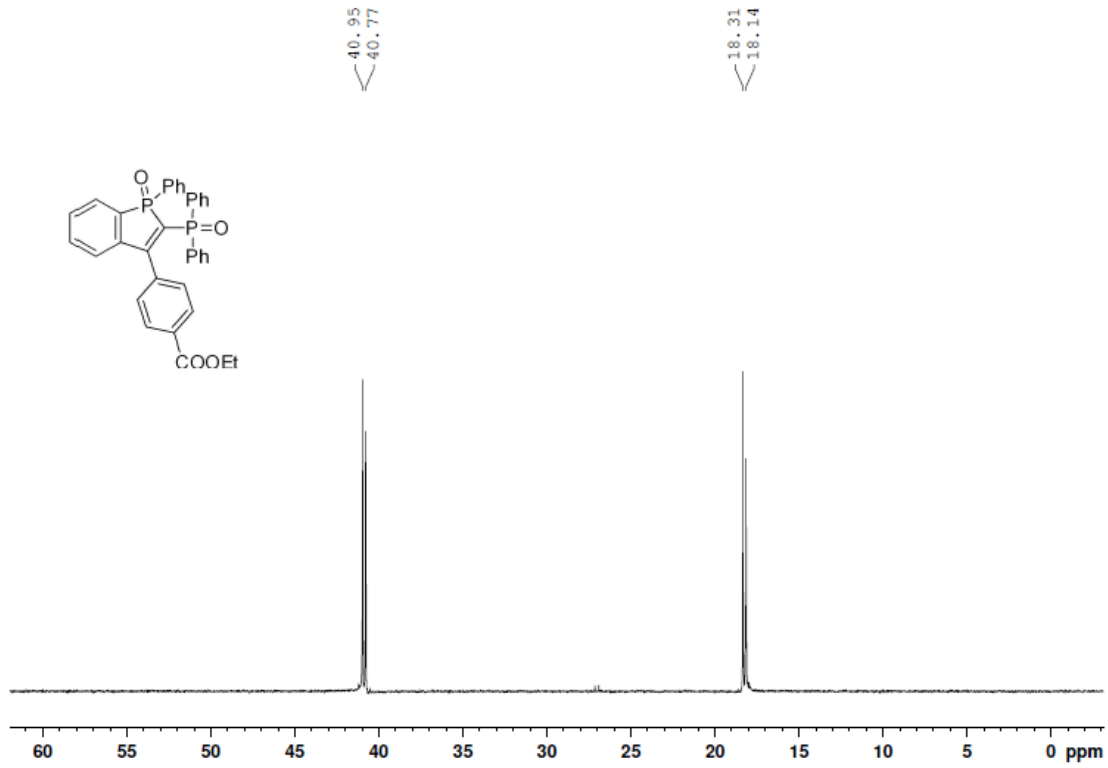

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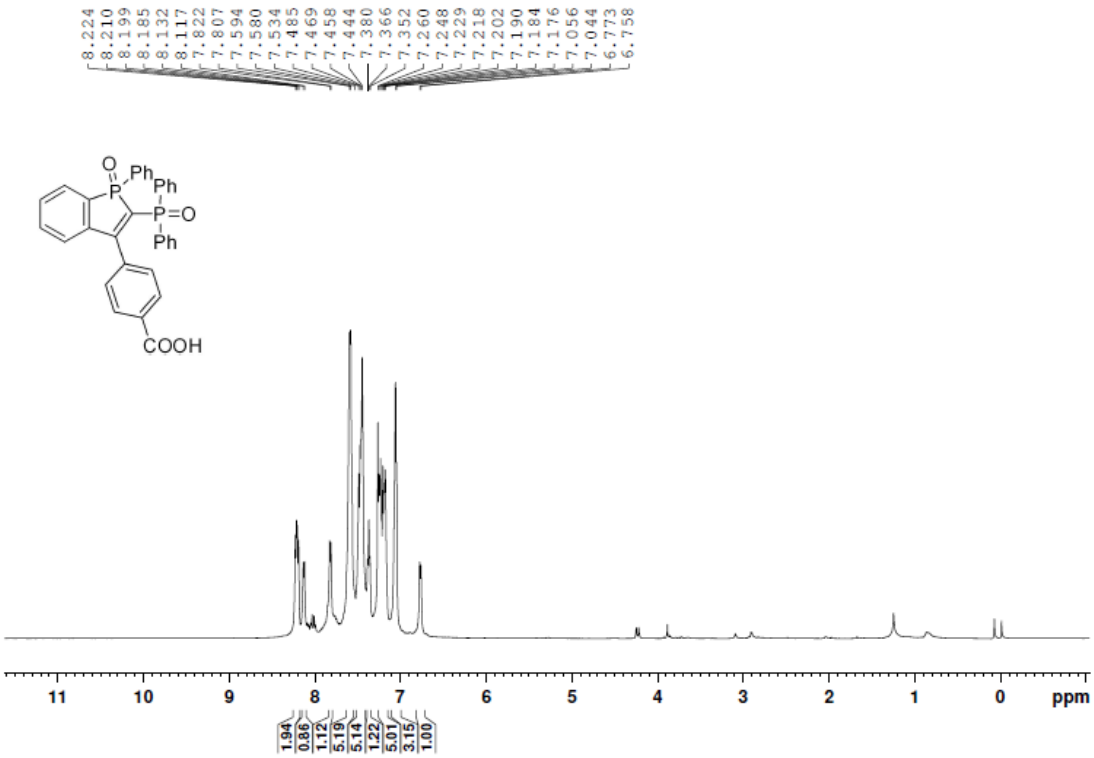

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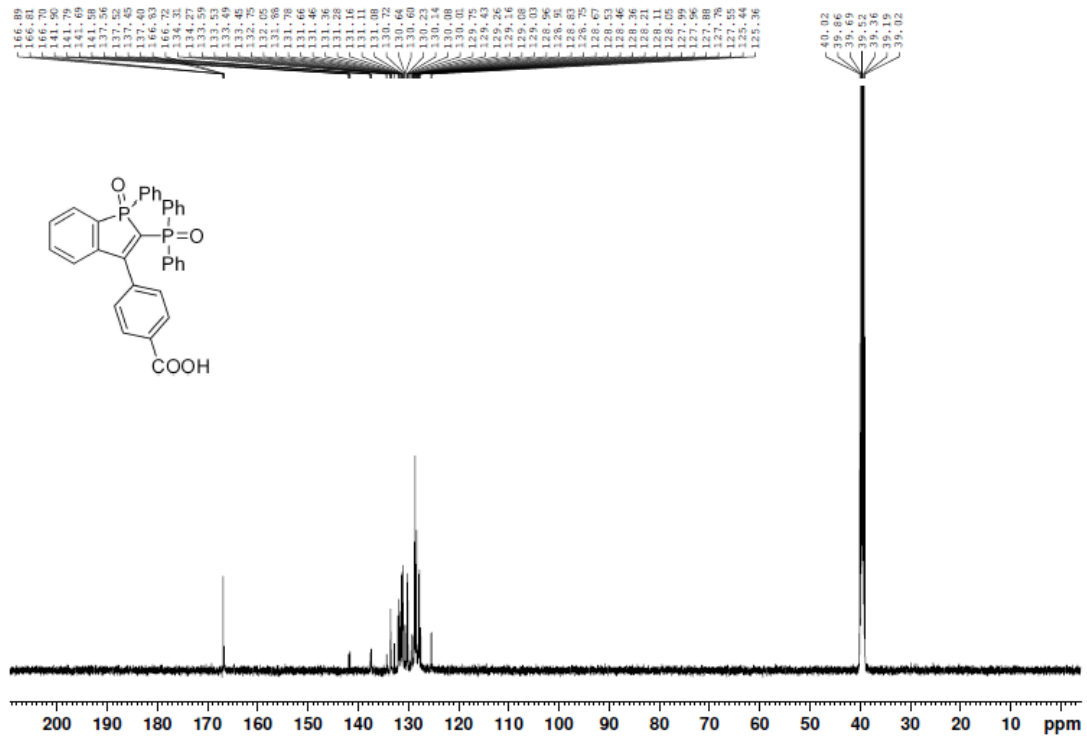

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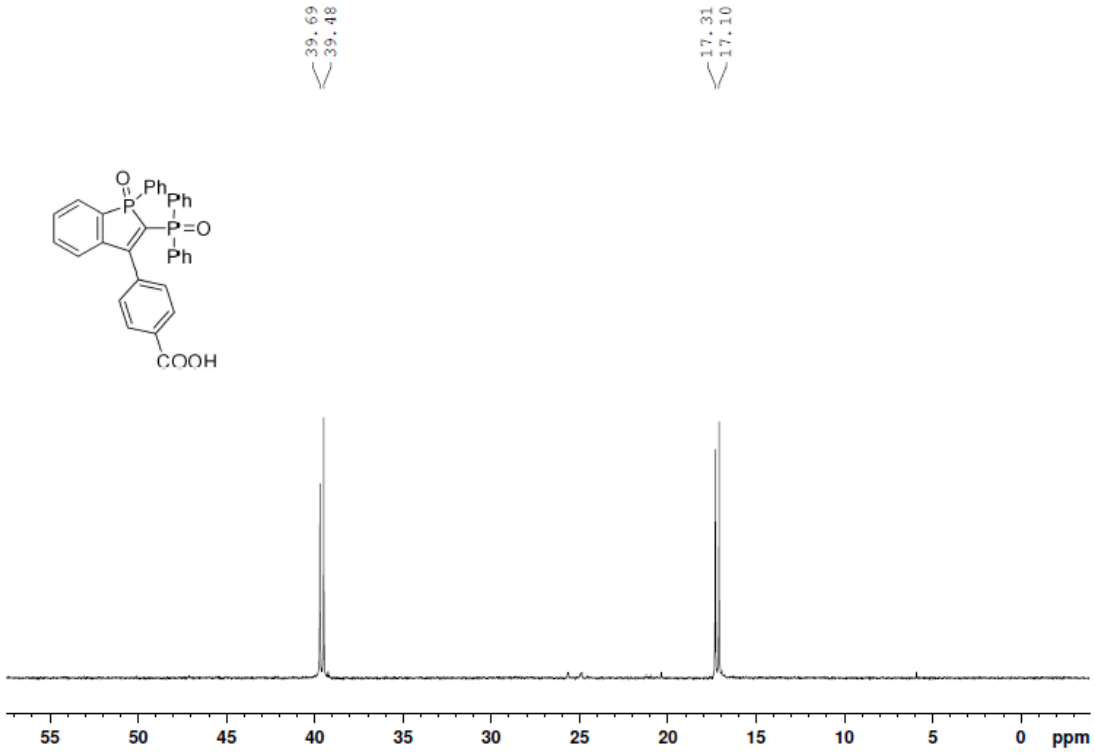

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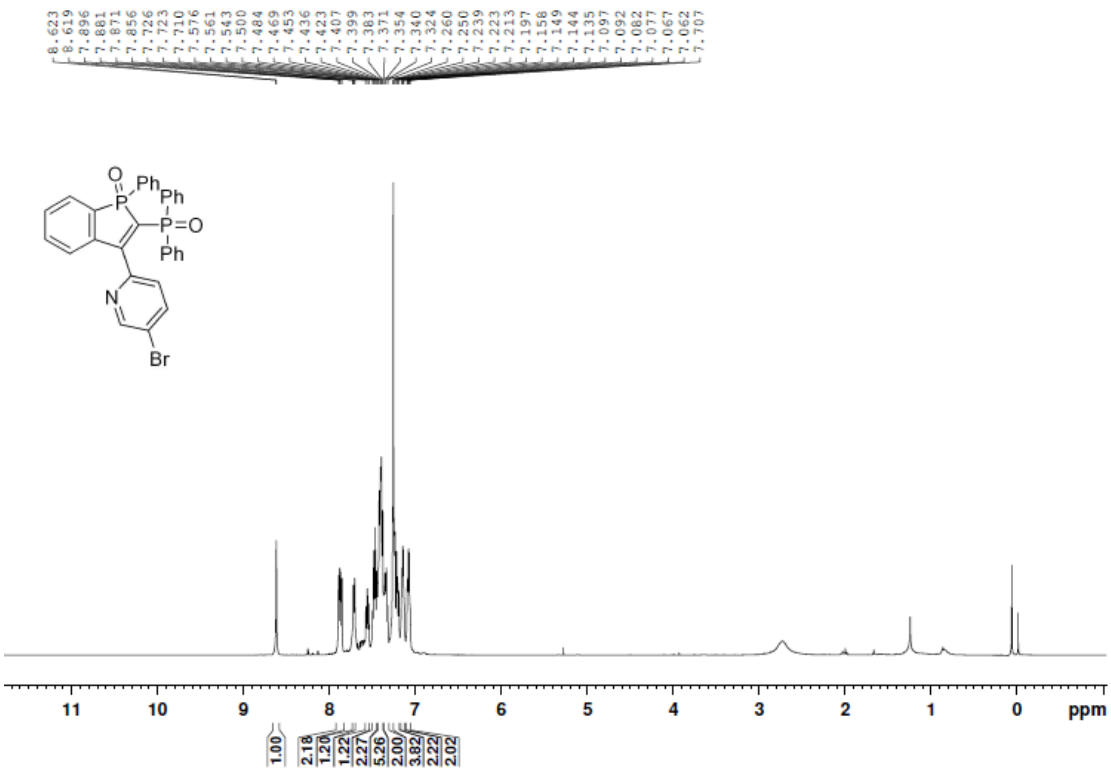

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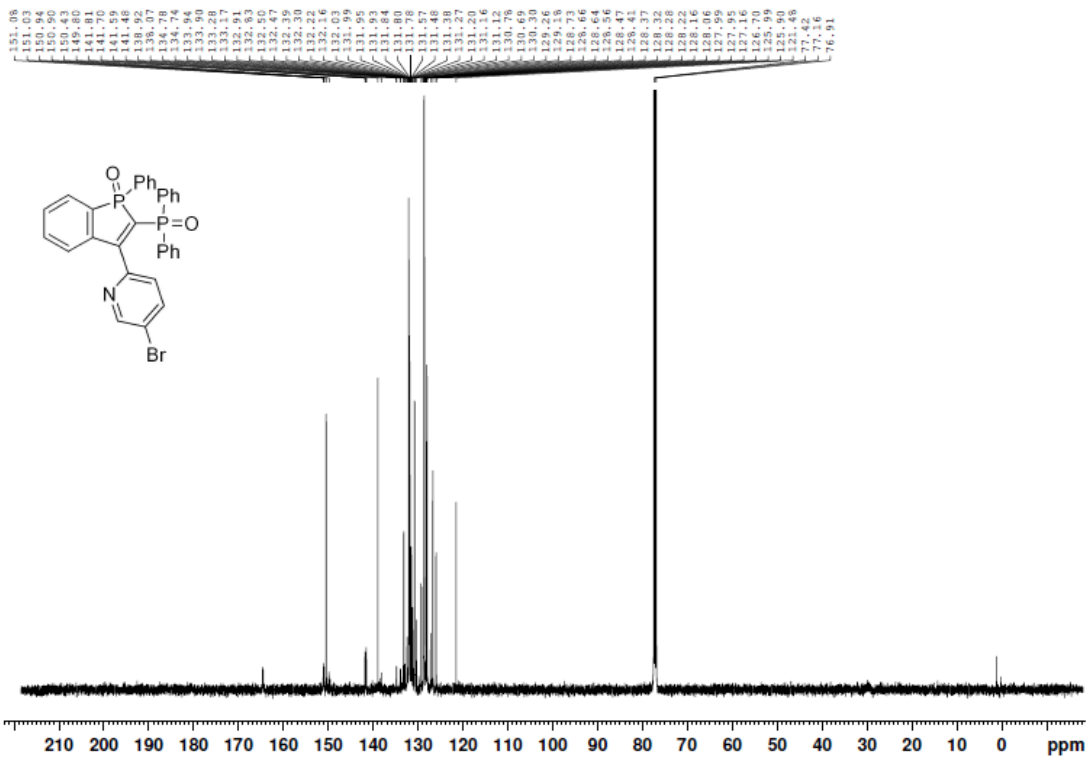

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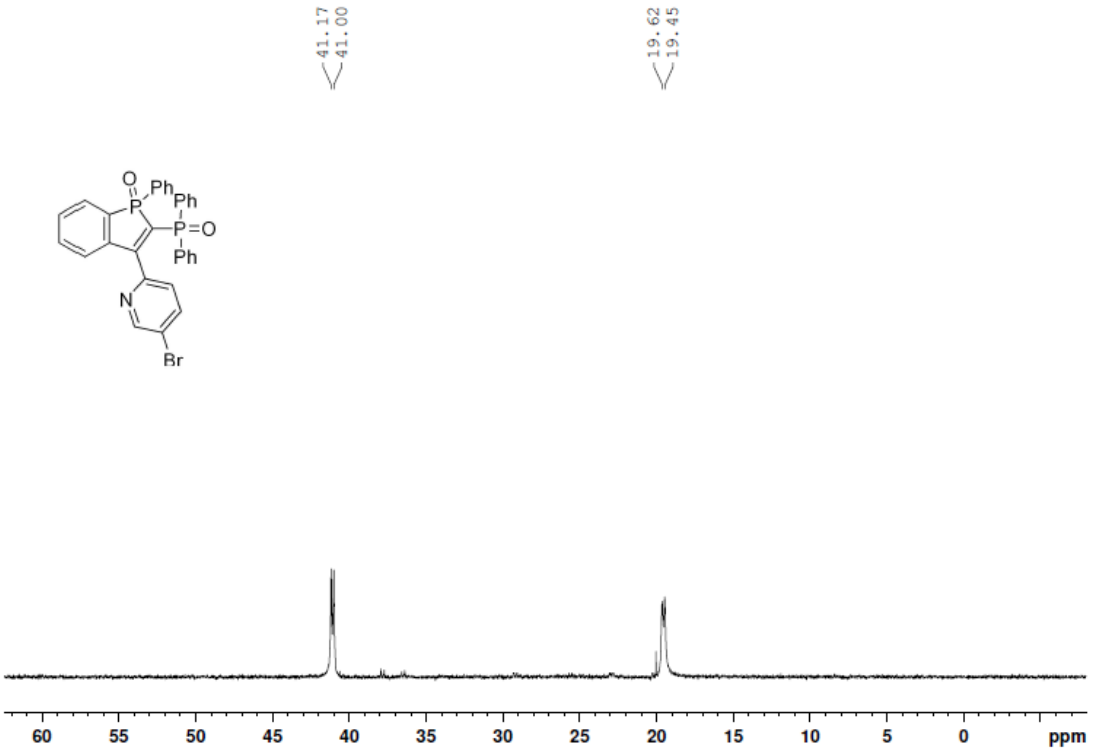

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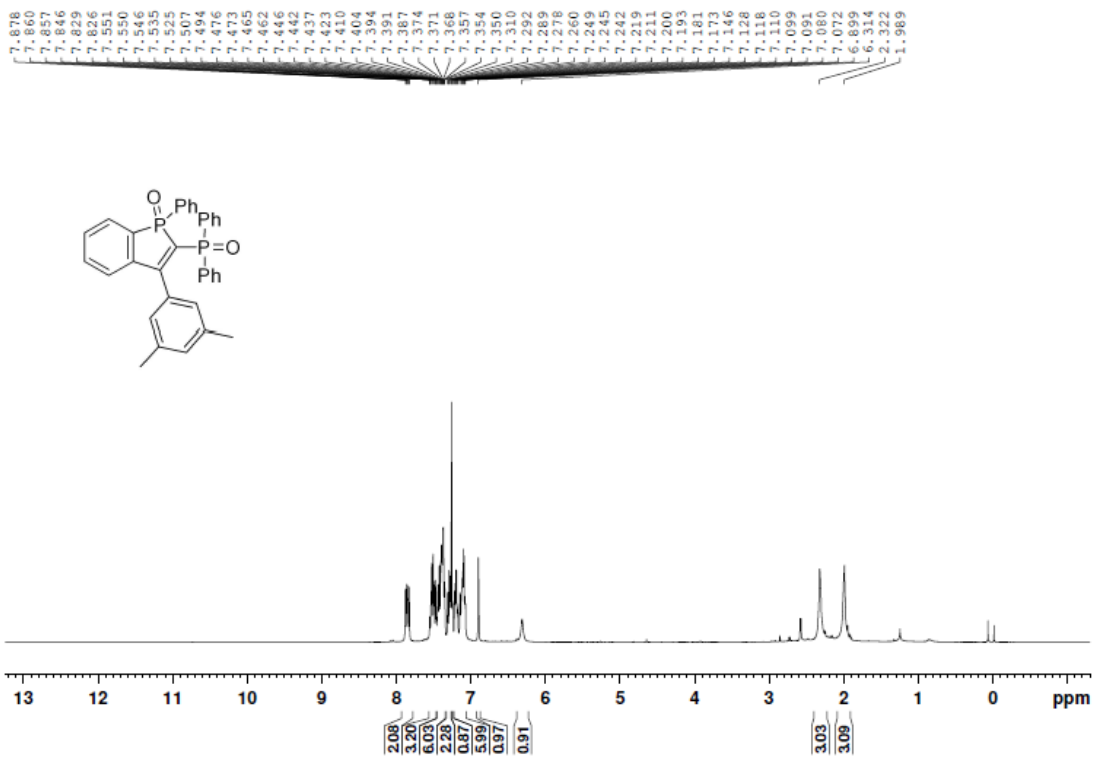
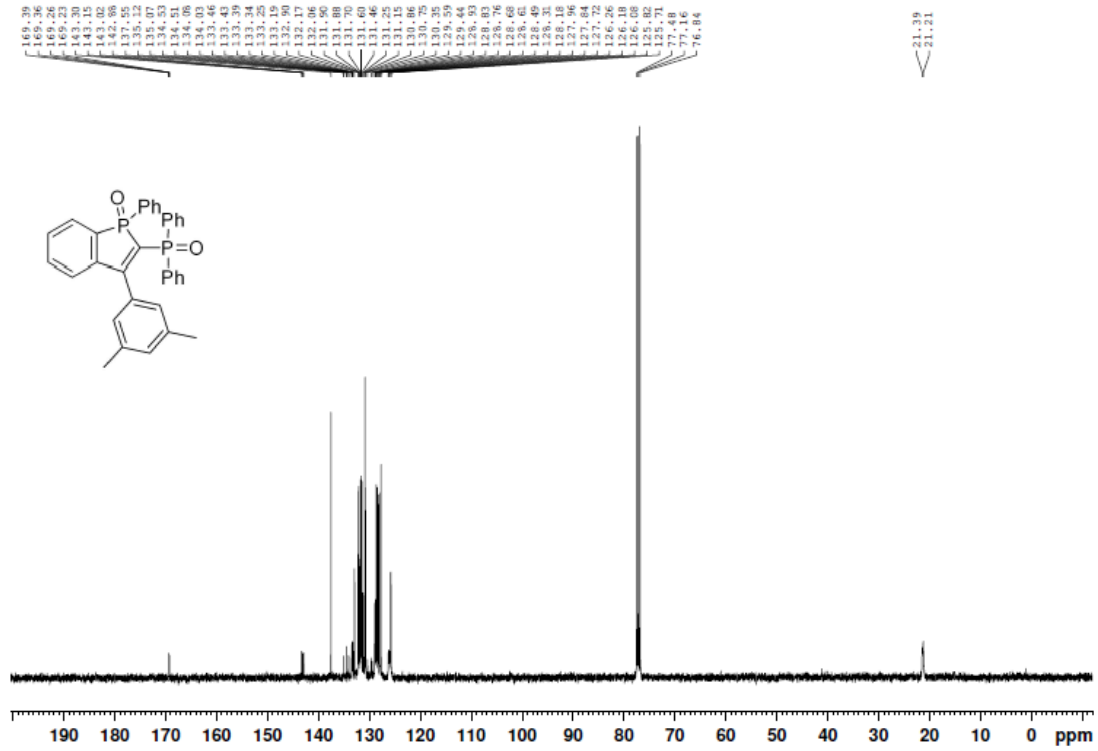
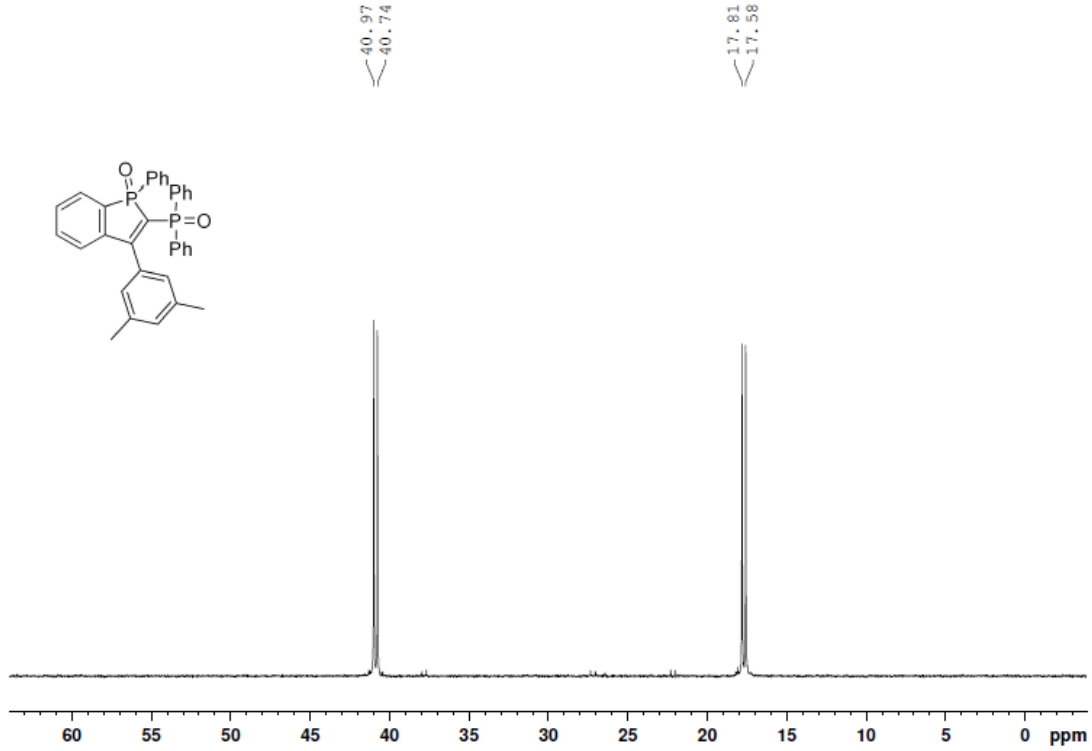


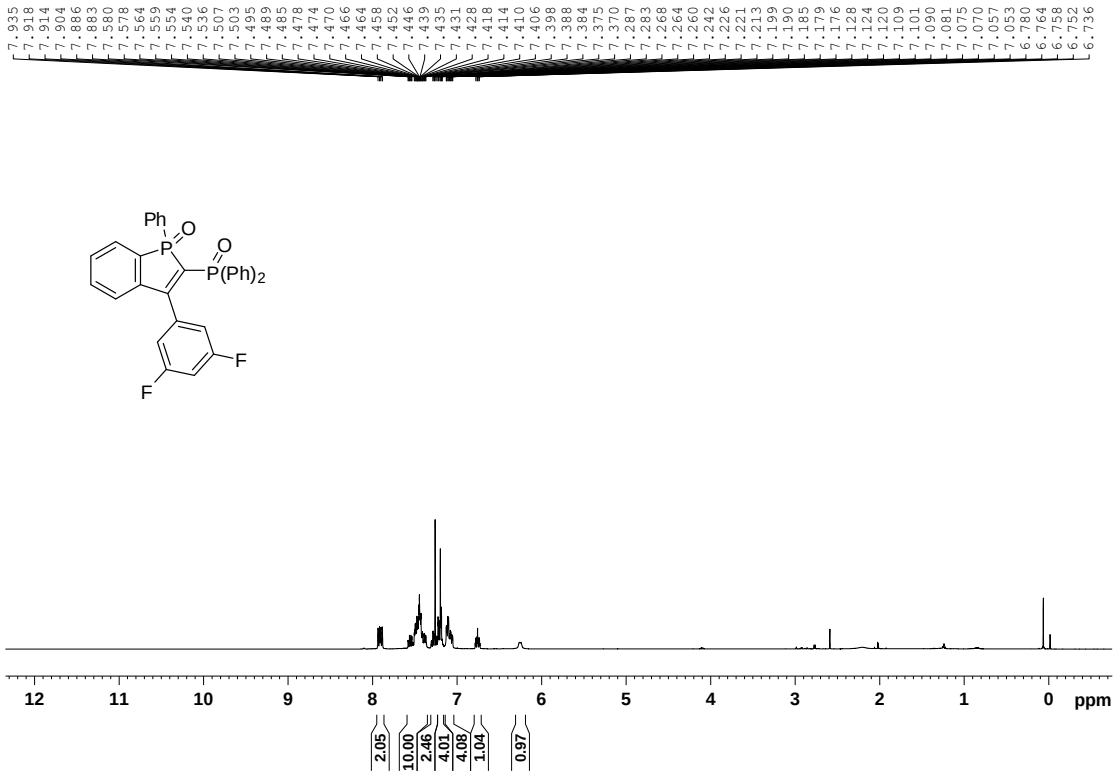
[13] C NMR of **3q**



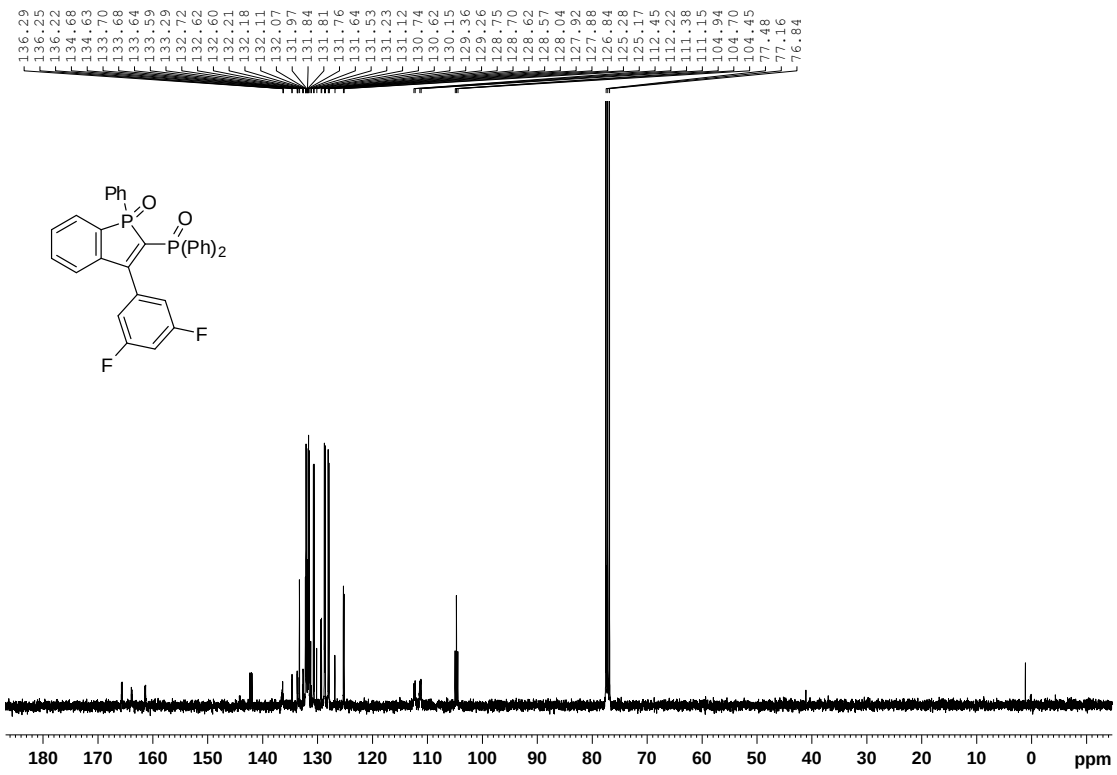
^{31} P NMR of **3q**



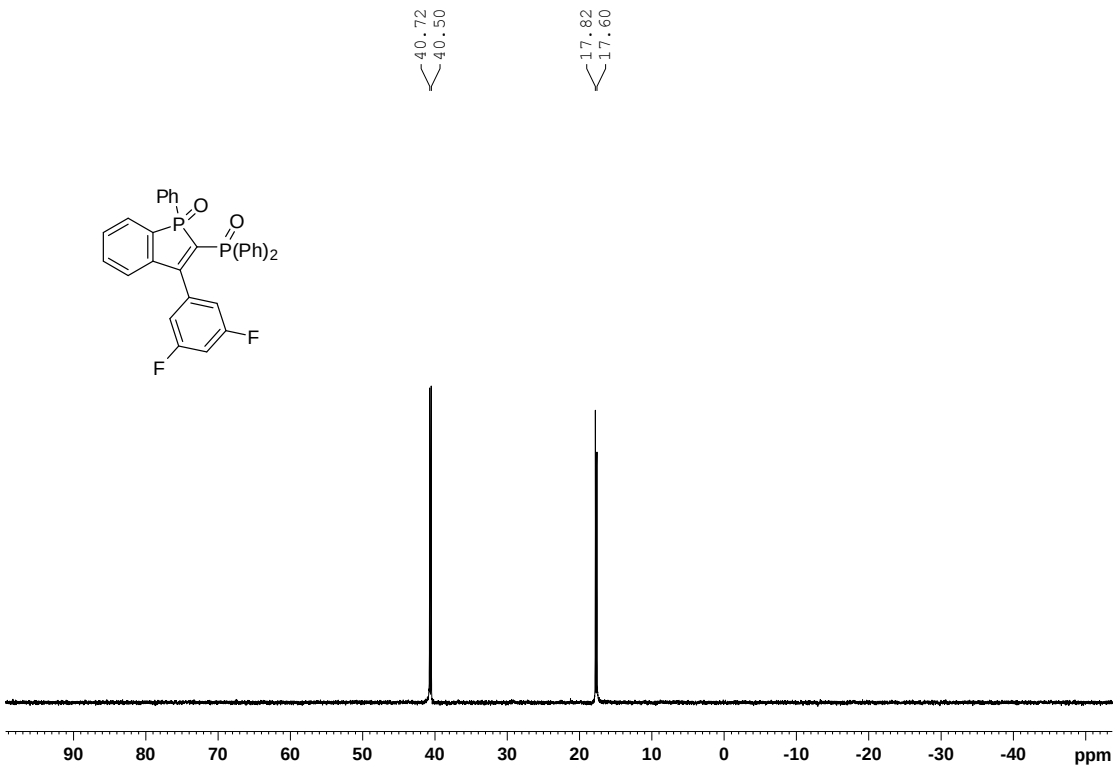
^{1} H NMR of **3r**



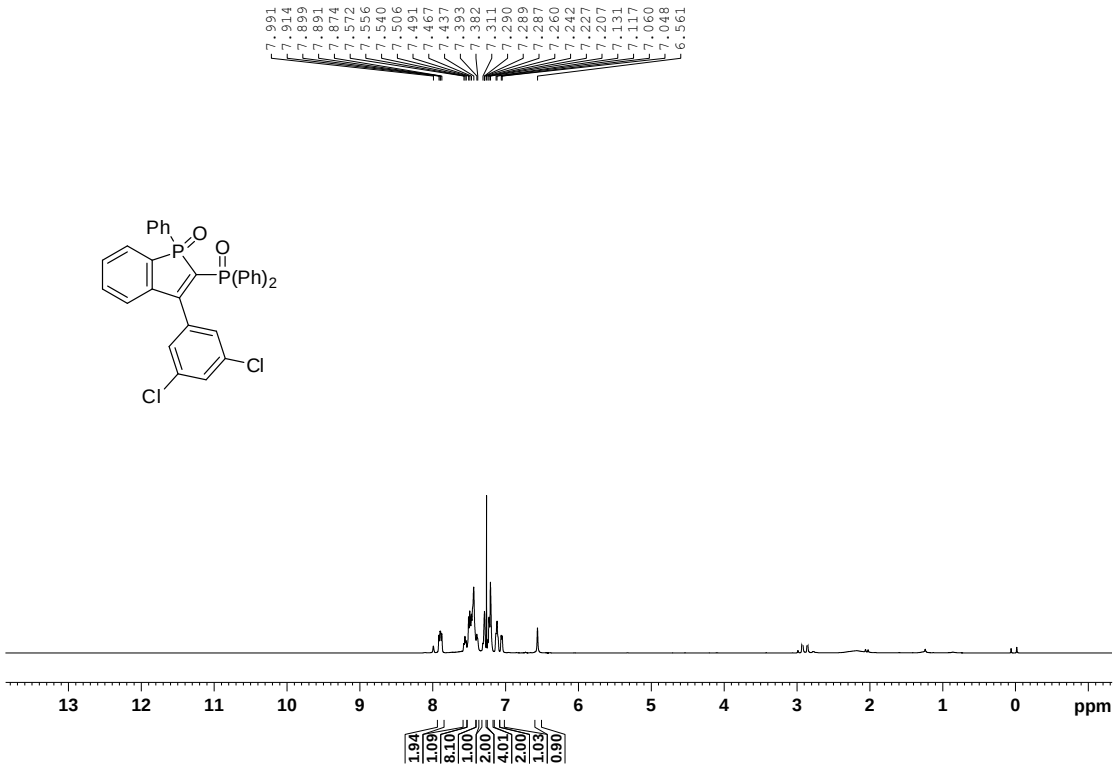
^{13} C NMR of **3r**



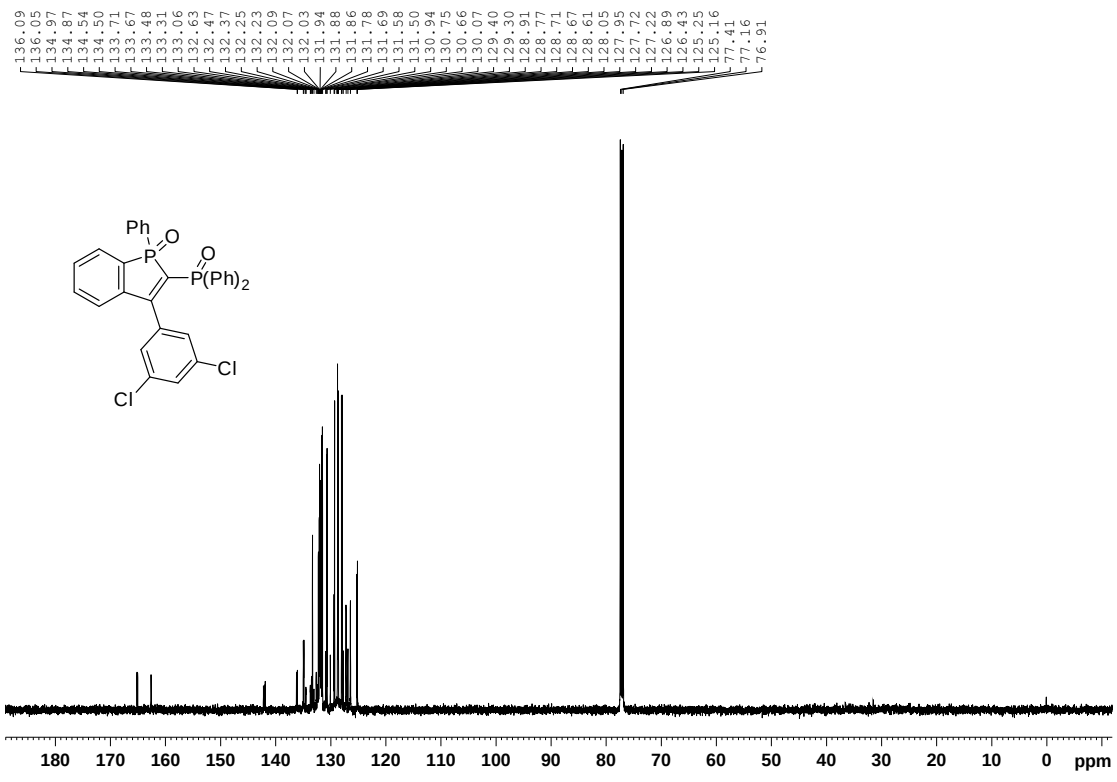
^{31} P NMR of **3r**



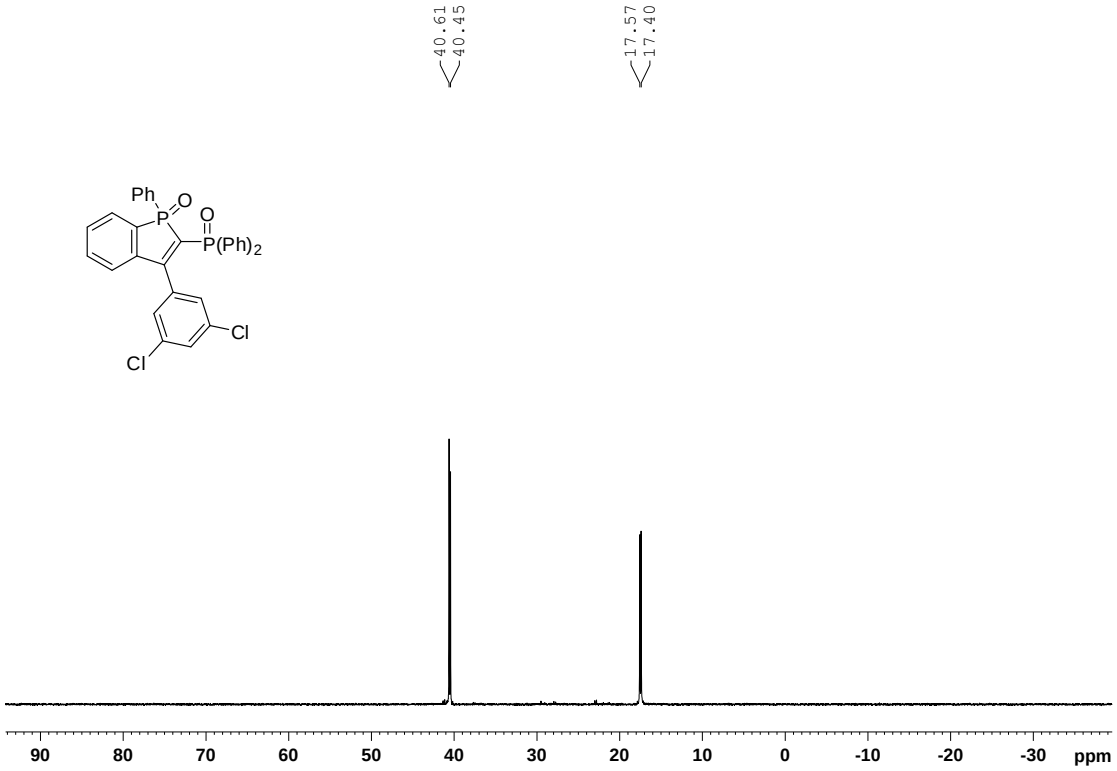
[1] H NMR of **3s**



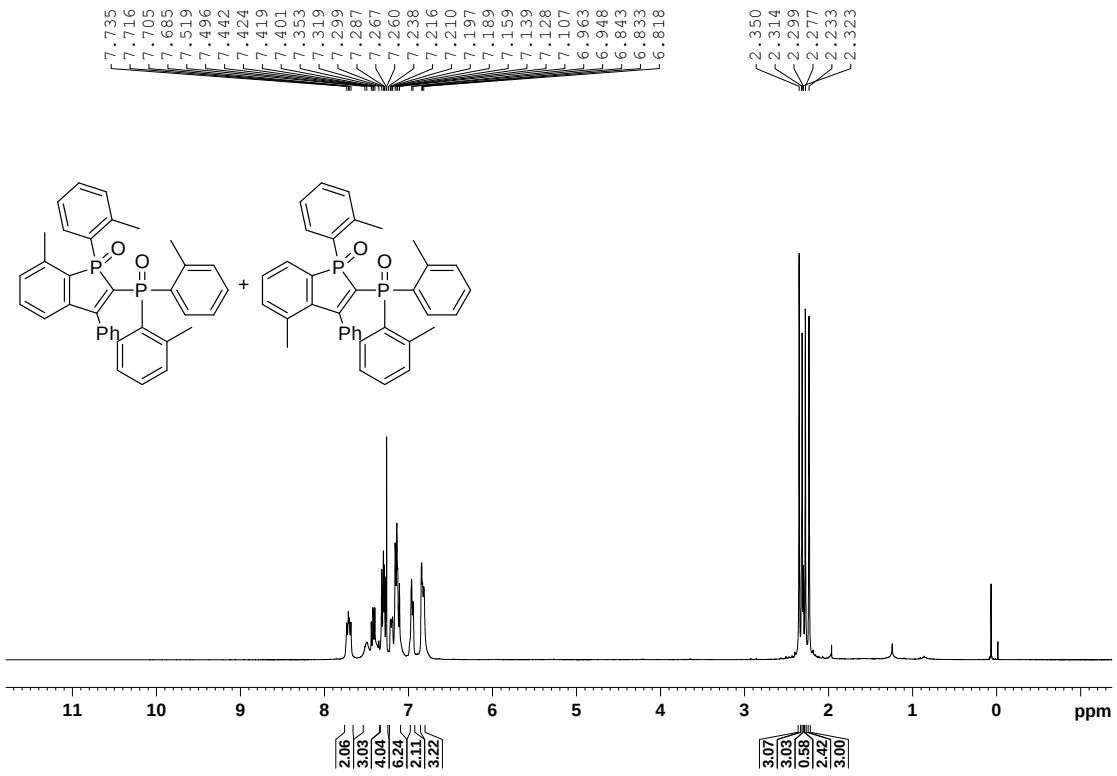
[13] C NMR of **3s**



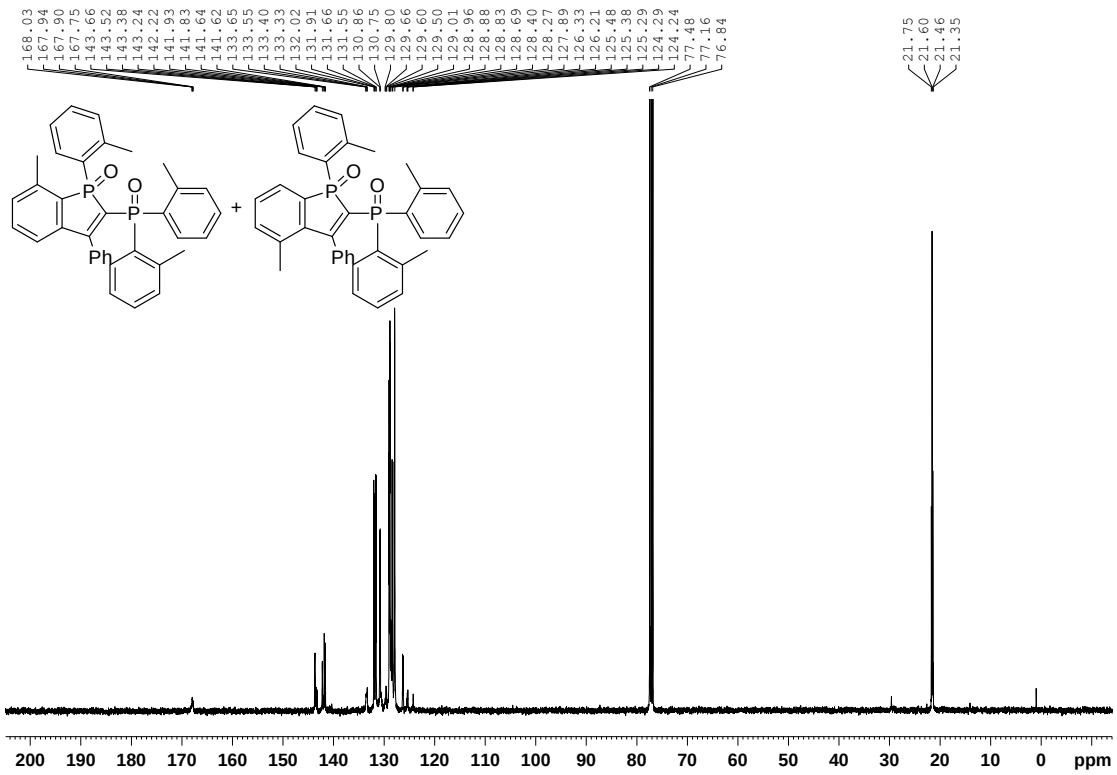
^{31} P NMR of **3s**



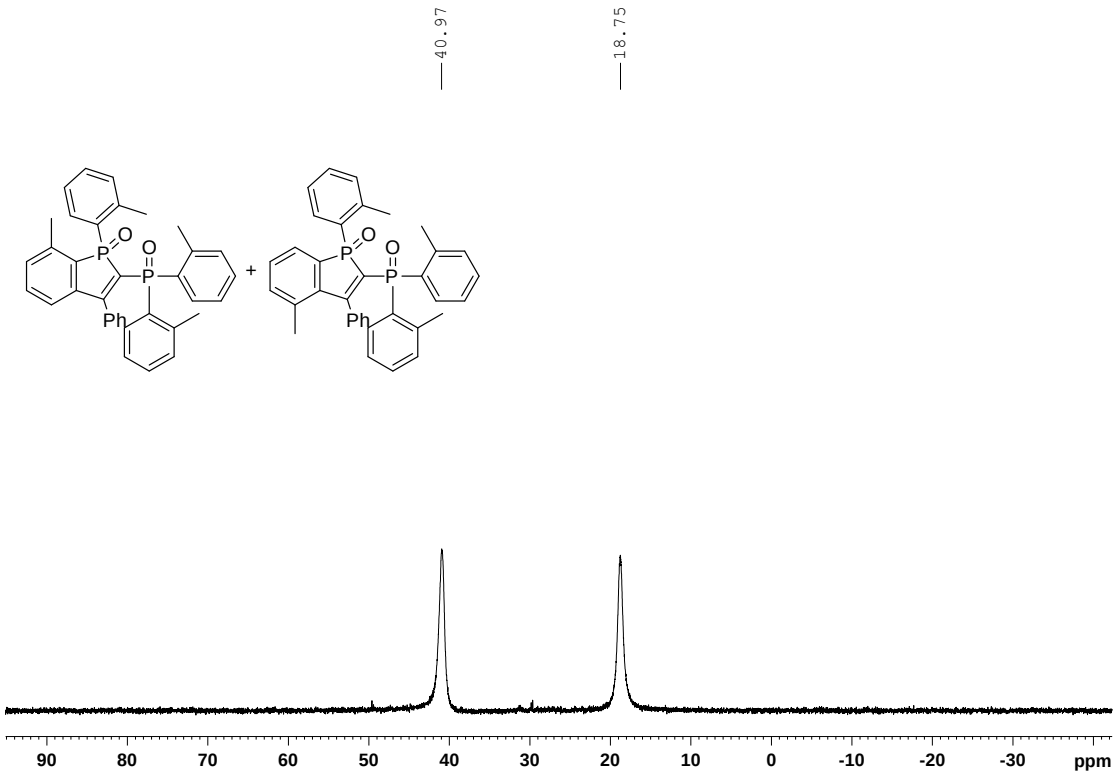
^{1} H NMR of **3t**



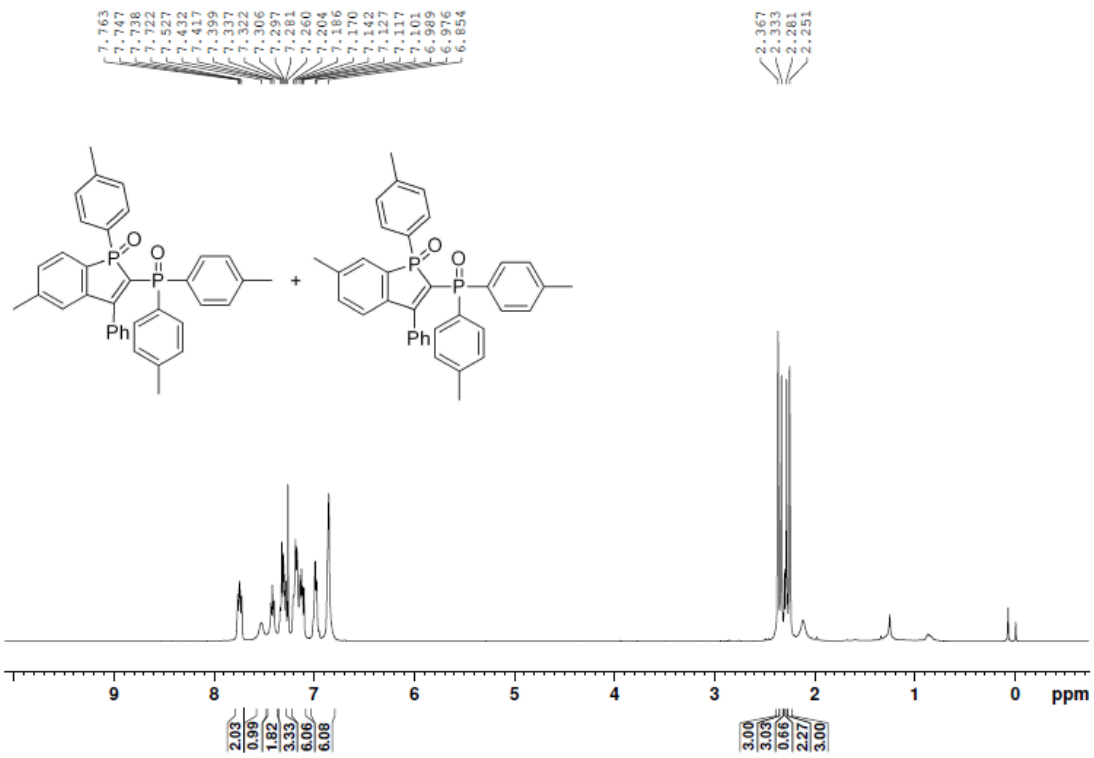
^{13}C NMR of **3t**



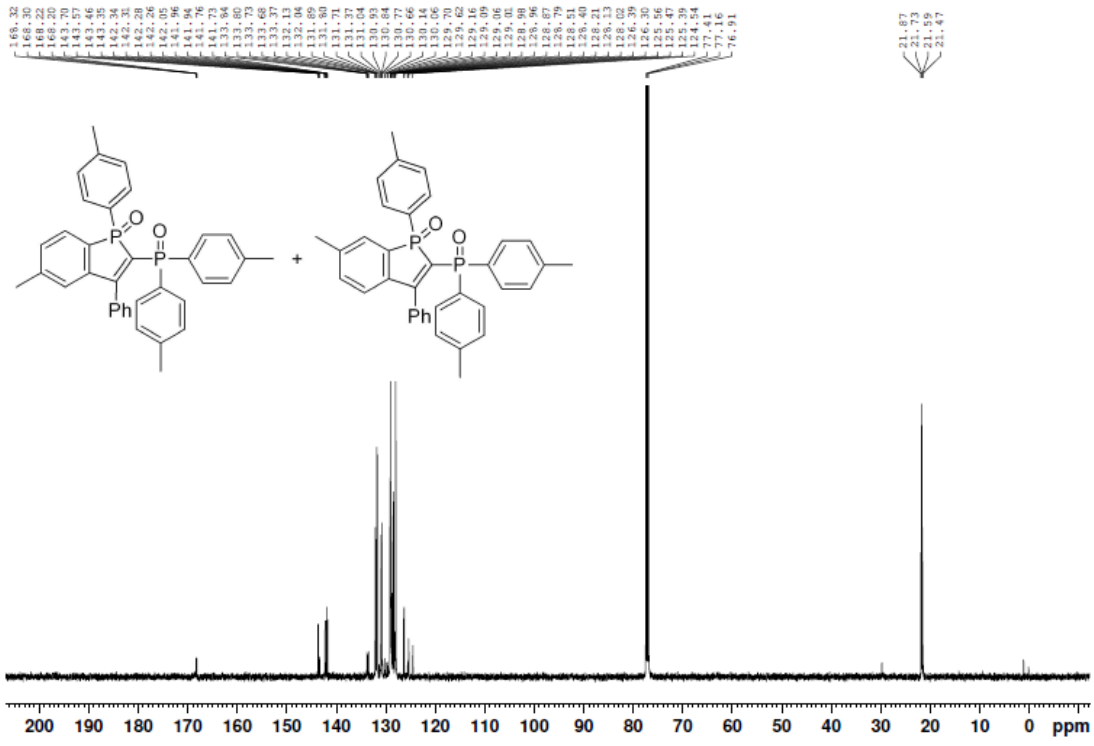
^{31}P NMR of **3t**



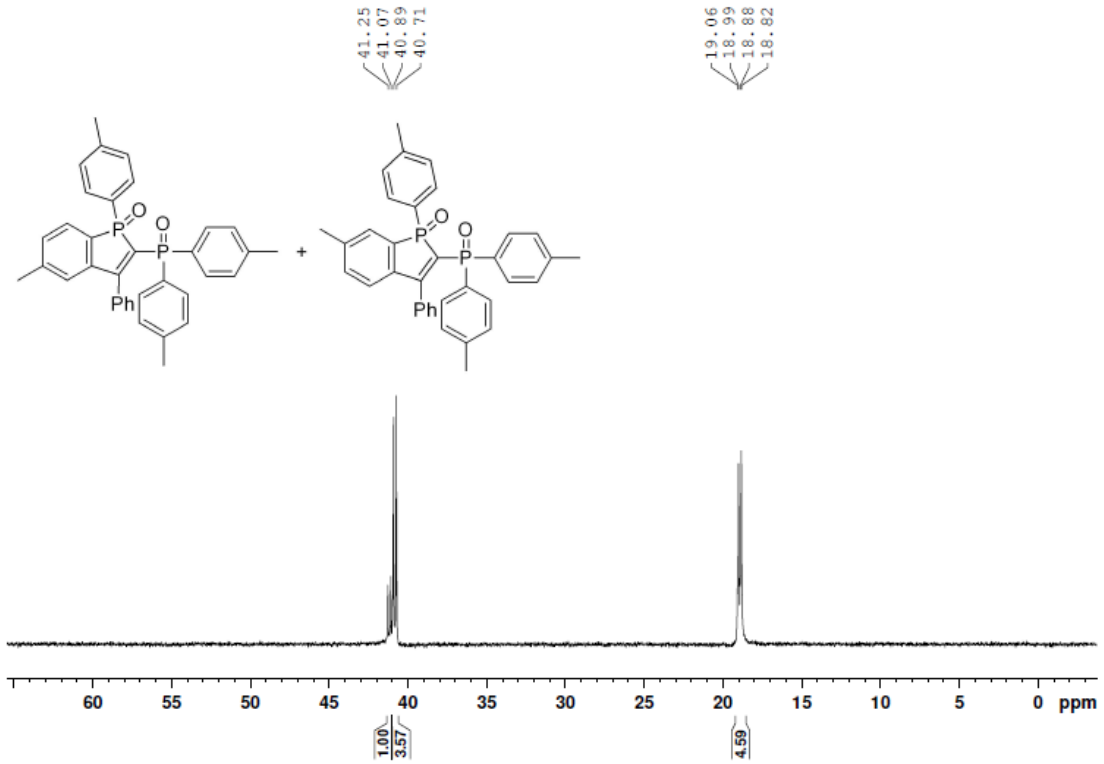
[1] H NMR of **3u**



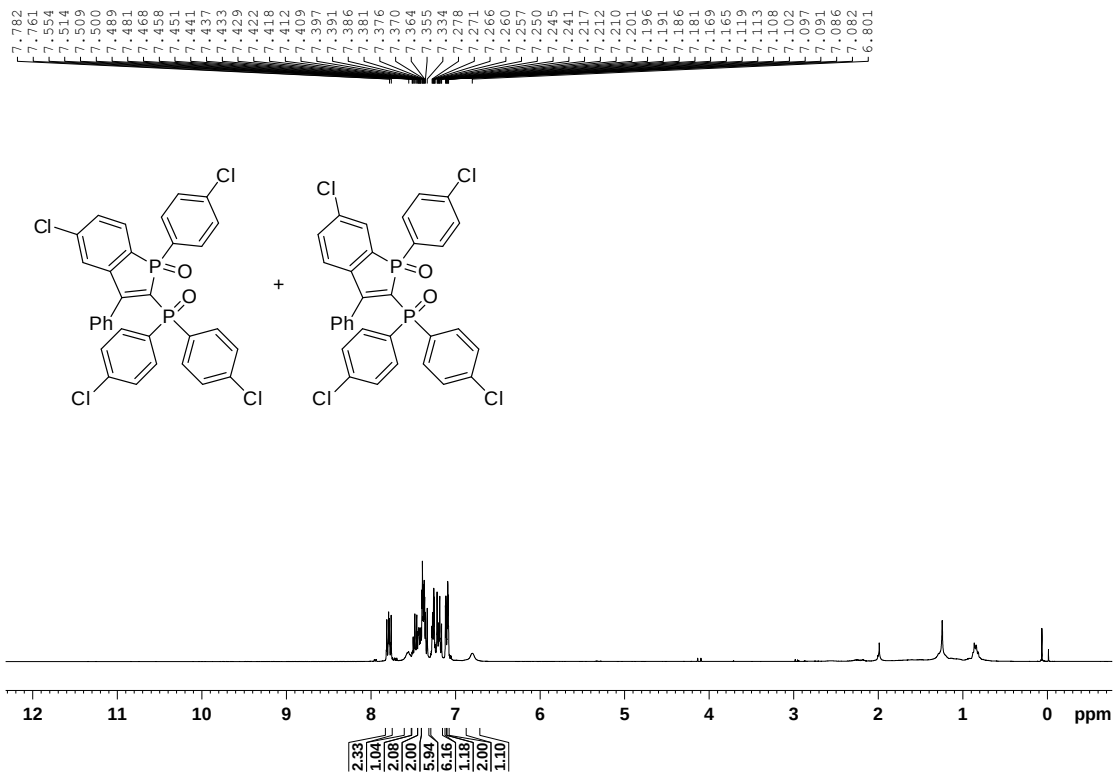
[13] C NMR of **3u**



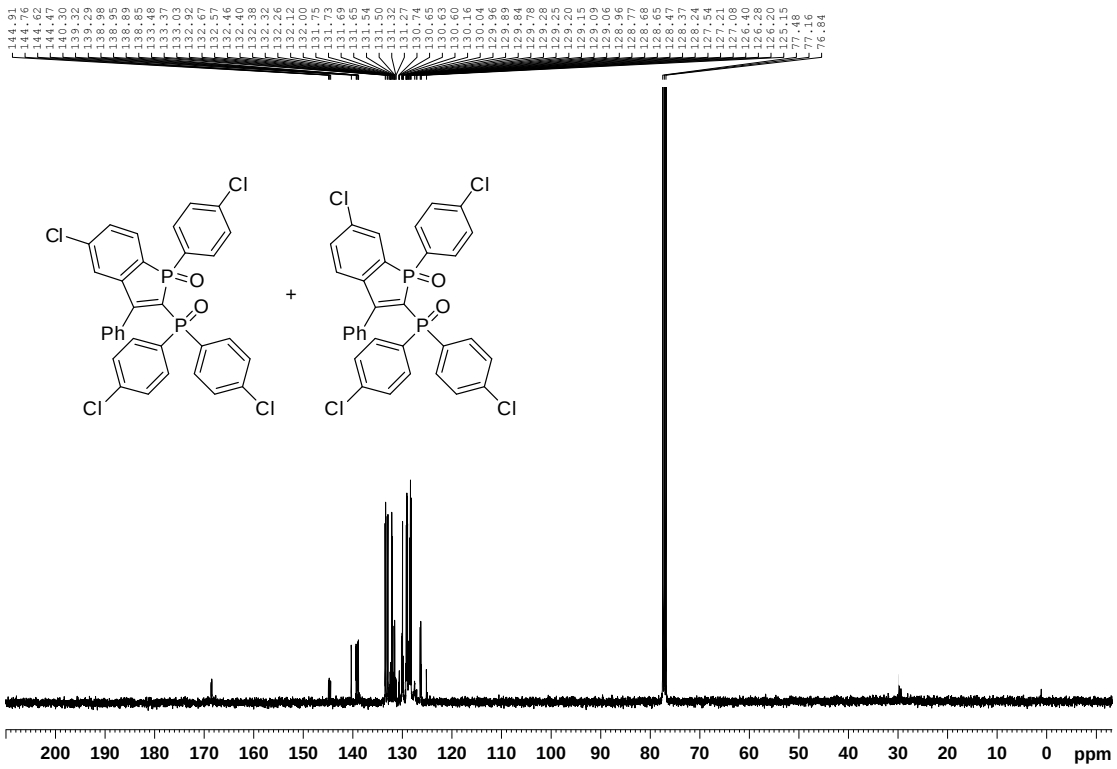
^{31} P NMR of **3u**



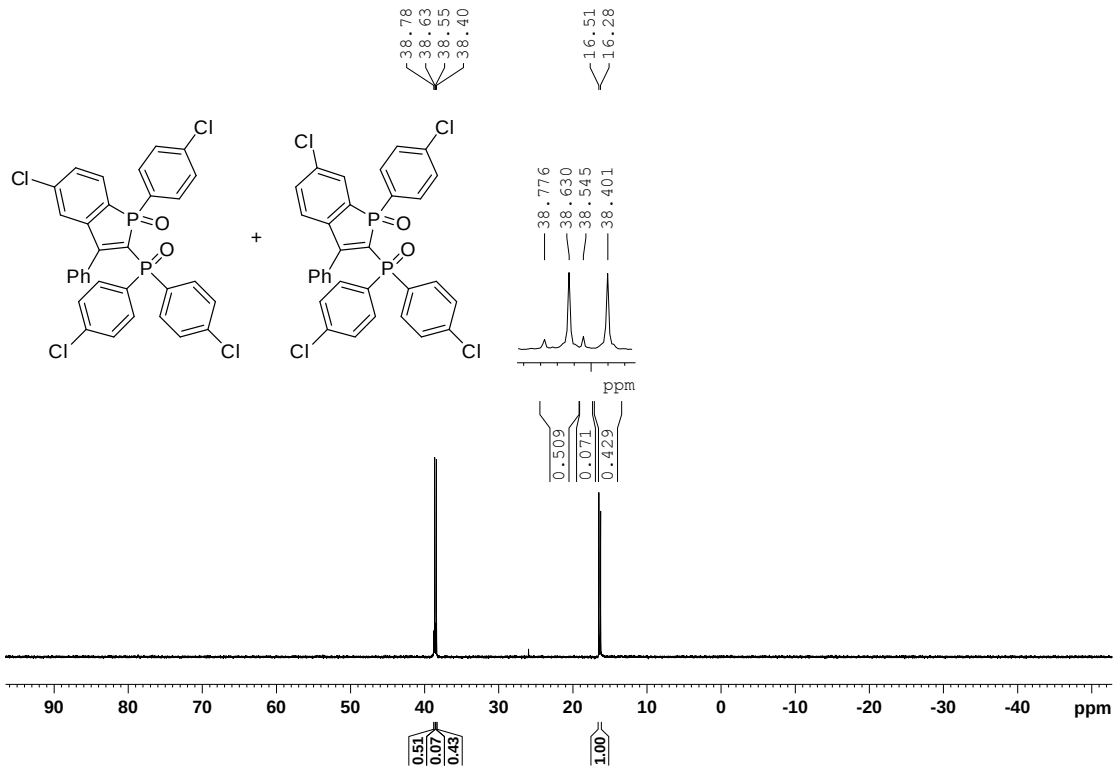
^{1} H NMR of **3v**



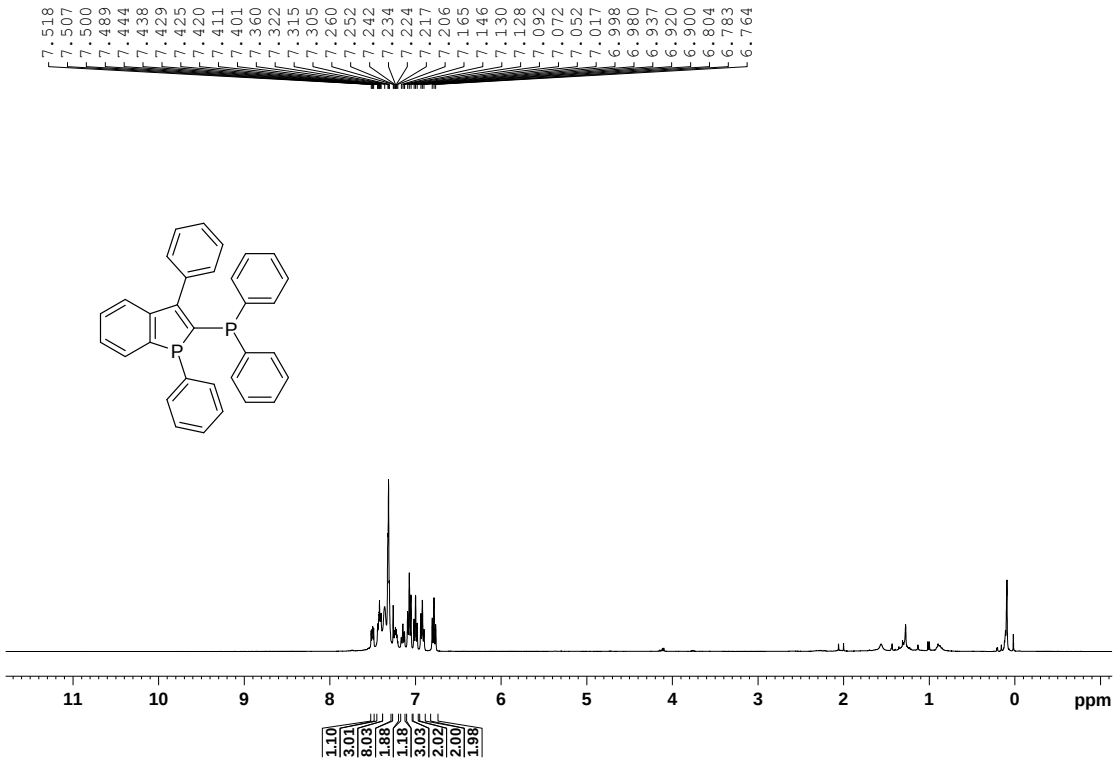
^{13}C NMR of **3v**



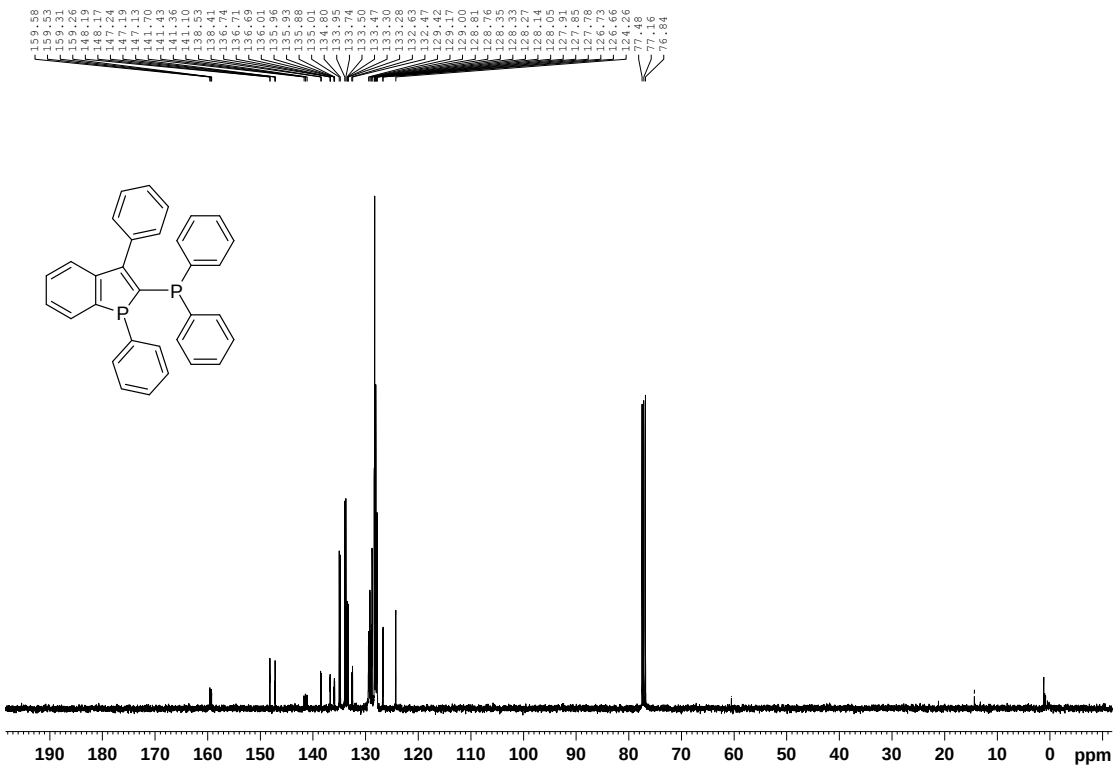
^{31}P NMR of **3v**



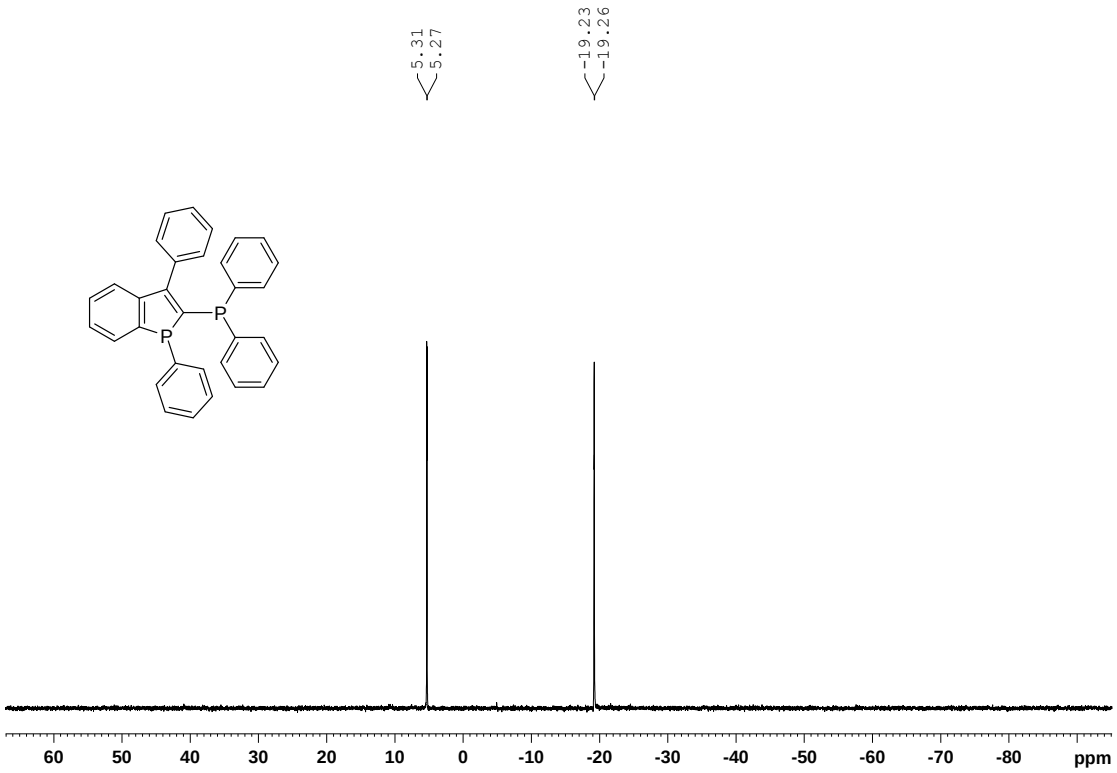
^{1}H NMR of **5**



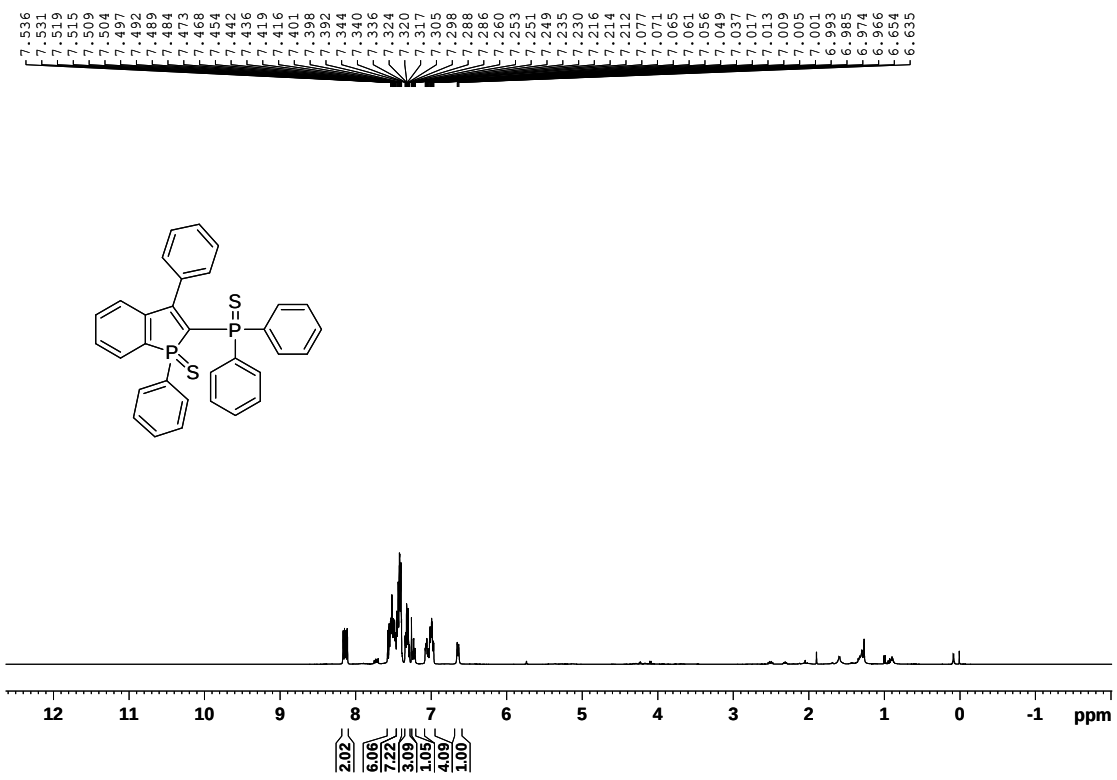
^{13}C NMR of **5**



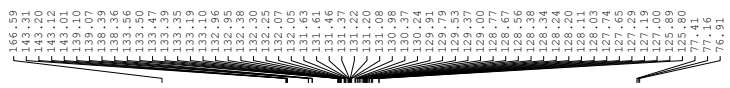
^{31} P NMR of **5**

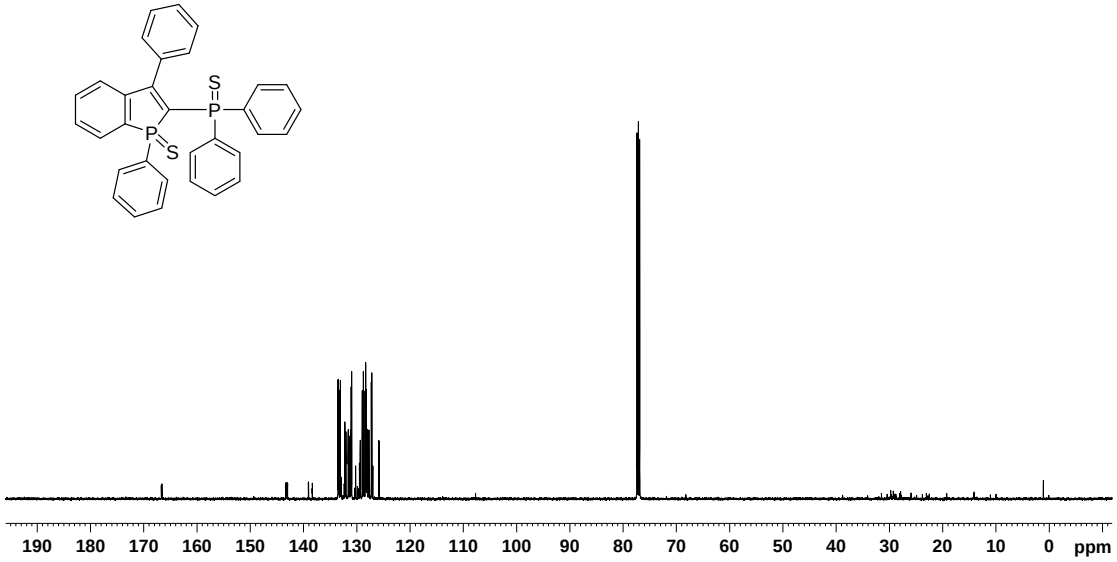


^{1} H NMR of **6**



^{13} C NMR of **6**





^{31} P NMR of **6**