

Supporting Information

Isocyanide Cycloaddition and Coordination Processes at Trigonal Phosphinidene-Bridged MoRe and MoMn Complexes

M. Angeles Alvarez, M. Esther García, Daniel García-Vivó,* Miguel A. Ruiz,* and Patricia Vega

Departamento de Química Orgánica e Inorgánica/IUQOEM, Universidad de Oviedo, E-33071 Oviedo, Spain.

Corresponding Author E-mail: garciavdaniel@uniovi.es (D.G.-V.); mara@uniovi.es (M.A.R.)

Table S1. Crystal Data for New Compounds

5a.1	
mol formula	C ₃₇ H ₄₁ MoNO ₇ Pre
mol wt	924.82
cryst syst	triclinic
space group	<i>P</i> −1
radiation (λ , Å)	1.54184
<i>a</i> , Å	8.9166(2)
<i>b</i> , Å	12.7525(4)
<i>c</i> , Å	17.1297(5)
α , deg	75.851(3)
β , deg	82.477(2)
γ , deg	87.540(2)
<i>V</i> , Å ³	1872.33(9)
<i>Z</i>	2
calcd density, g cm ^{−3}	1.640
absorp coeff, mm ^{−1}	9.756
temperature, K	153(4)
θ range (deg)	3.5700 / 69.4730
index ranges (<i>h</i> , <i>k</i> , <i>l</i>)	−10, 10; −15, 15 −18, 20
no. of reflns collected	18192
no. of indep reflns (<i>R</i> _{int})	6940(0.0548)
reflns with $I > 2\sigma(I)$	6250
<i>R</i> indexes [data with $I > 2\sigma(I)$] ^a	<i>R</i> ₁ = 0.0428 w <i>R</i> ₂ = 0.1131 ^b
<i>R</i> indexes (all data) ^a	<i>R</i> ₁ = 0.0467 w <i>R</i> ₂ = 0.1173 ^b
GOF	1.035
no. of restraints/params	0 / 419
$\Delta\rho$ (max., min.), eÅ ^{−3}	1.736 / −1.225
CCDC deposition no	2283058

^a $R = \sum ||F_o| - |F_c|| / \sum |F_o|$. $wR = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w|F_o|^2]^{1/2}$. $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where $P = (F_o^2 + 2F_c^2)/3$. ^b $a = 0.0746$, $b = 0.9740$.

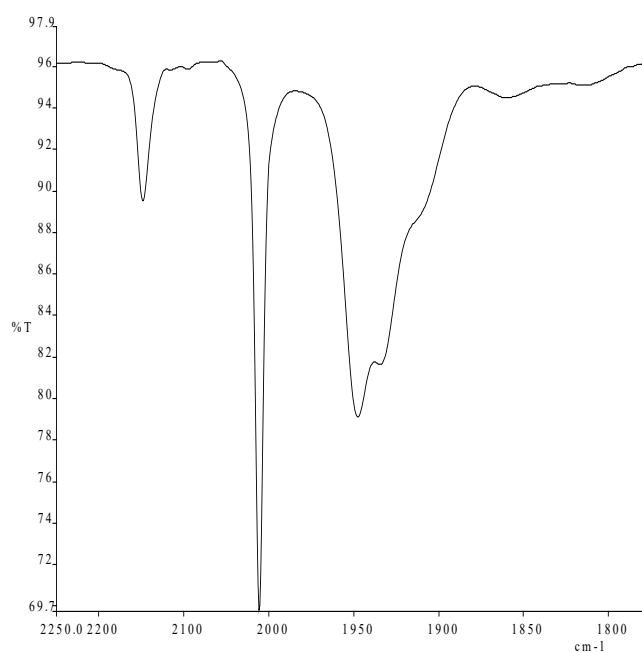


Figure S1. IR spectrum of compound **2a.1** in dichloromethane solution.

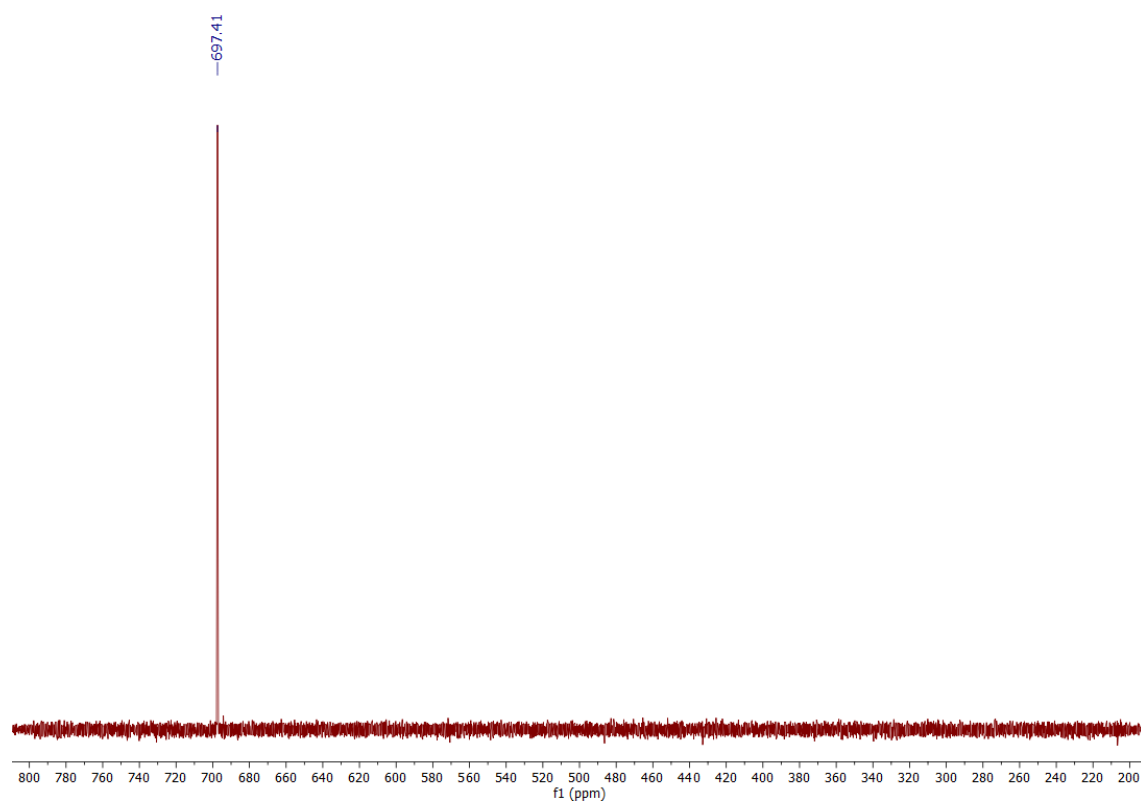


Figure S2. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **2a.1** (CD_2Cl_2).

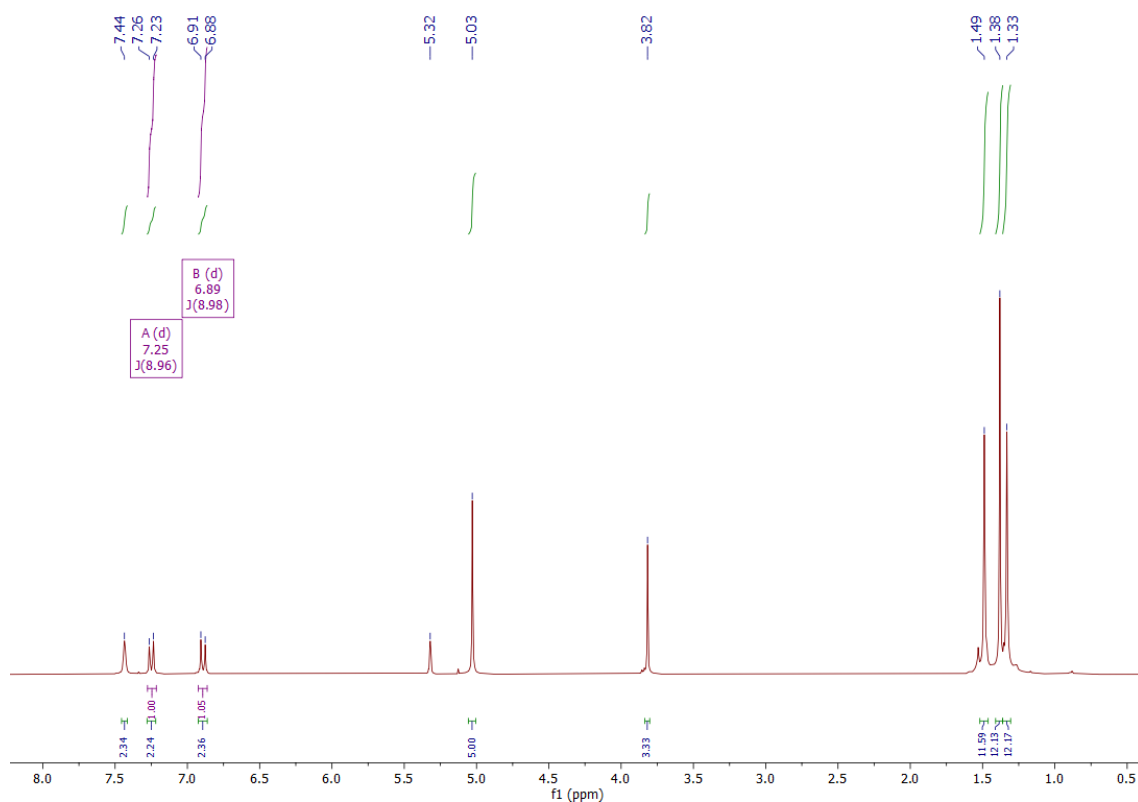


Figure S3. ¹H NMR spectrum of compound **2a.1** (CD₂Cl₂).

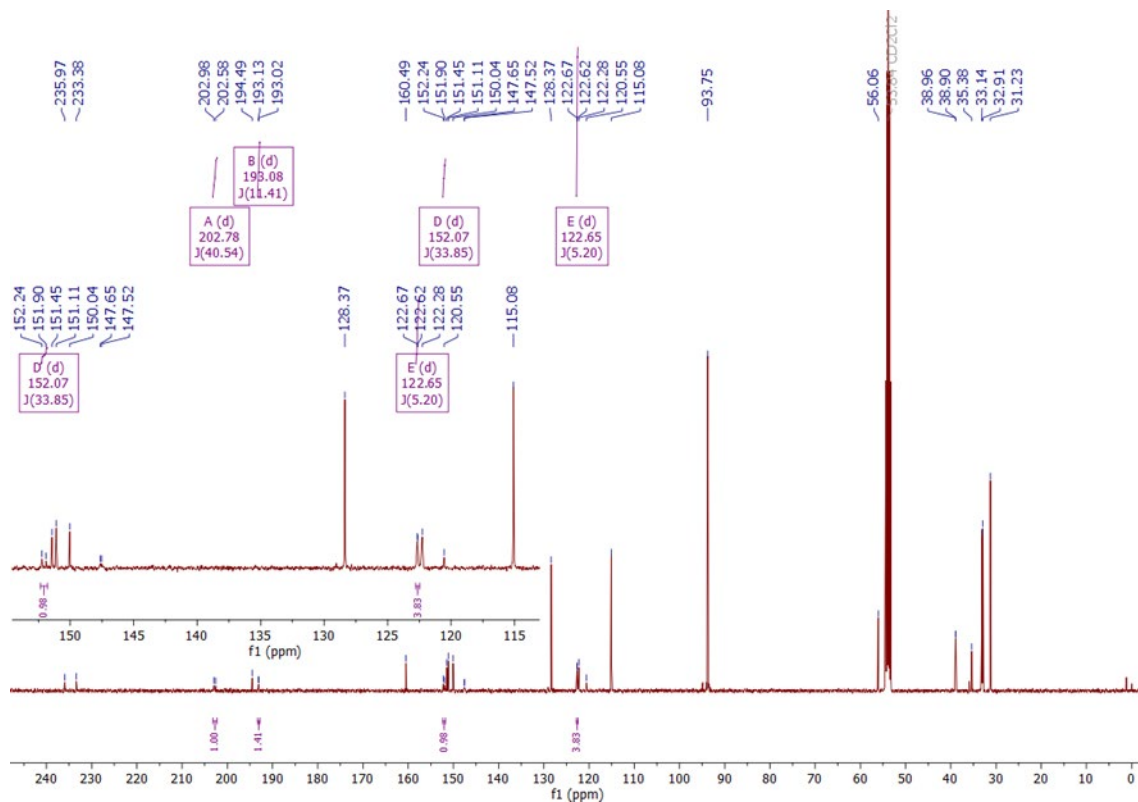


Figure S4. ¹³C{¹H} NMR spectrum of compound **2a.1** (CD₂Cl₂).

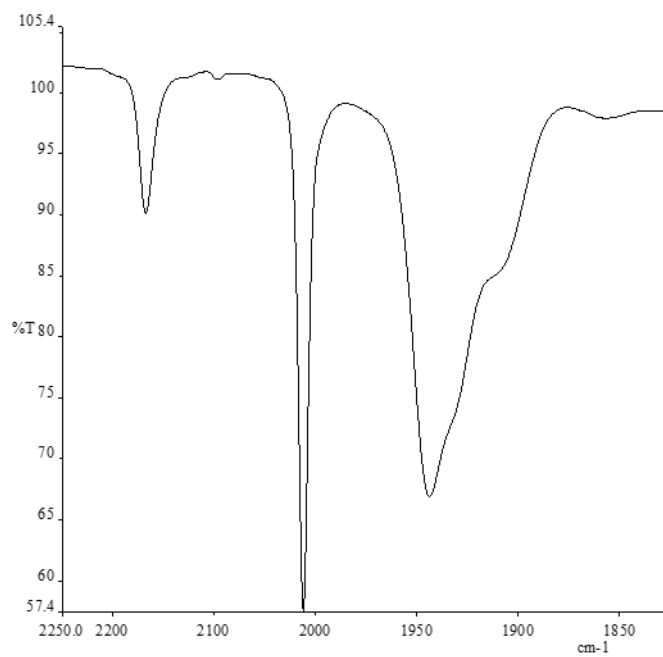


Figure S5. IR spectrum of compound **2a.2** in dichloromethane solution.

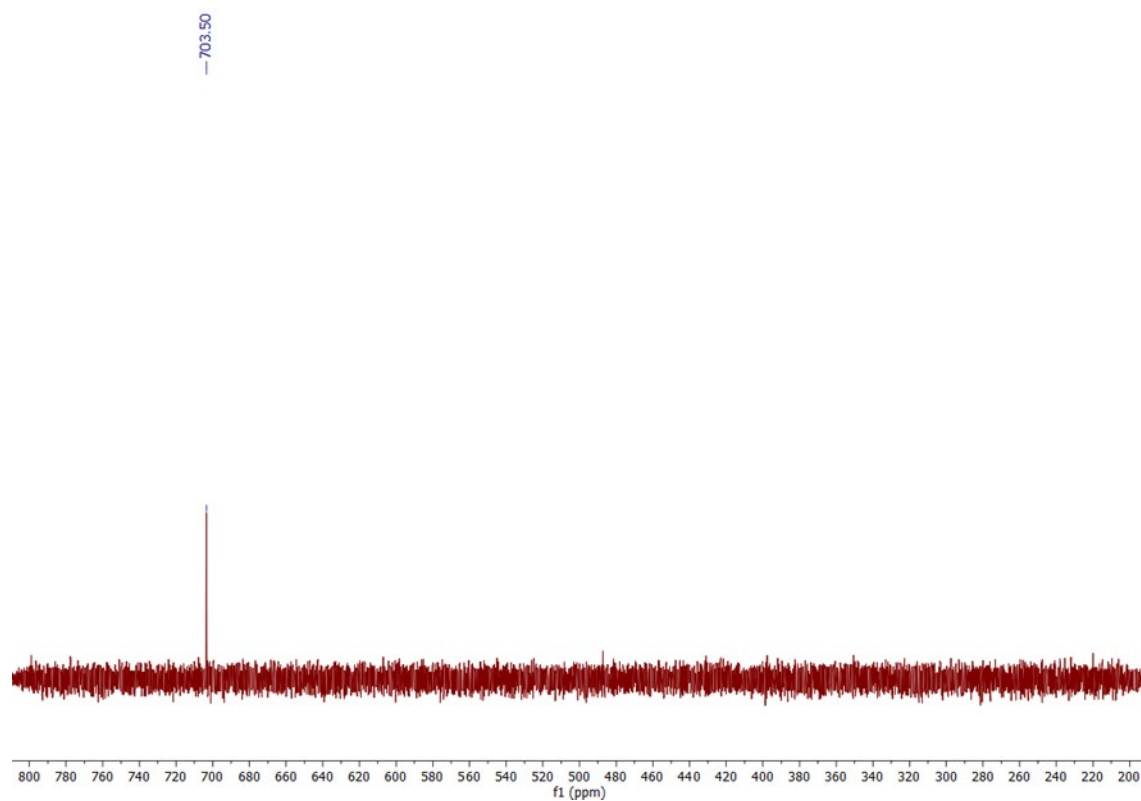


Figure S6. ³¹P{¹H} NMR spectrum of compound **2a.2** (CD₂Cl₂).

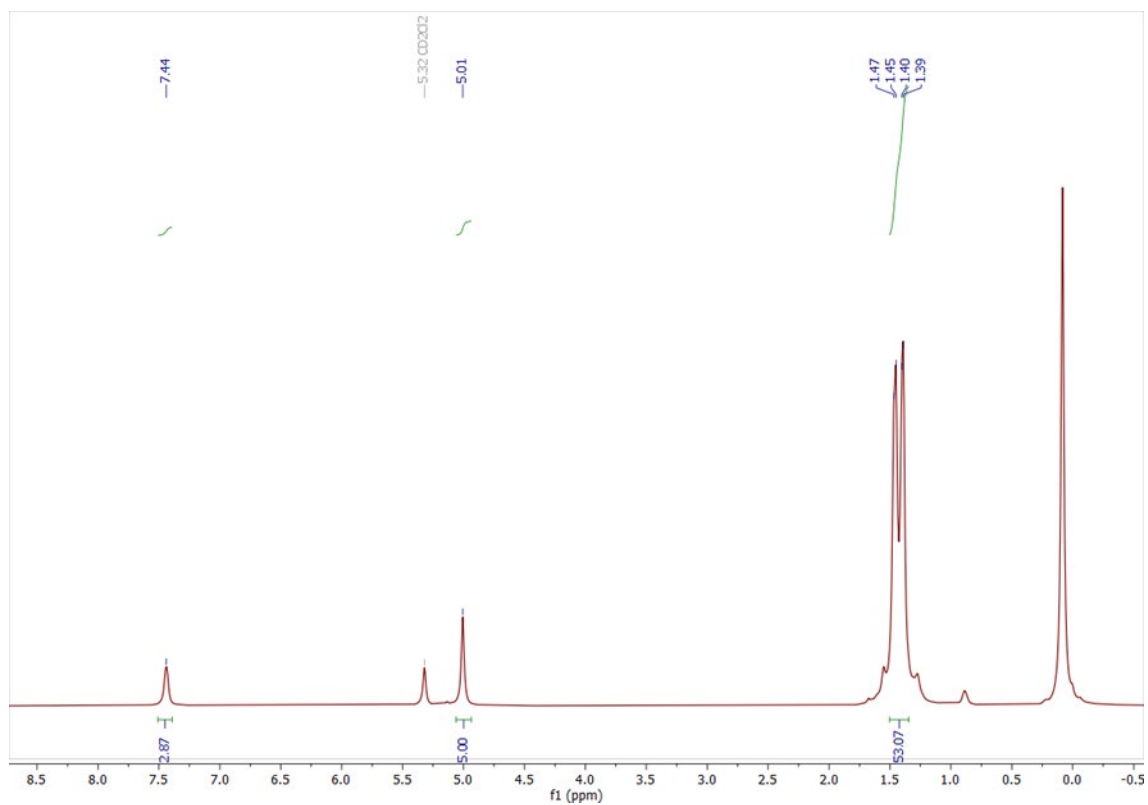


Figure S7. ¹H NMR spectrum of compound **2a.2** (CD₂Cl₂).

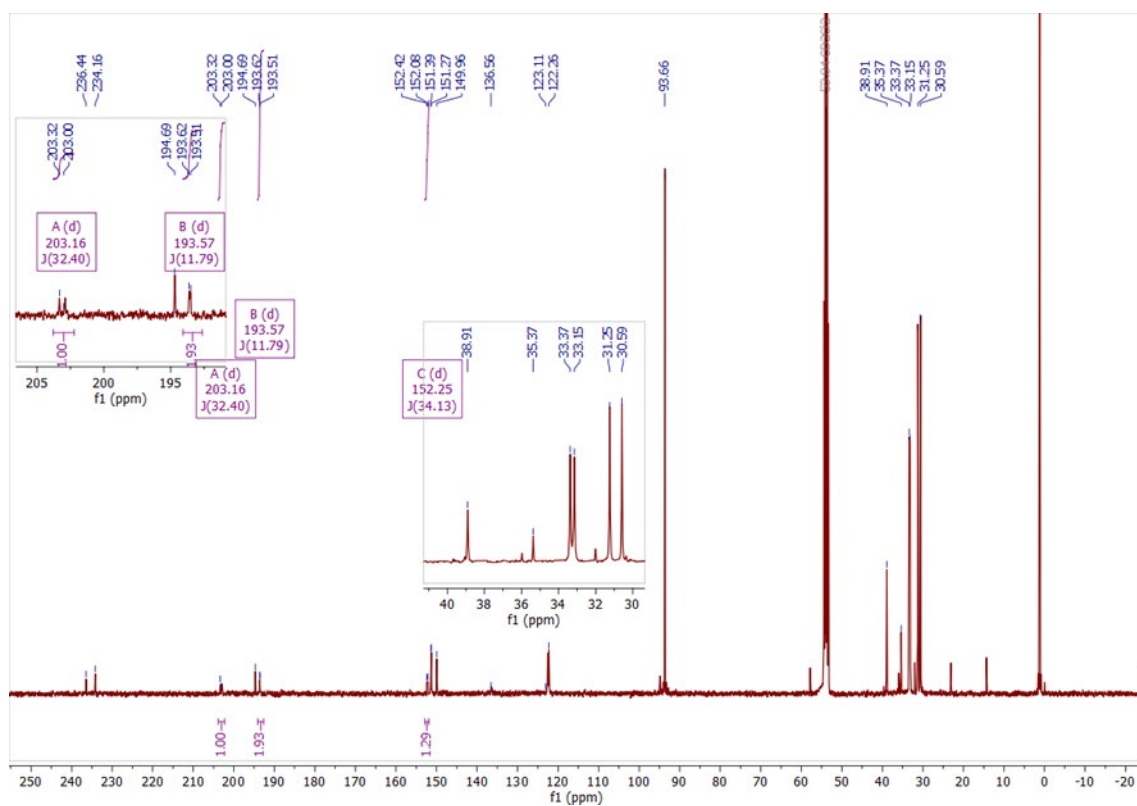


Figure S8. ¹³C{¹H} NMR spectrum of compound **2a.2** (CD₂Cl₂).

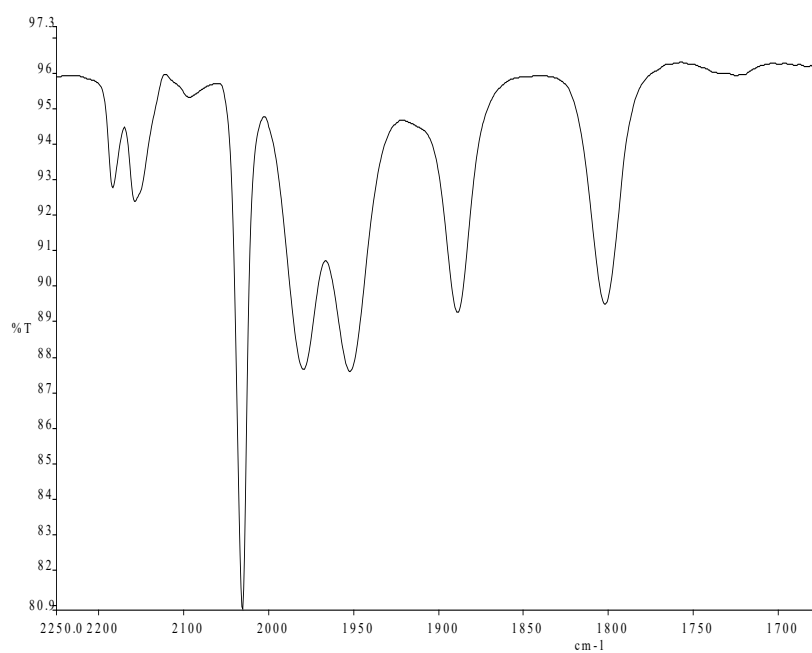


Figure S9. IR spectrum of compound **3a.1** in dichloromethane solution.

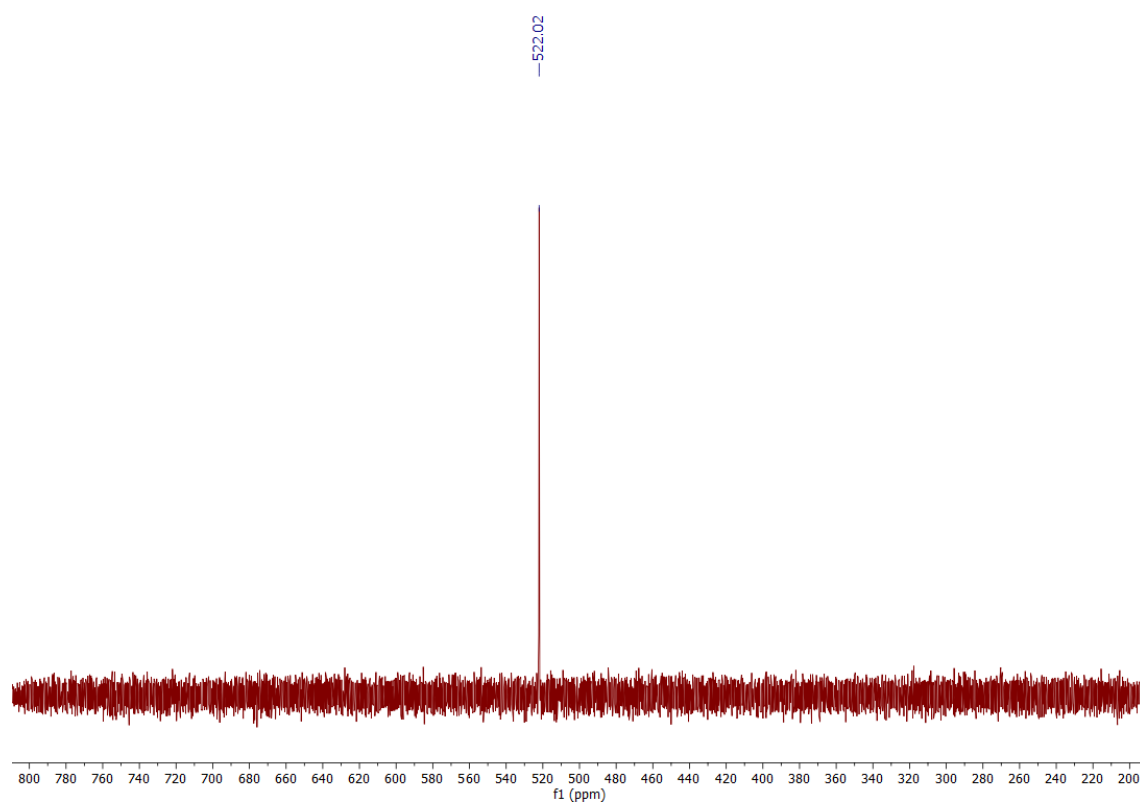


Figure S10. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **3a.1** (CD_2Cl_2).

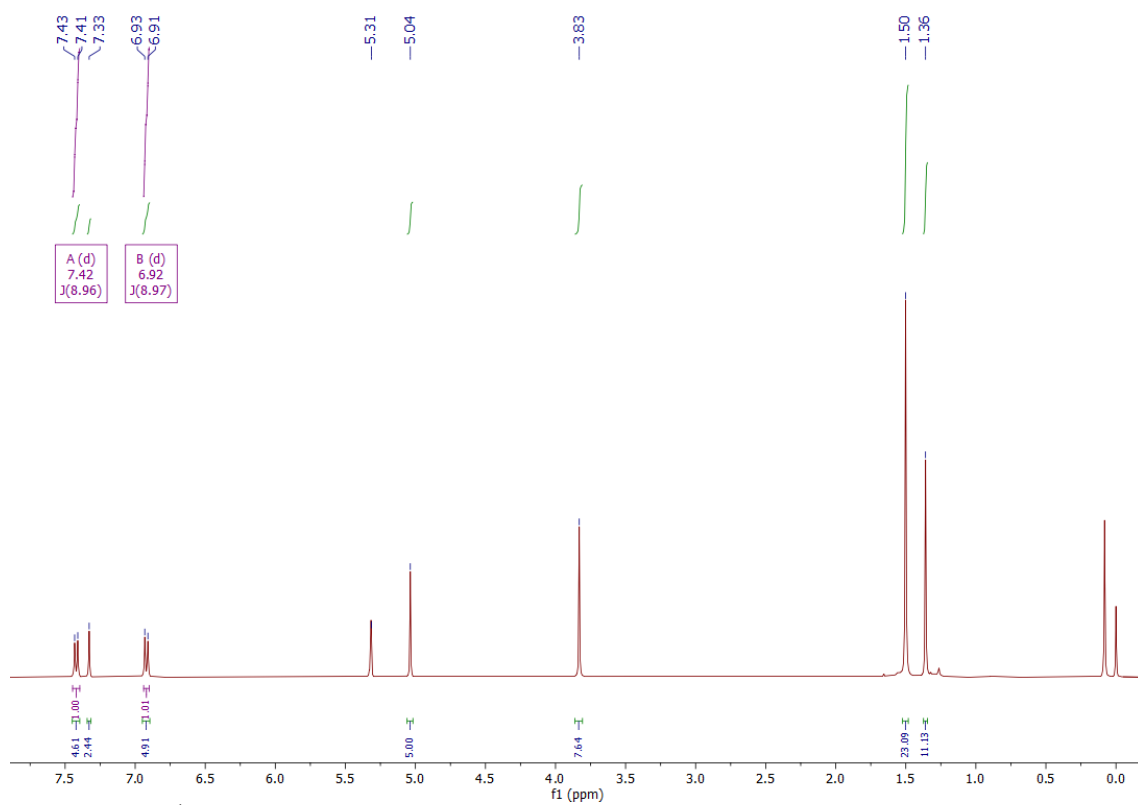


Figure S11. ¹H NMR spectrum of compound **3a.1** (CD₂Cl₂).

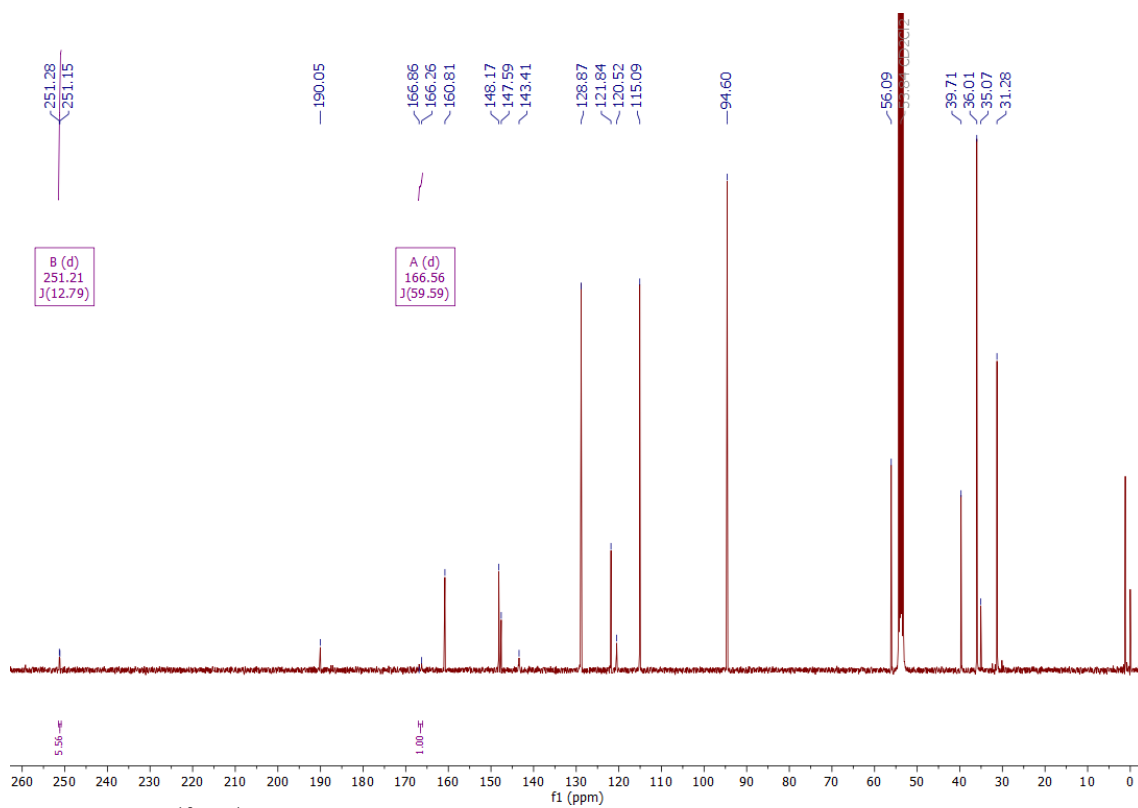


Figure S12. ¹³C{¹H} NMR spectrum of compound **3a.1** (CD₂Cl₂).

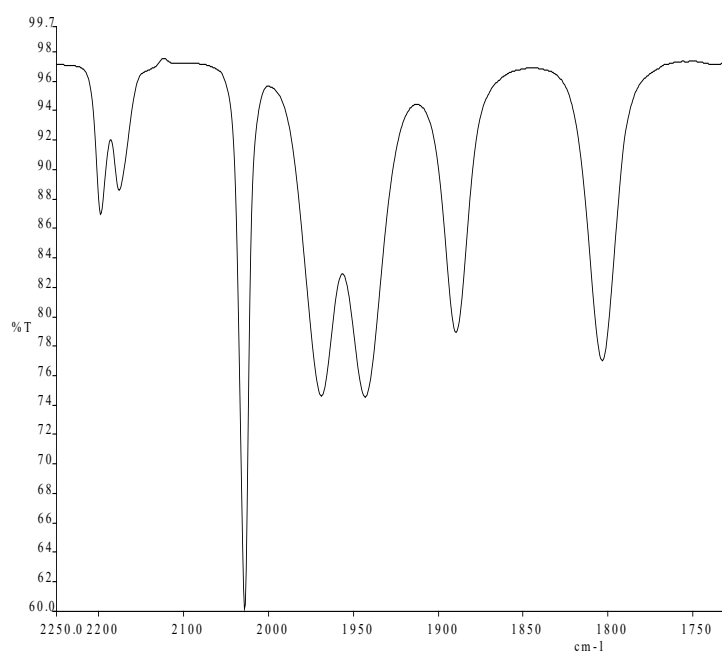


Figure S13. IR spectrum of compound **3a.2** in dichloromethane solution.

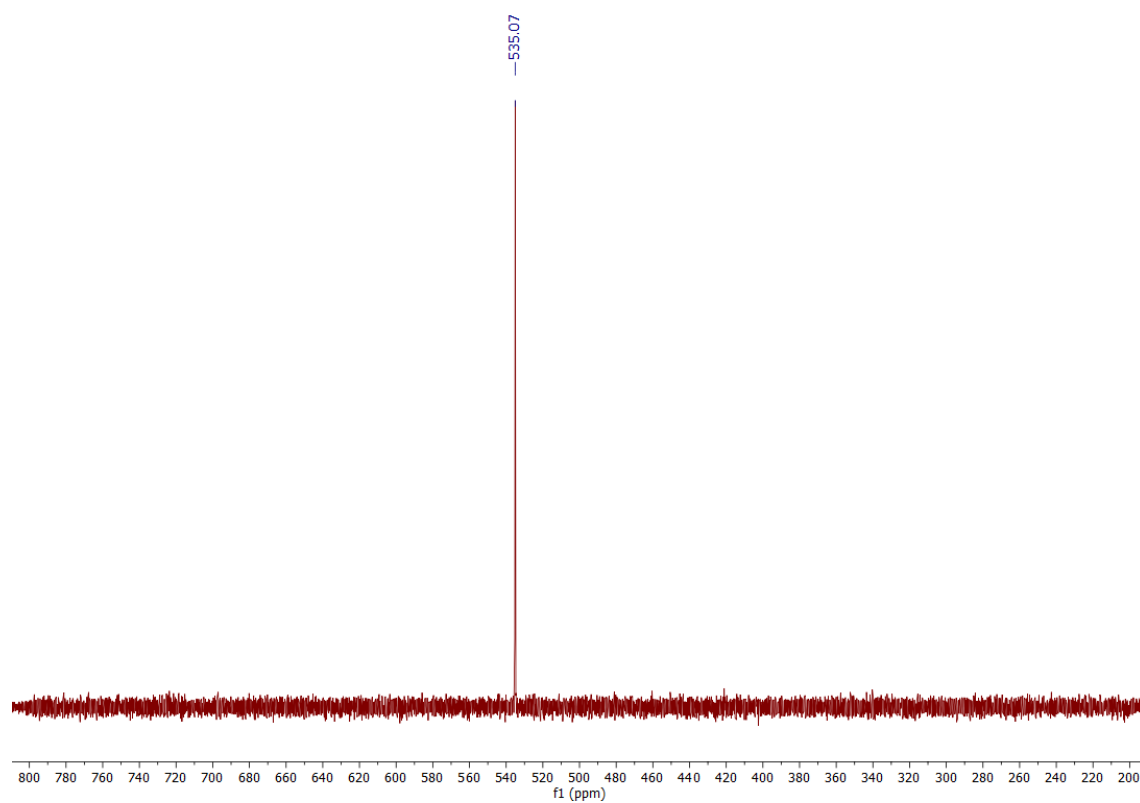


Figure S14. ³¹P{¹H} NMR spectrum of compound **3a.2** (CD₂Cl₂).

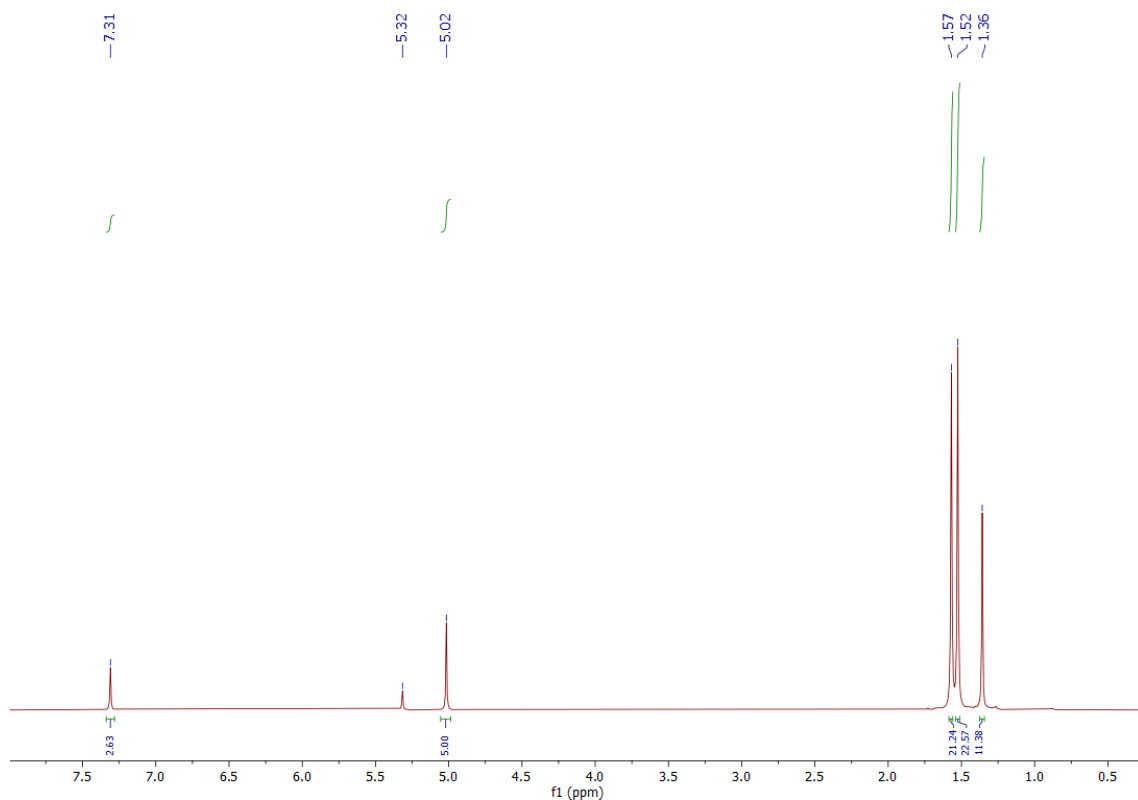


Figure S15. ¹H NMR spectrum of compound **3a.2** (CD₂Cl₂).

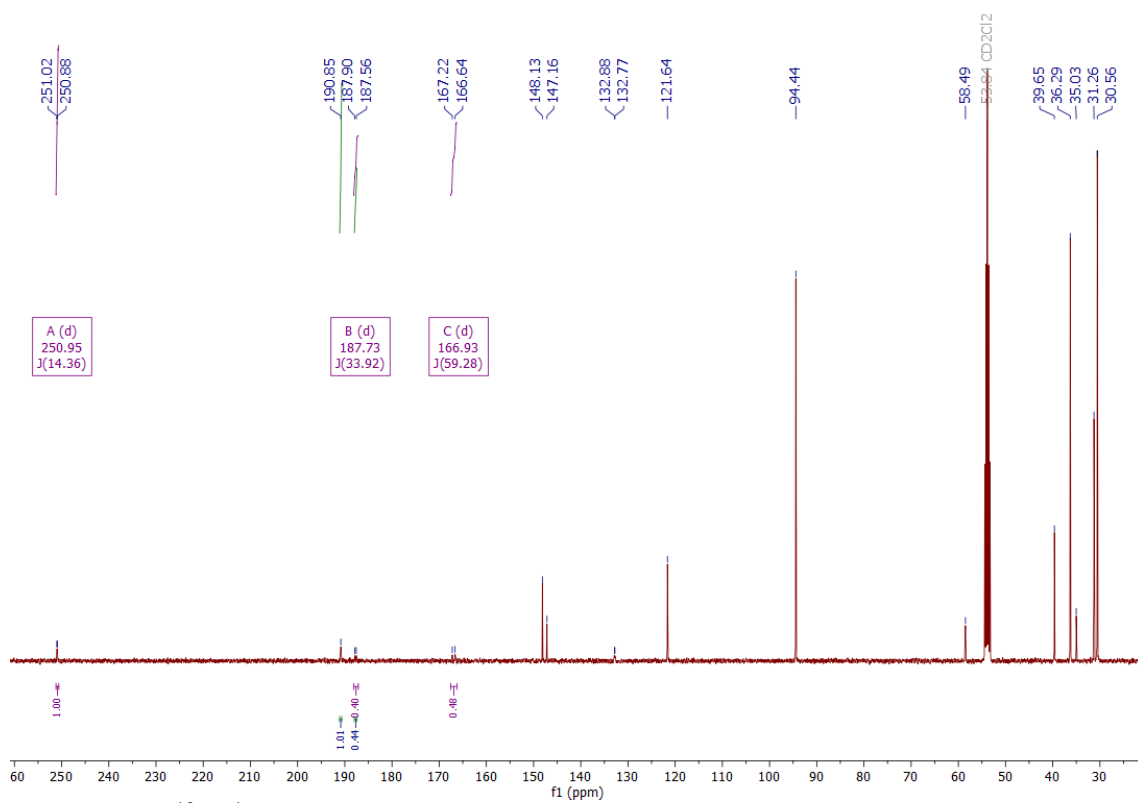


Figure S16. ¹³C{¹H} NMR spectrum of compound **3a.2** (CD₂Cl₂).

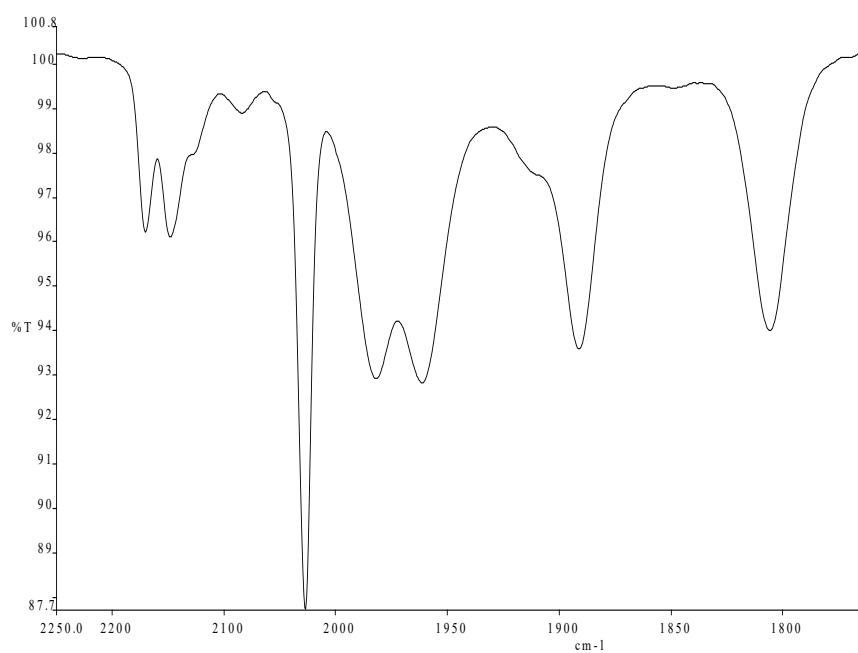


Figure S17. IR spectrum of compound **3b.1** in dichloromethane solution.

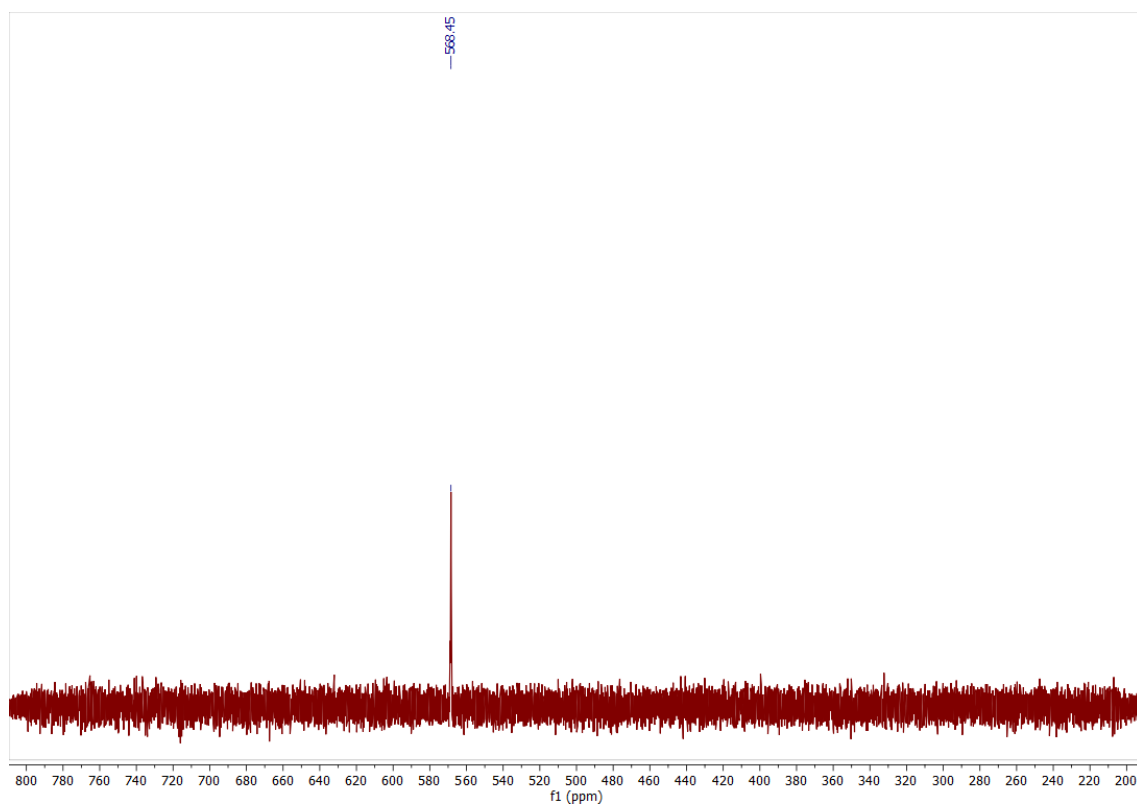


Figure S18. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **3b.1** (CD_2Cl_2).

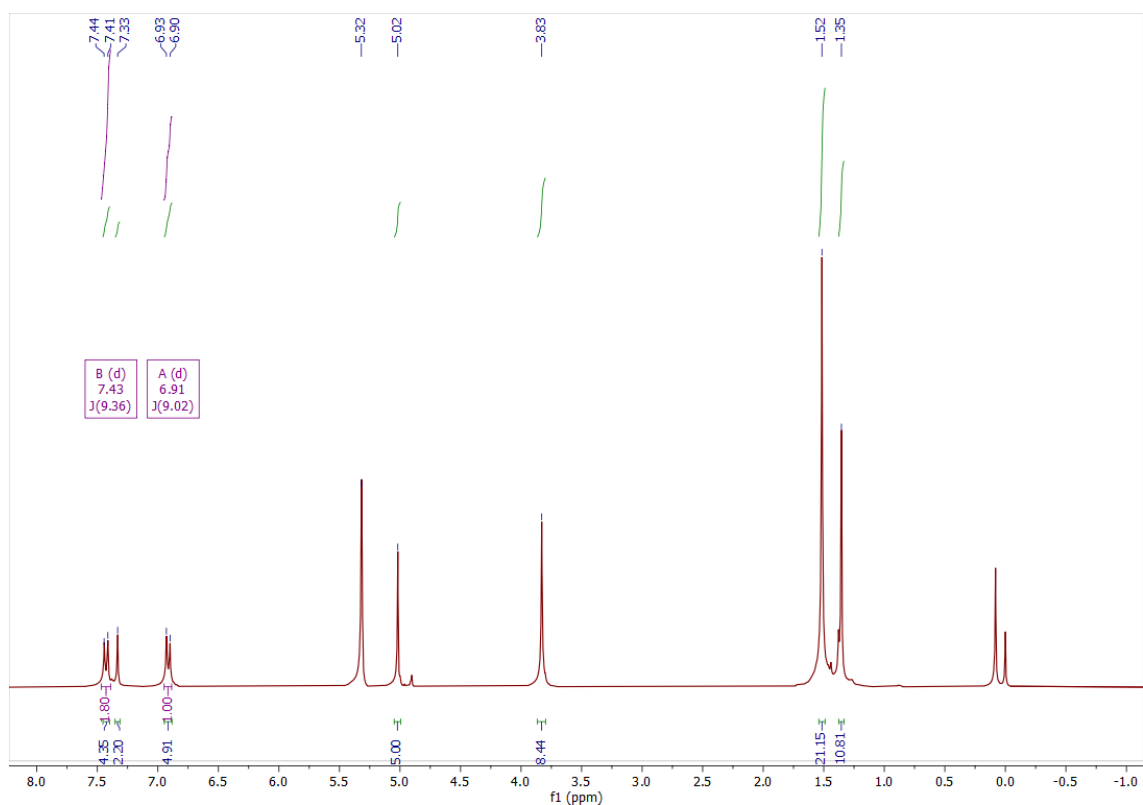


Figure S19. ^1H NMR spectrum of compound **3b.1** (CD_2Cl_2).

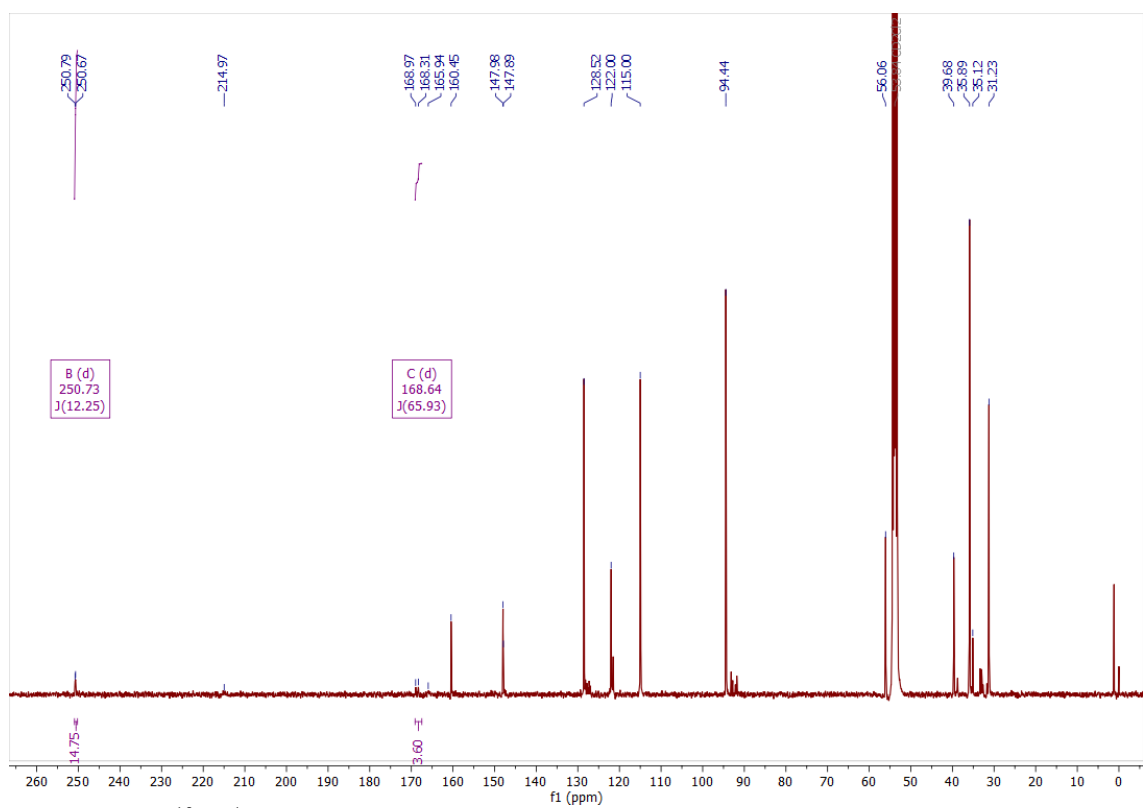


Figure S20. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **3b.1** (CD_2Cl_2).

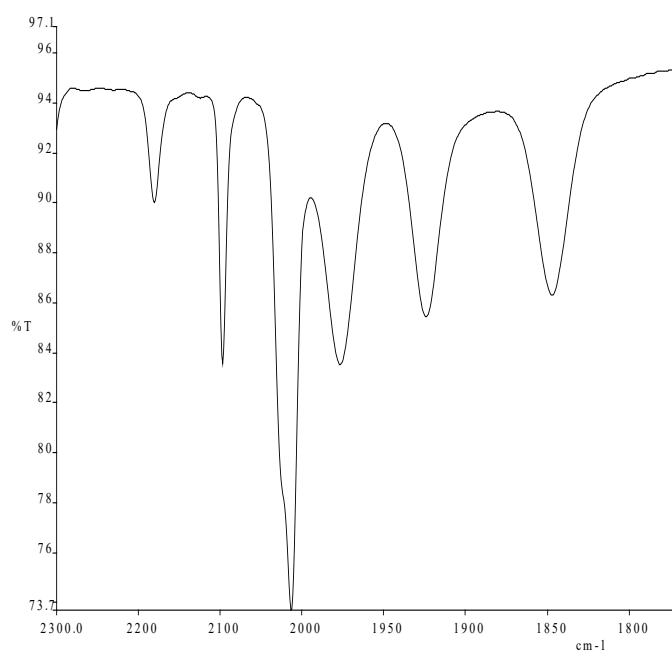


Figure S21. IR spectrum of compound **4a.1** in dichloromethane solution.

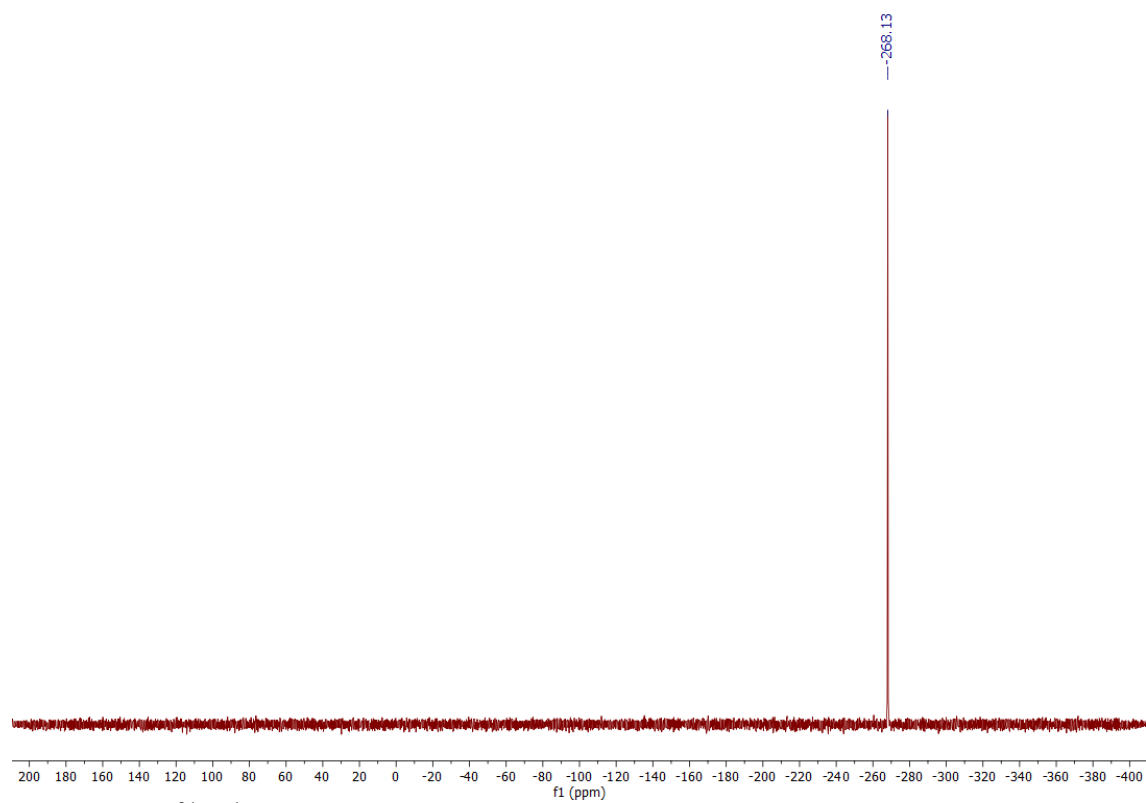


Figure S22. ³¹P{¹H} NMR spectrum of compound **4a.1** (CD₂Cl₂).

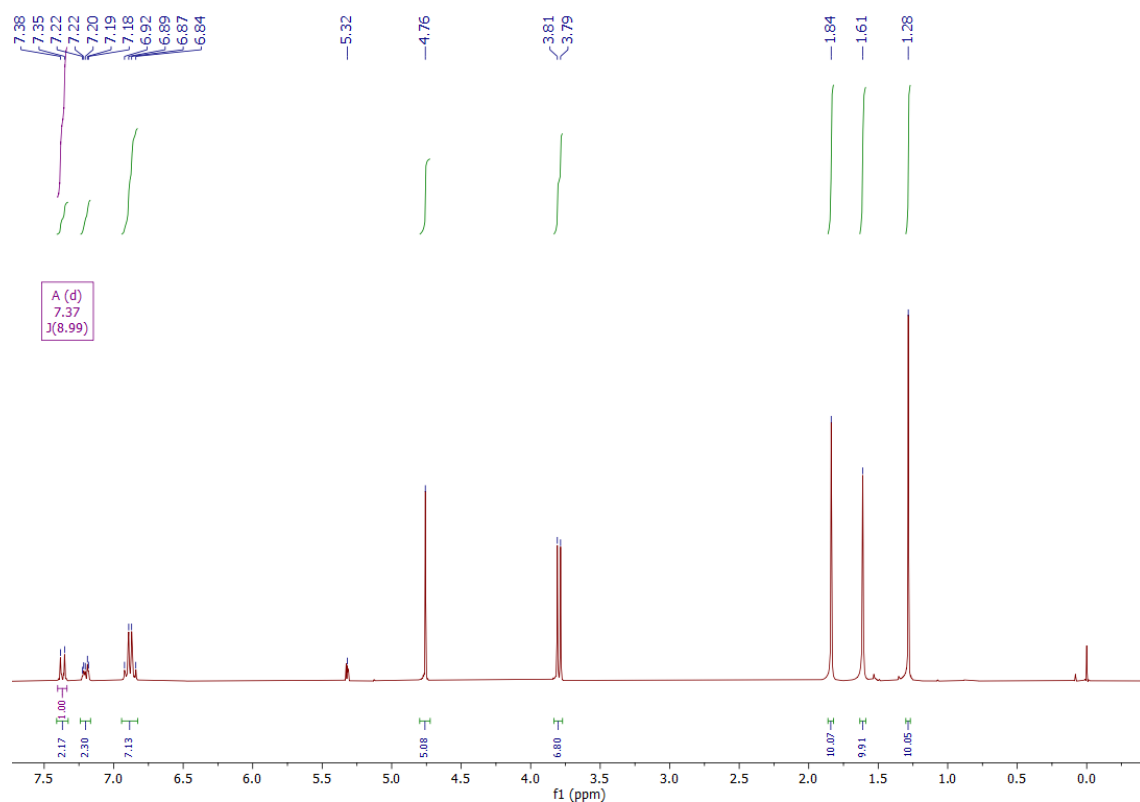


Figure S23. ^1H NMR spectrum of compound **4a.1** (CD_2Cl_2).

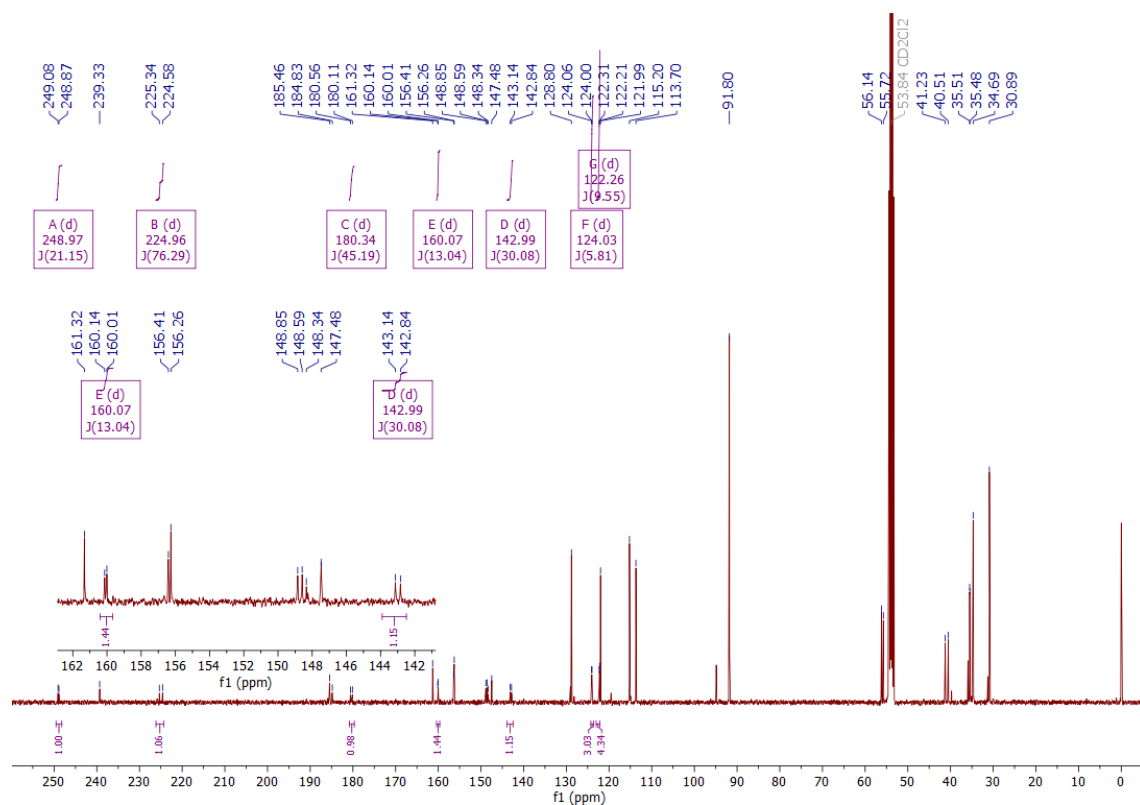


Figure S24. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **4a.1** (CD_2Cl_2).

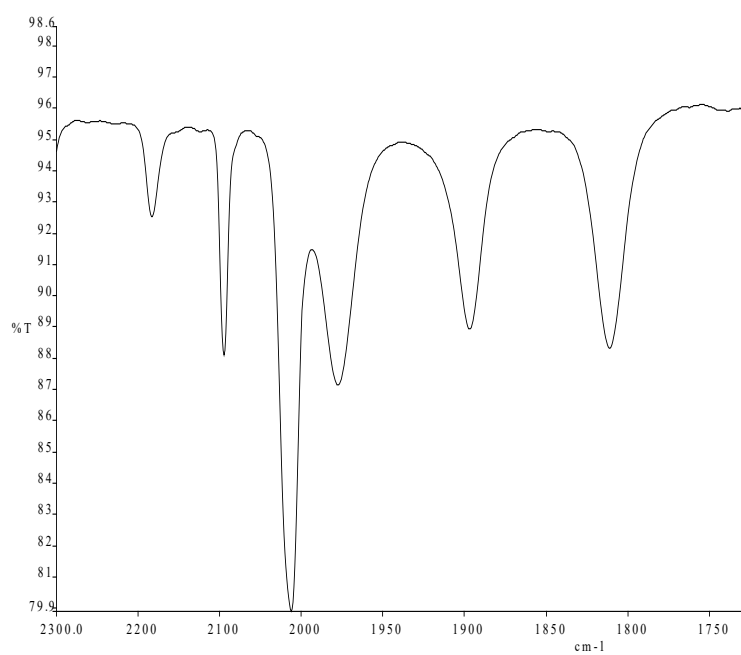


Figure S25. IR spectrum of compound **5a.1** in dichloromethane solution.

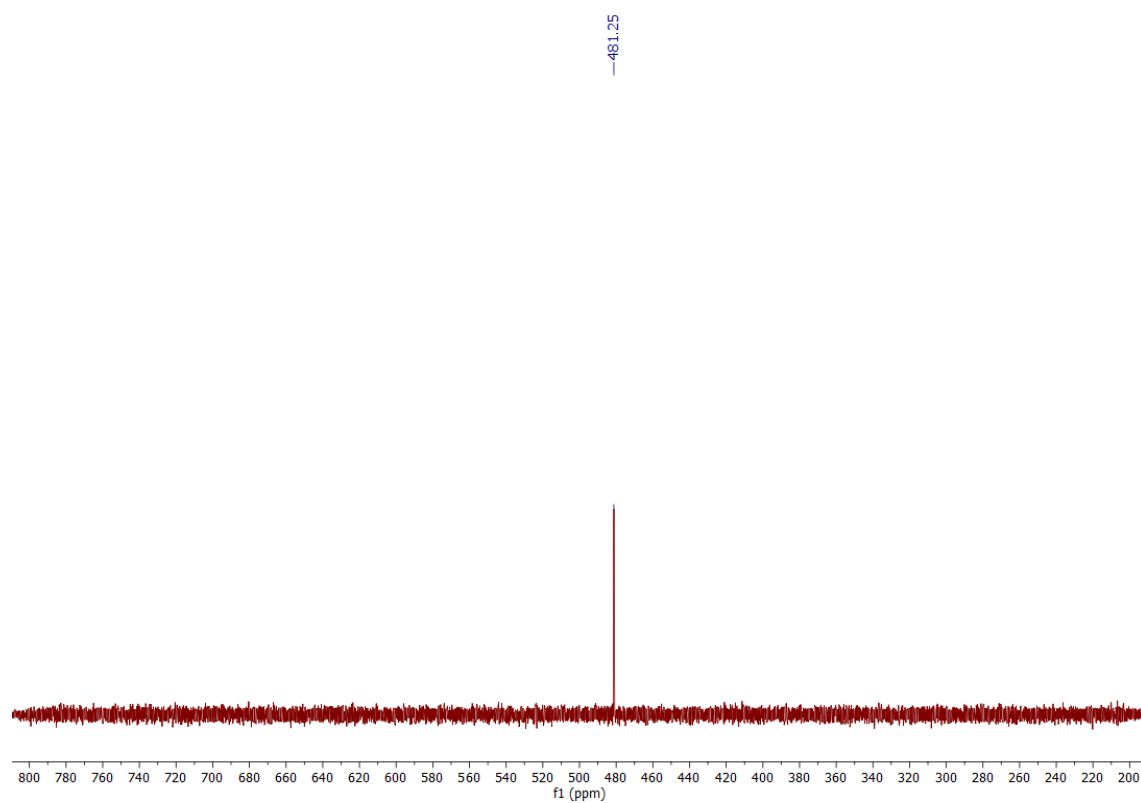


Figure S26. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **5a.1** (CD_2Cl_2).

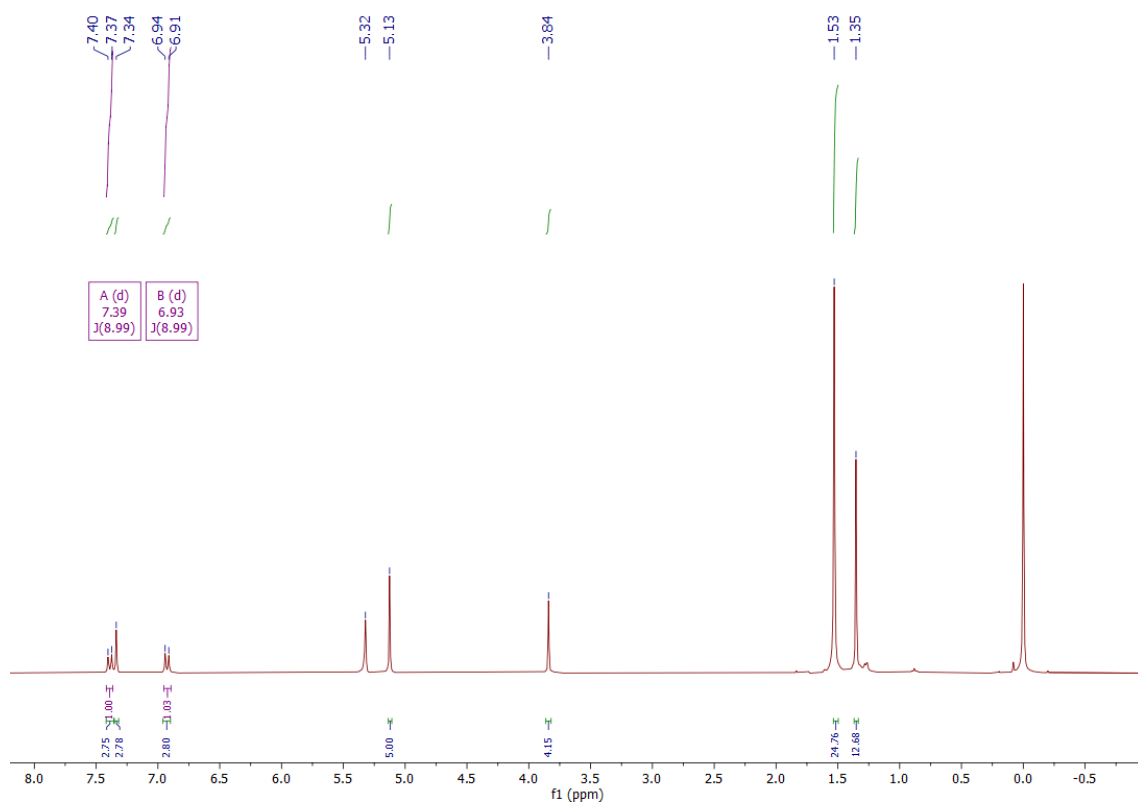


Figure S27. ^1H NMR spectrum of compound **5a.1** (CD_2Cl_2).

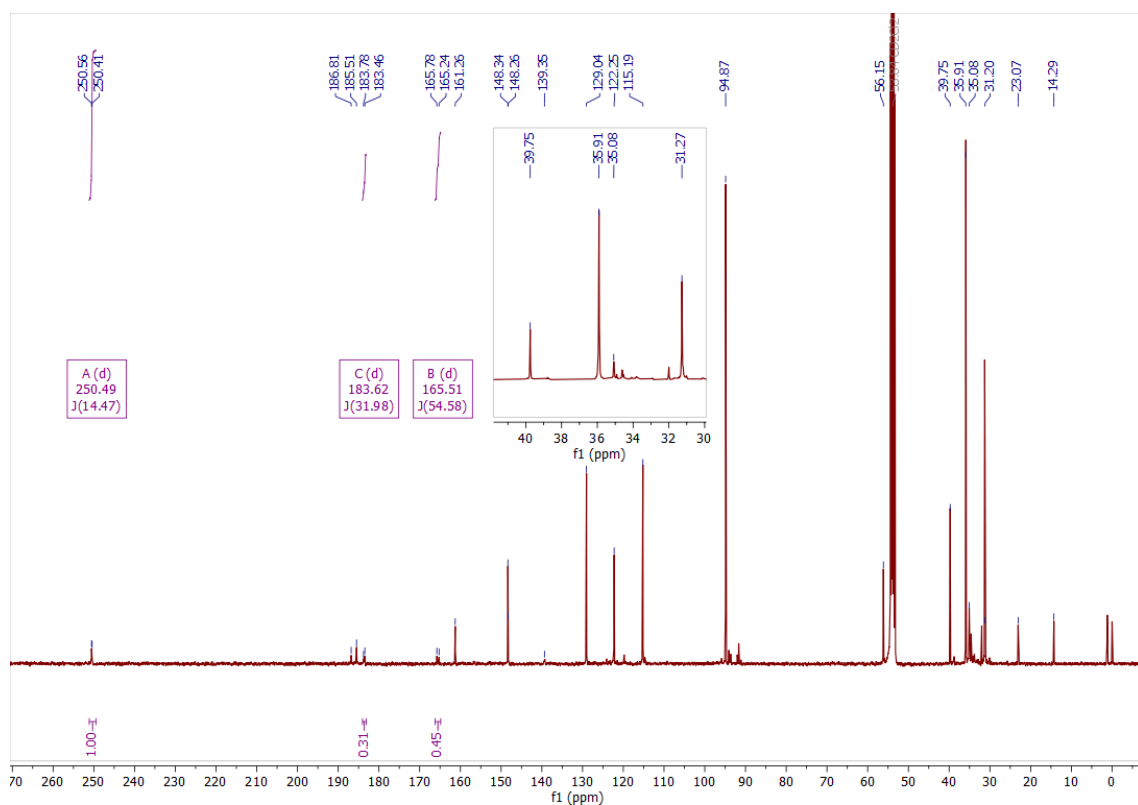


Figure S28. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **5a.1** (CD_2Cl_2).

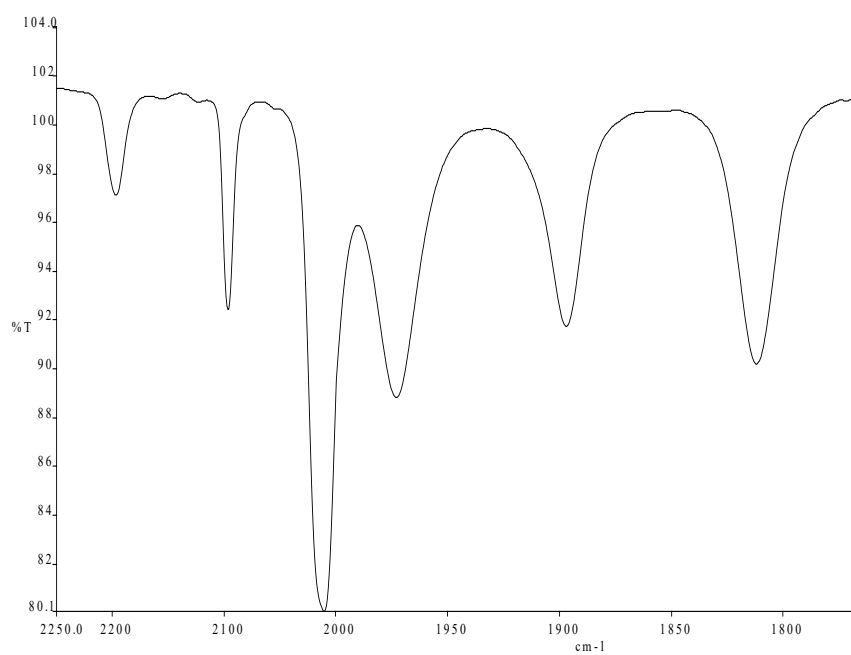


Figure S29. IR spectrum of compound **5a.2** in dichloromethane solution.

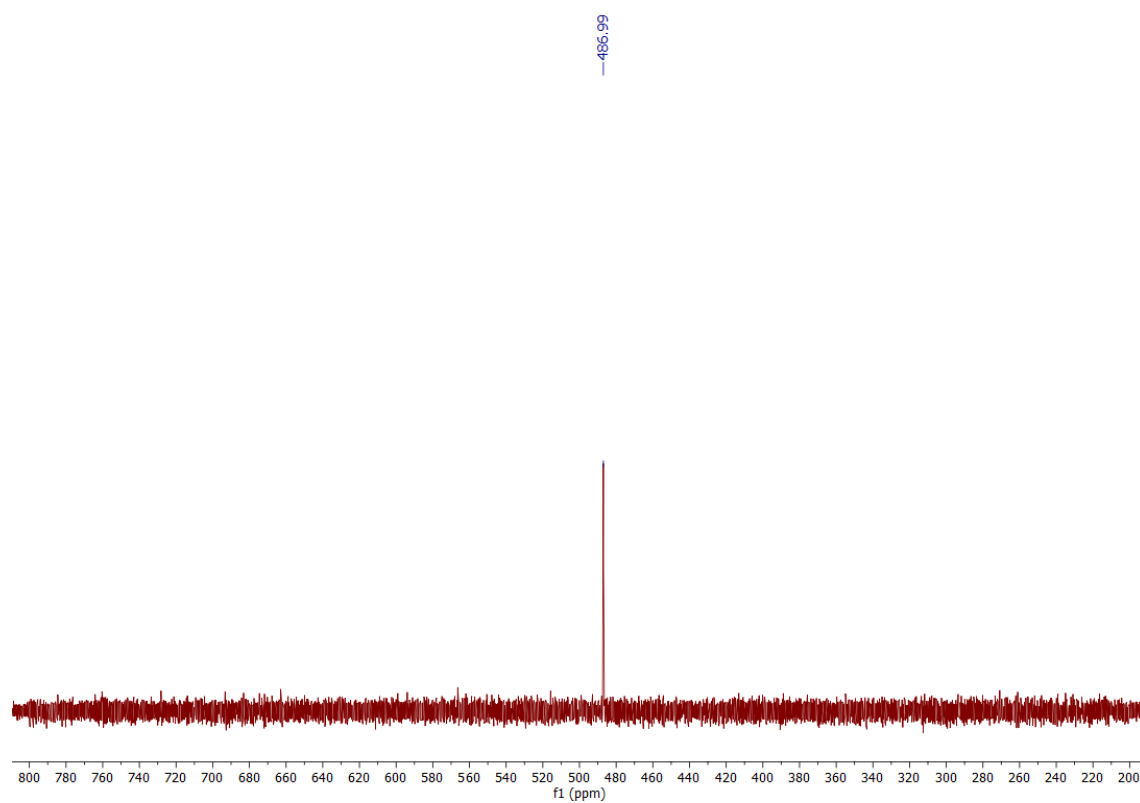


Figure S30. ³¹P{¹H} NMR spectrum of compound **5a.2** (CD₂Cl₂).

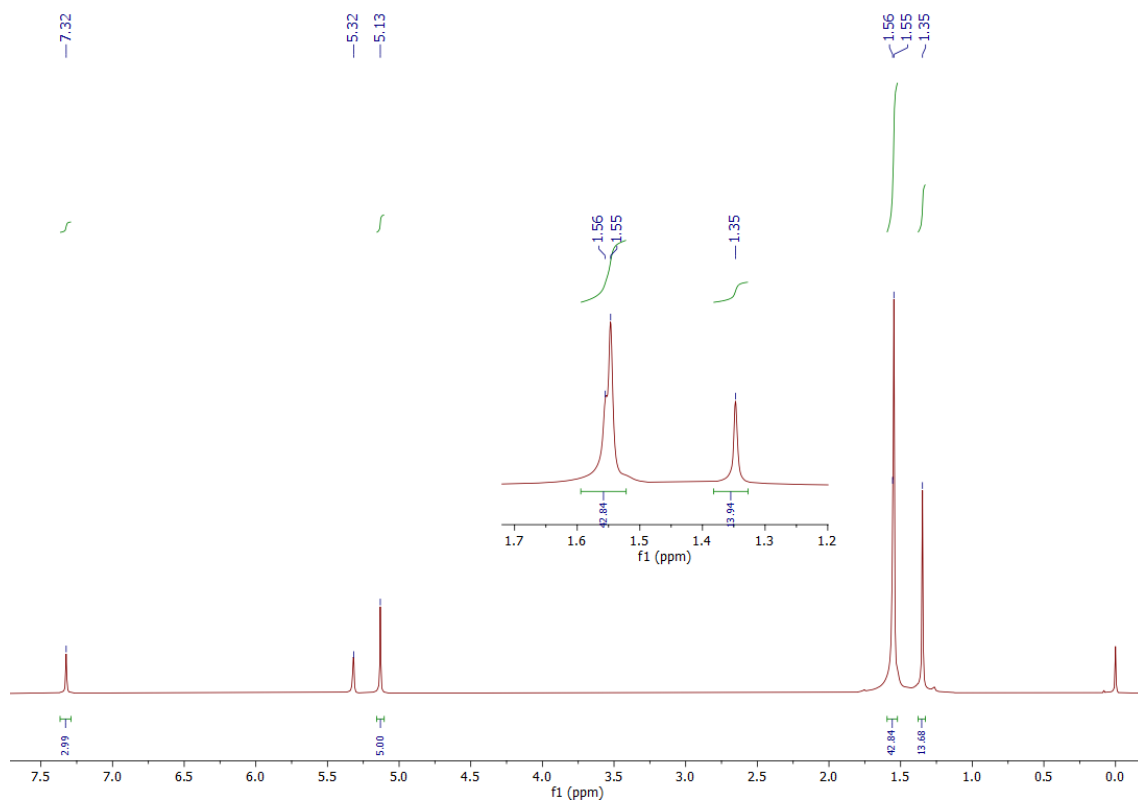


Figure S31. ¹H NMR spectrum of compound **5a.2** (CD₂Cl₂).

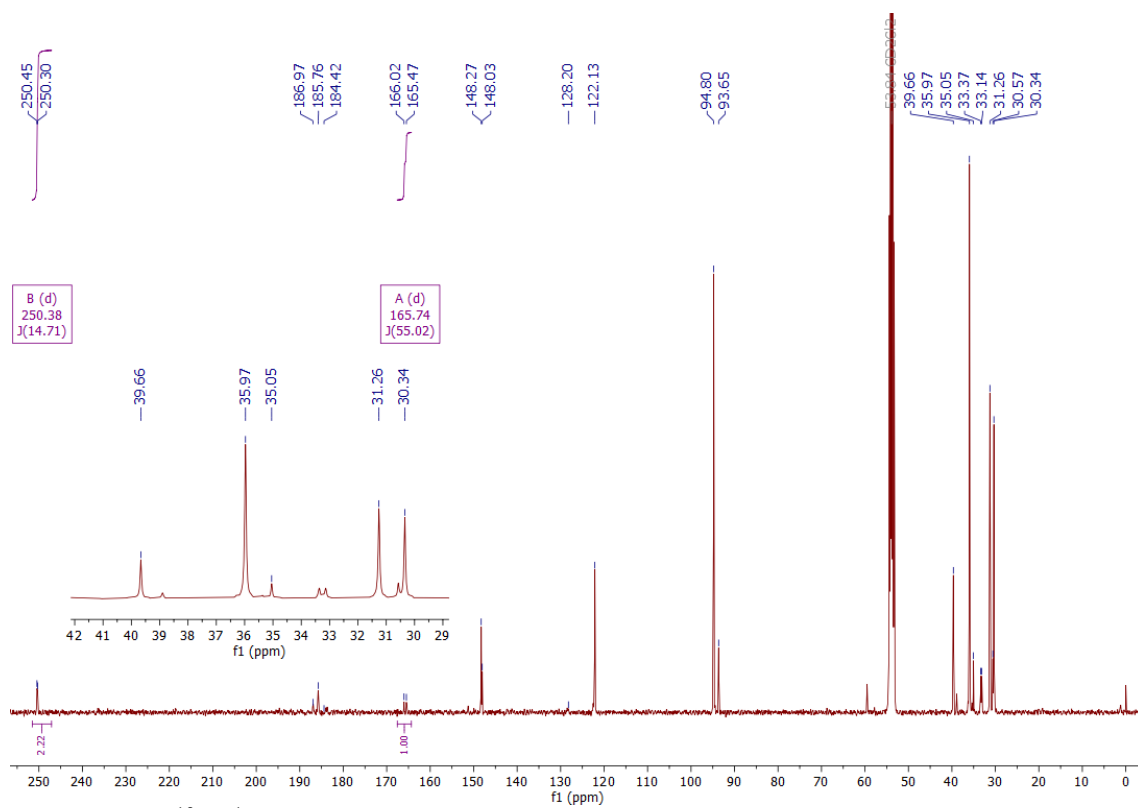


Figure S32. ¹³C{¹H} NMR spectrum of compound **5a.2** (CD₂Cl₂).

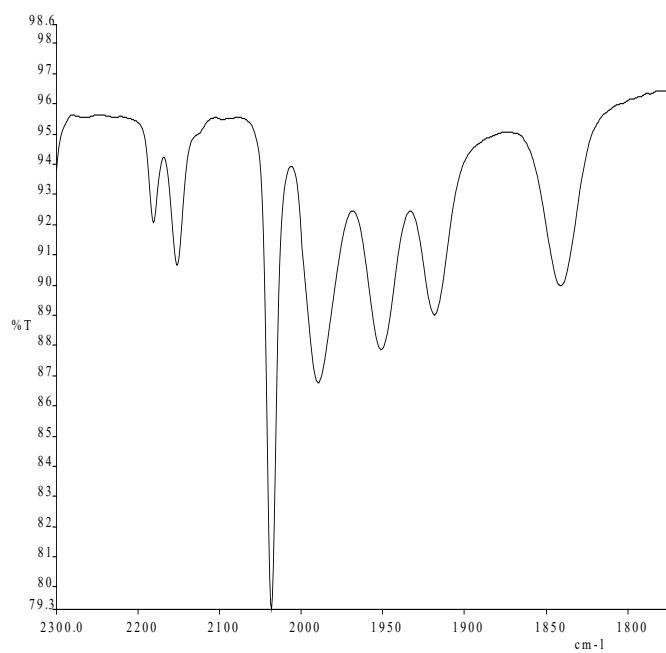


Figure S33. IR spectrum of compound **6a.1** in dichloromethane solution.

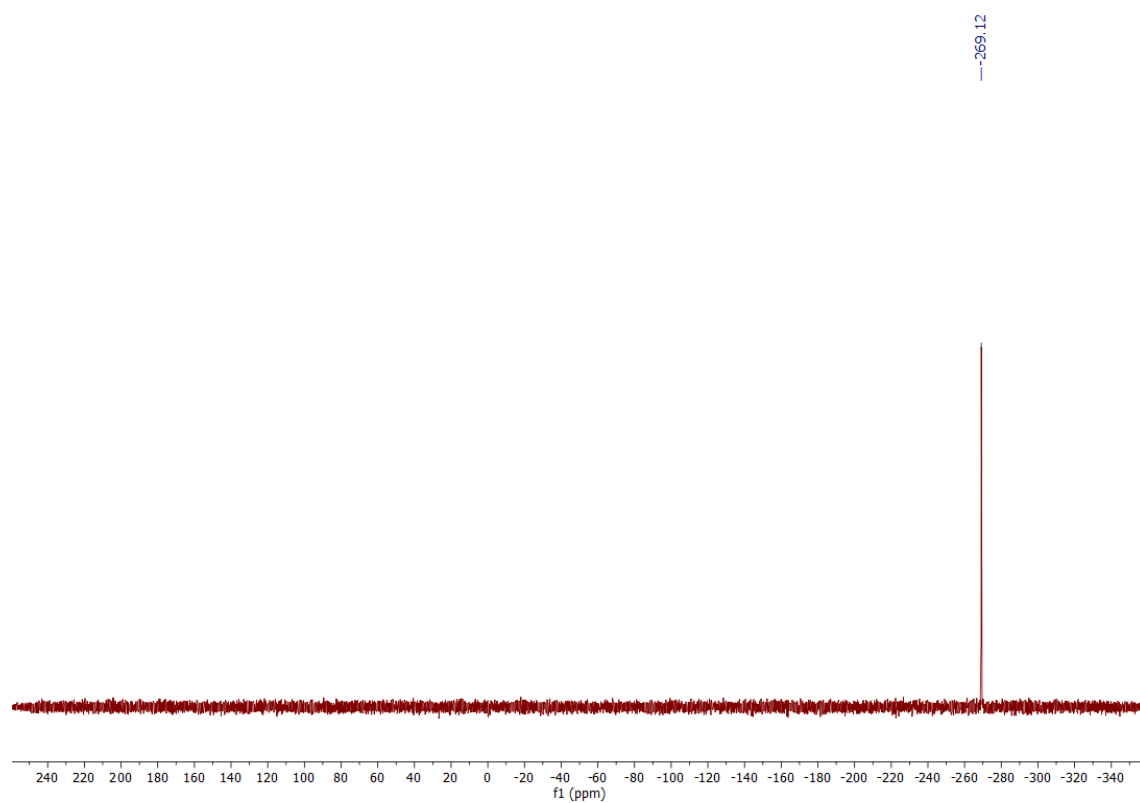


Figure S34. ³¹P{¹H} NMR spectrum of compound **6a.1** (CD₂Cl₂).

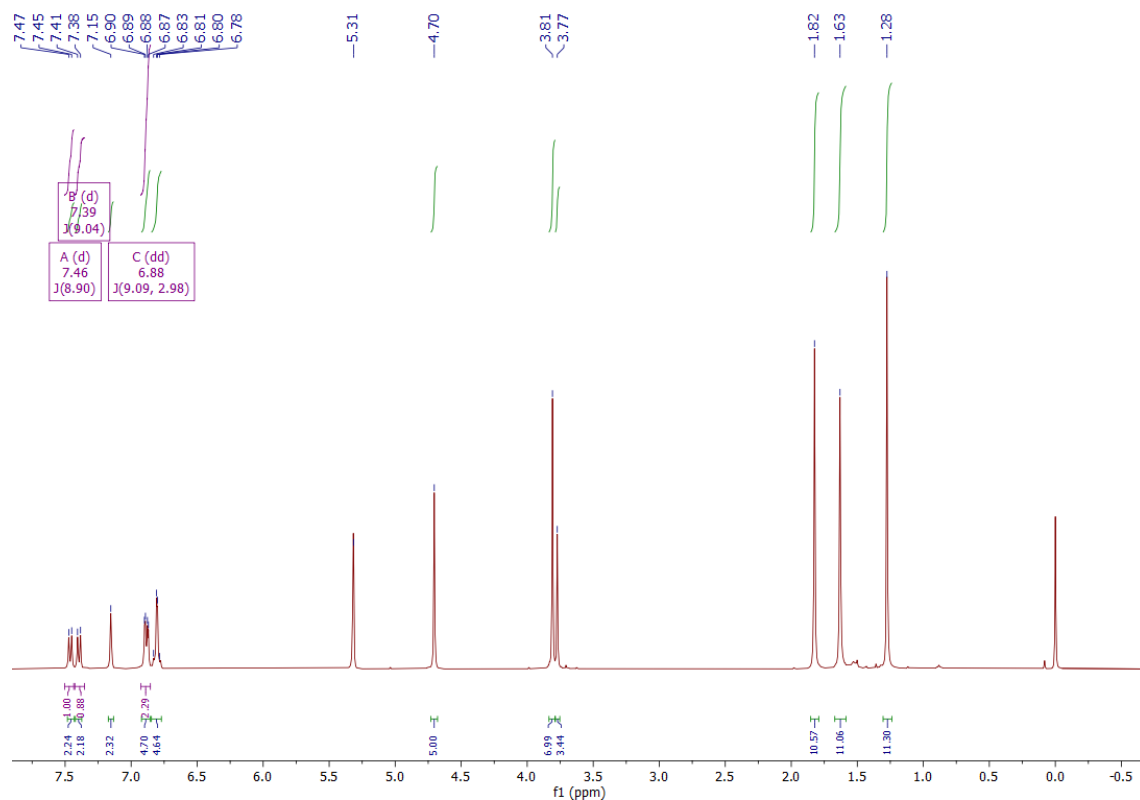


Figure S35. ¹H NMR spectrum of compound **6a.1** (CD₂Cl₂).

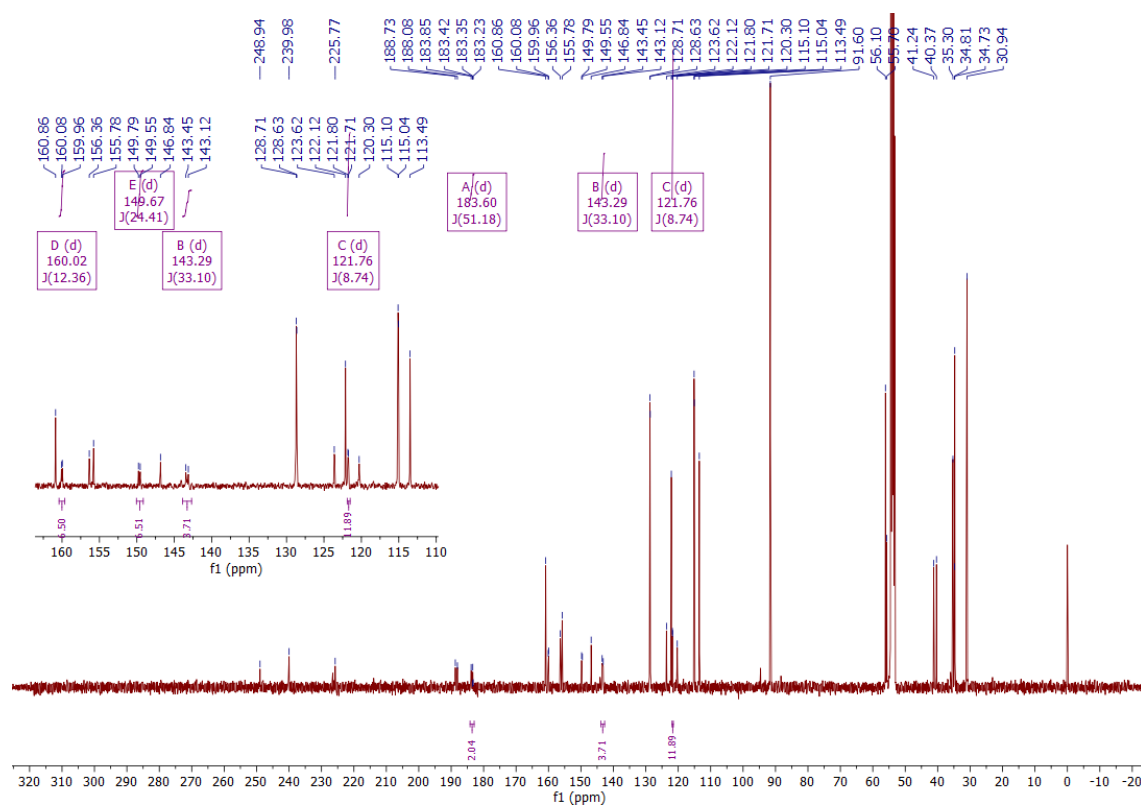


Figure S36. ¹³C{¹H} NMR spectrum of compound **6a.1** (CD₂Cl₂).

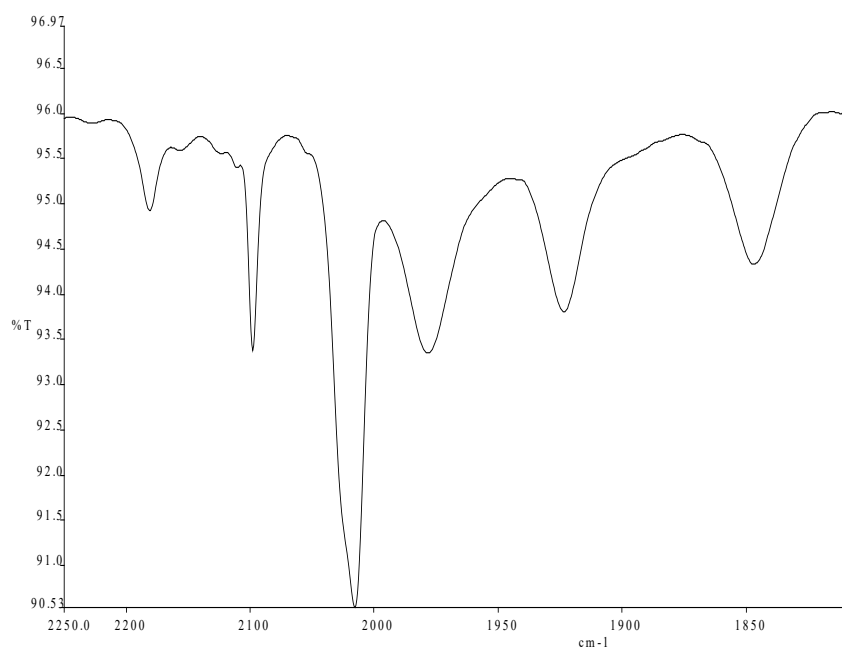


Figure S37. IR spectrum of compound **7a.3** in dichloromethane solution.

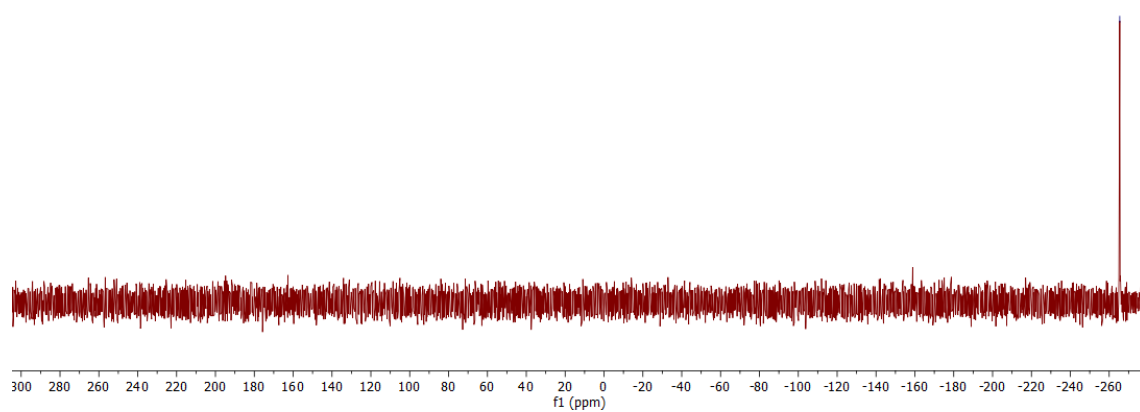


Figure S38. ³¹P{¹H} NMR spectrum of compound **7a.3** (CD₂Cl₂).

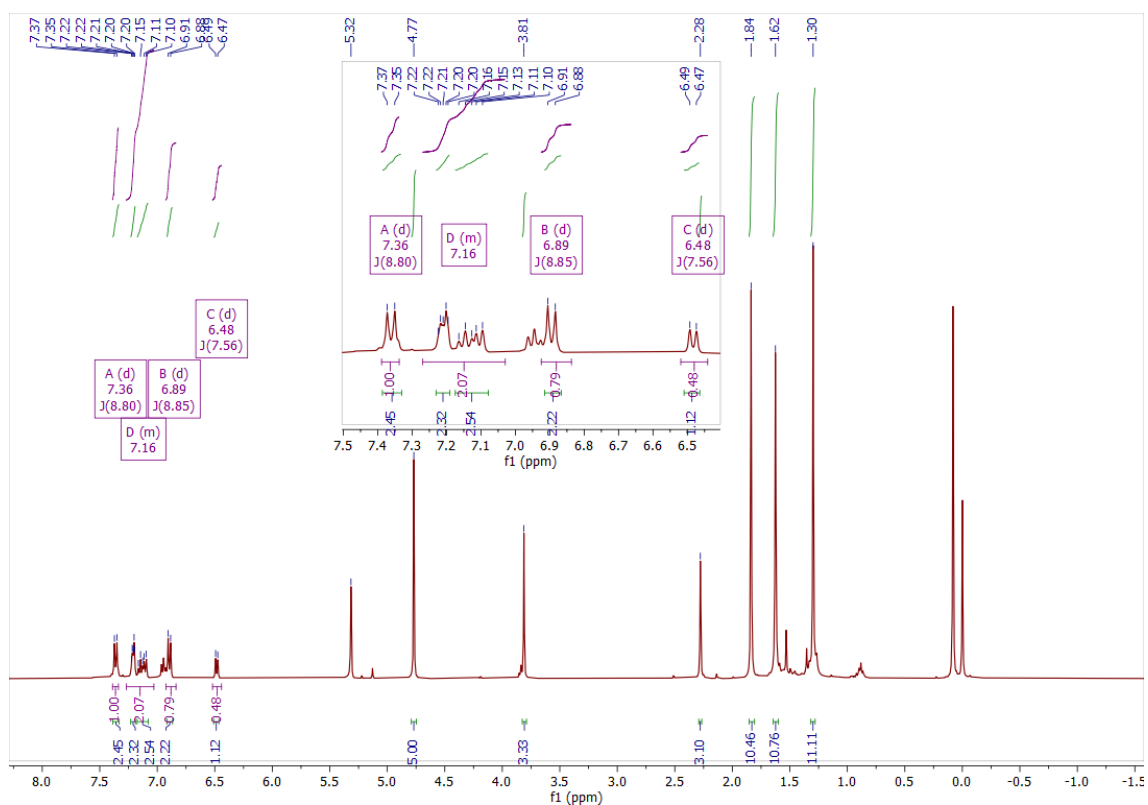


Figure S39. ^1H NMR spectrum of compound **7a.3** (CD_2Cl_2).

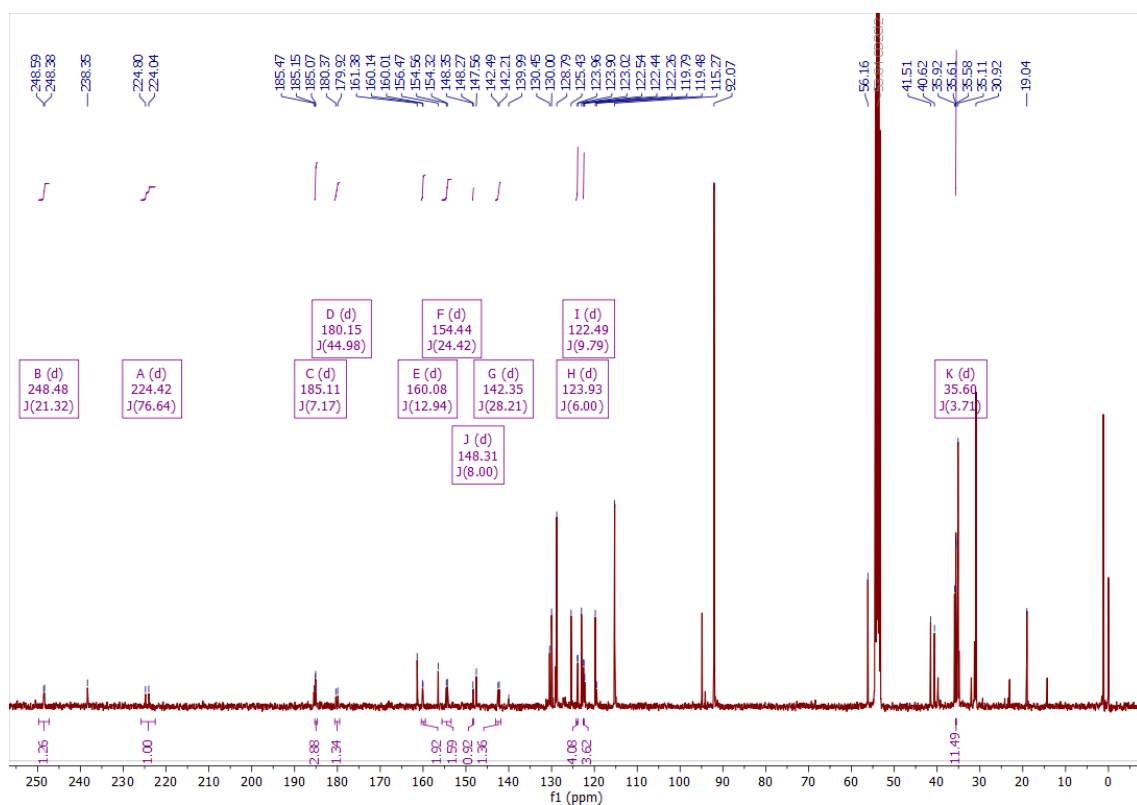


Figure S40. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **7a.3** (CD_2Cl_2).

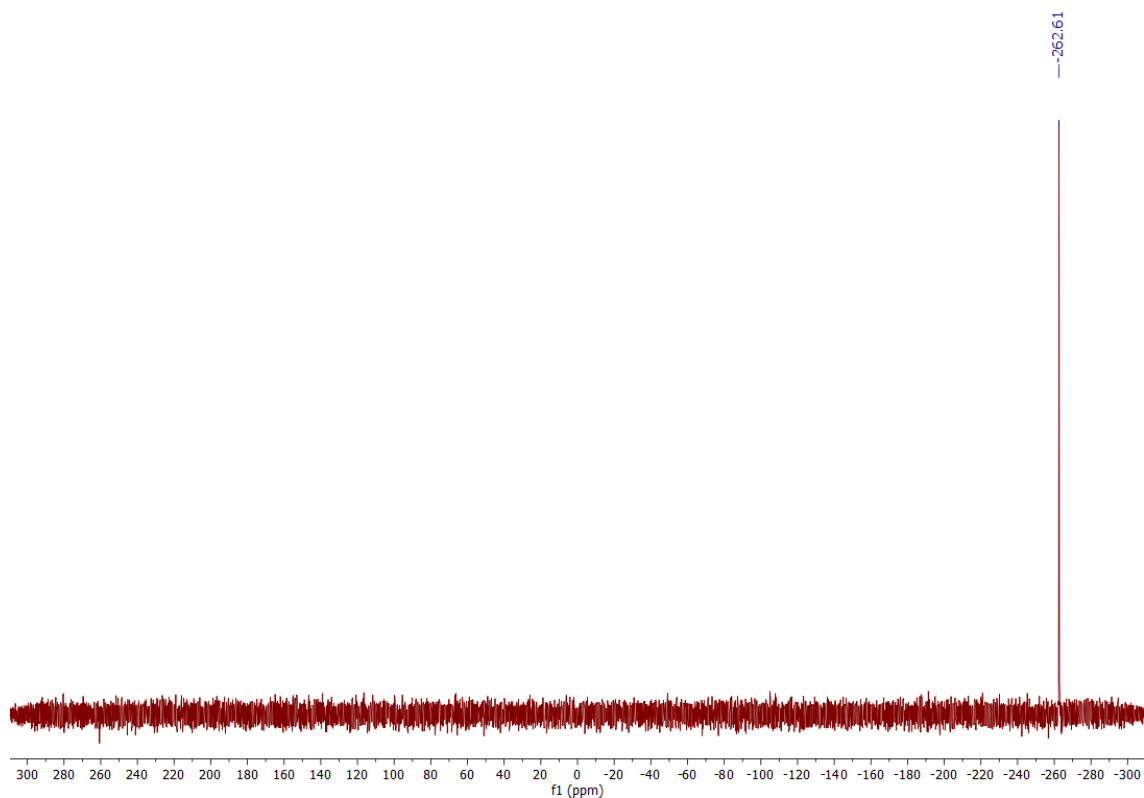


Figure S41. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **7a.4** ($\text{toluene-}d_8$).

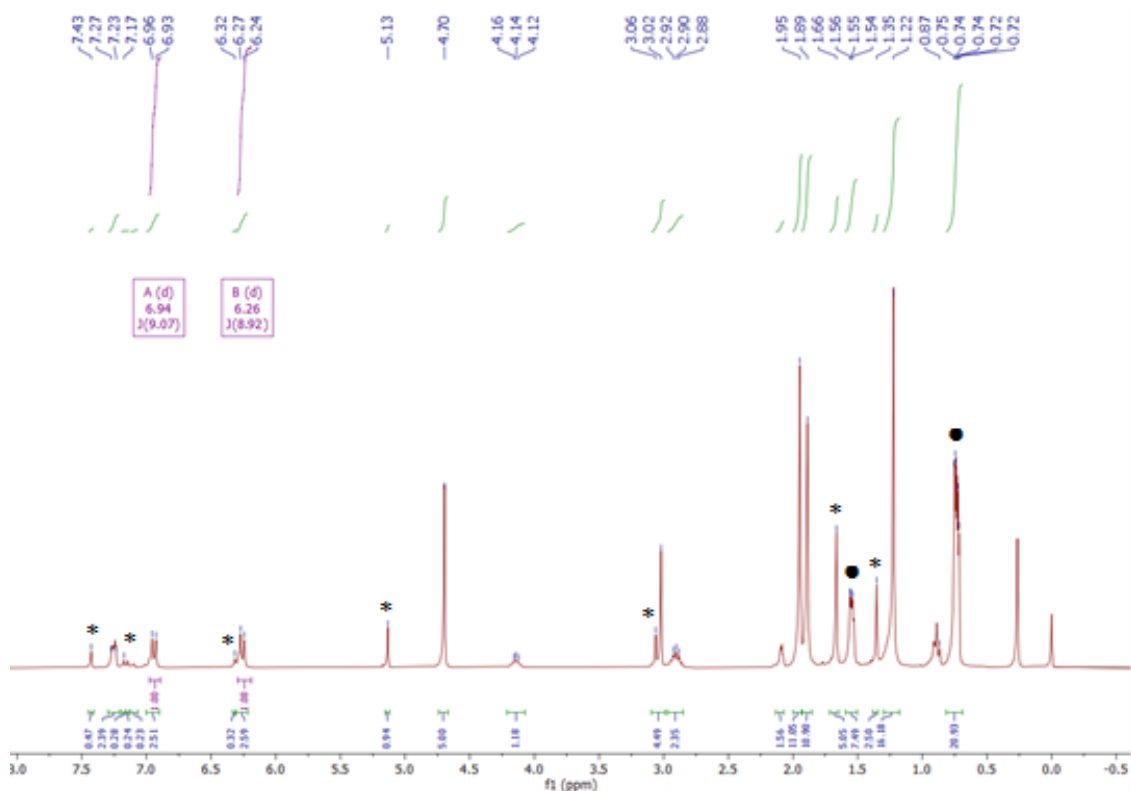


Figure S42. ^1H NMR spectrum of crude compound **7a.4** ($\text{toluene-}d_8$), with resonances of the parent compound **5a.1** (*) and free CN^iPr (●) marked.

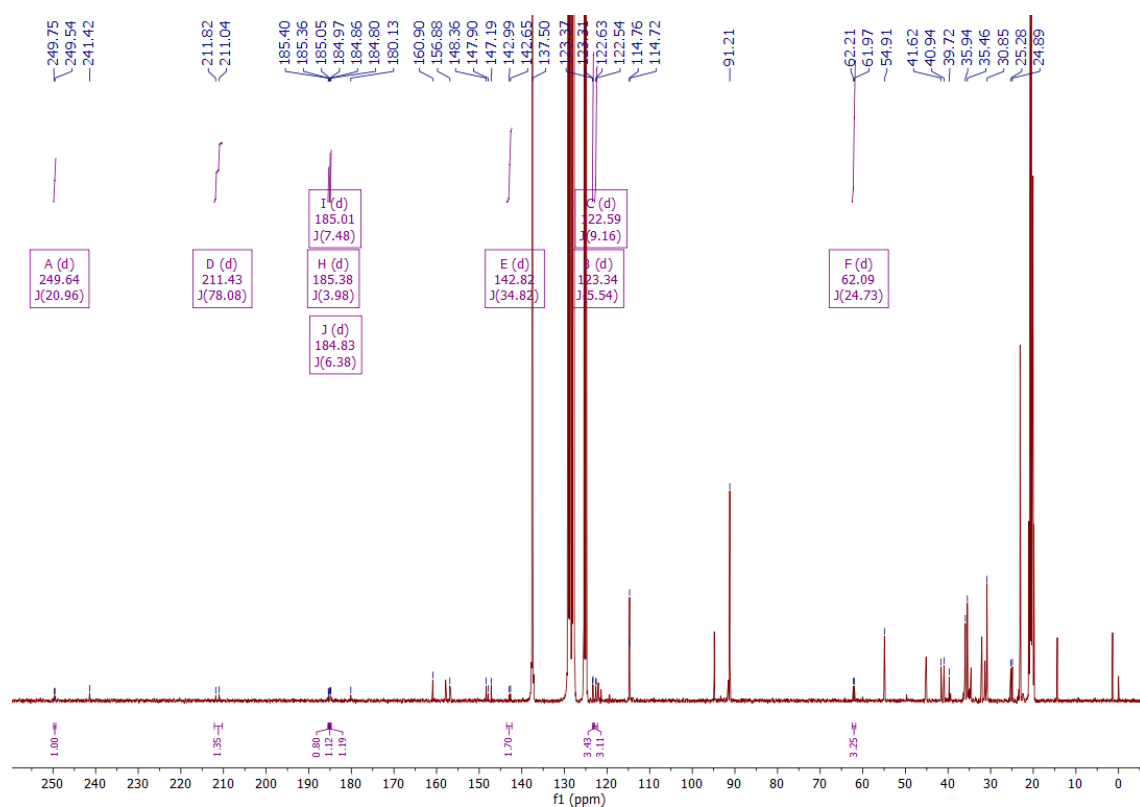


Figure S43. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of crude compound **7a.4** (toluene- d_8).

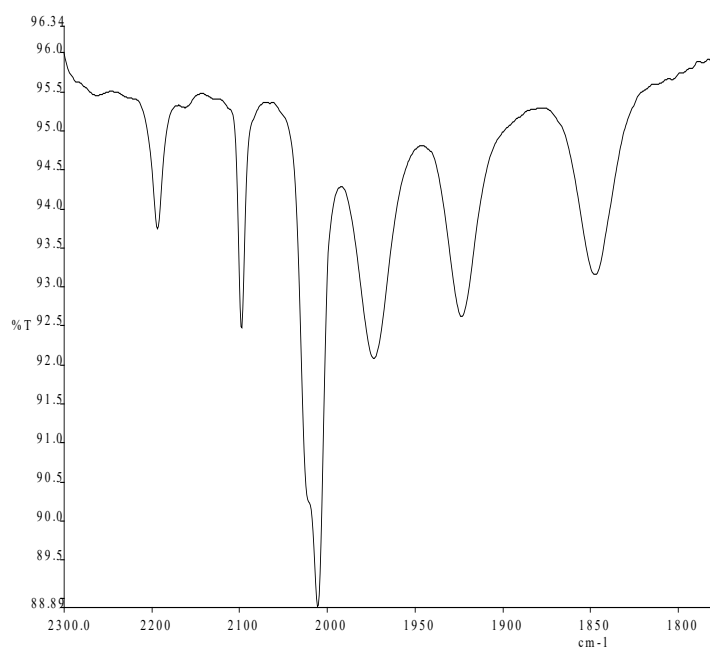


Figure S44. IR spectrum of compound **7a.5** in dichloromethane solution.

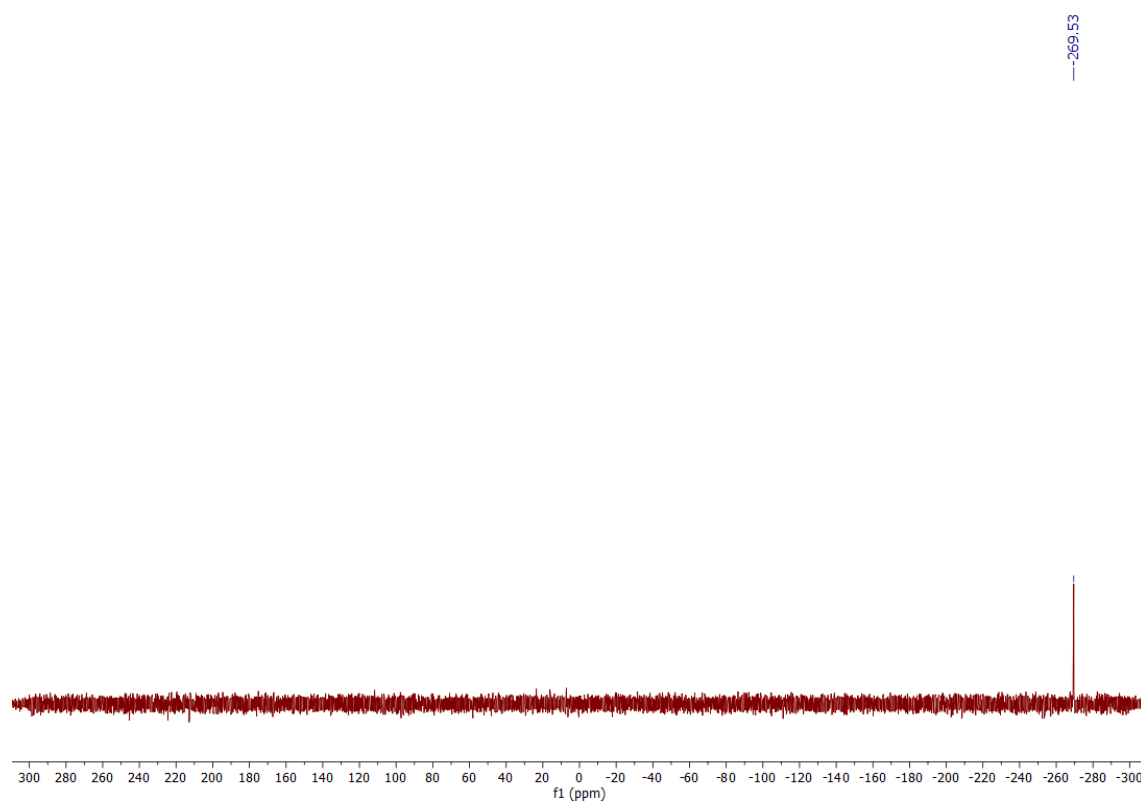


Figure S45. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **7a.5** (CD_2Cl_2).

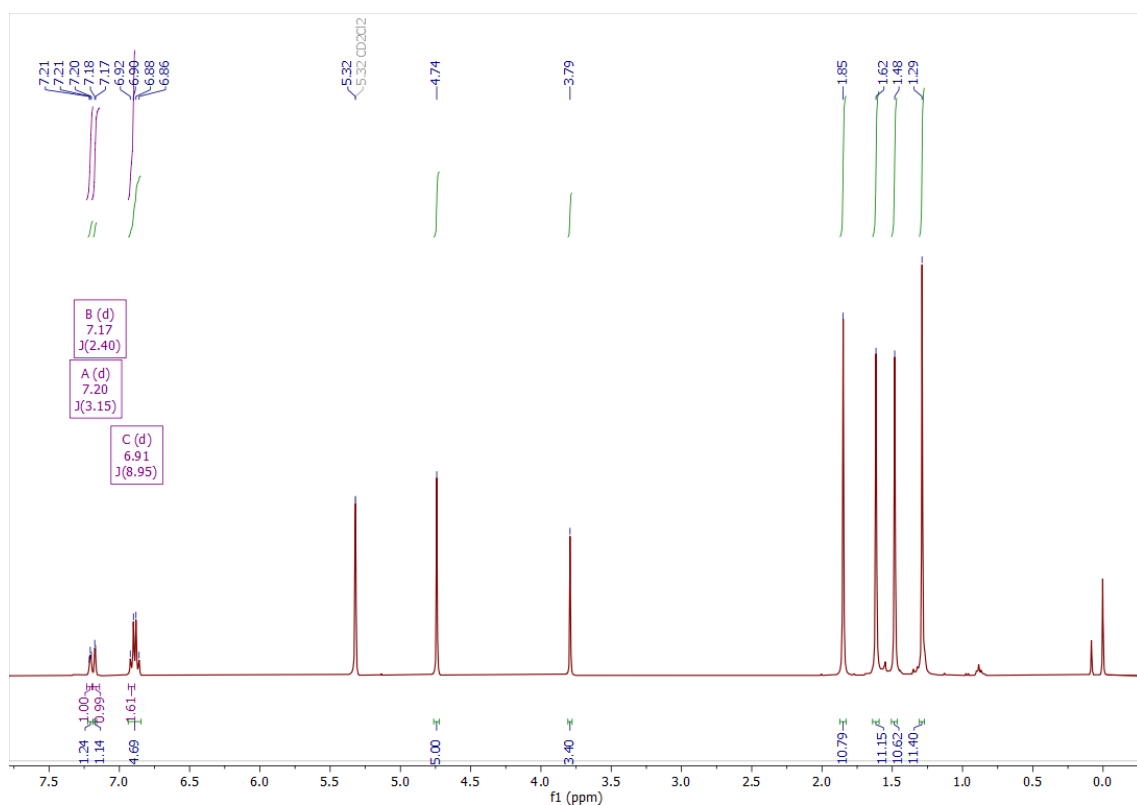


Figure S46. ¹H NMR spectrum of compound **7a.5** (CD₂Cl₂).

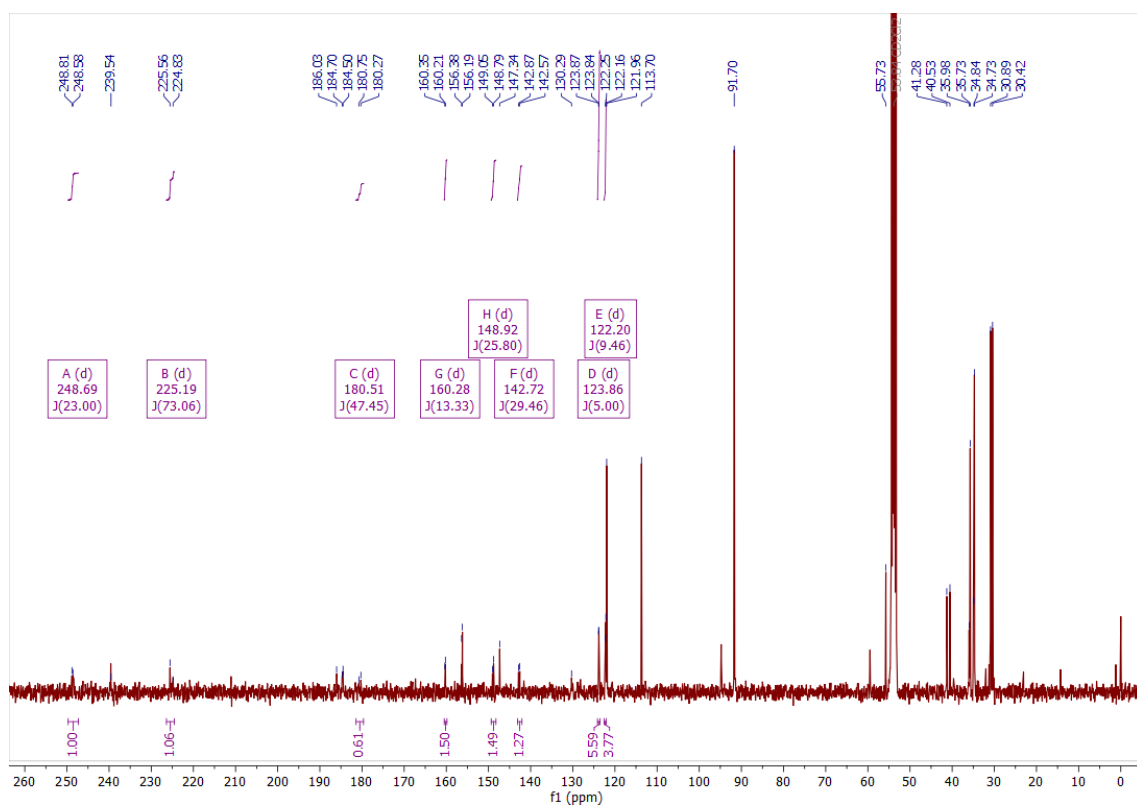


Figure S47. ¹³C{¹H} NMR spectrum of compound **7a.5** (CD₂Cl₂).

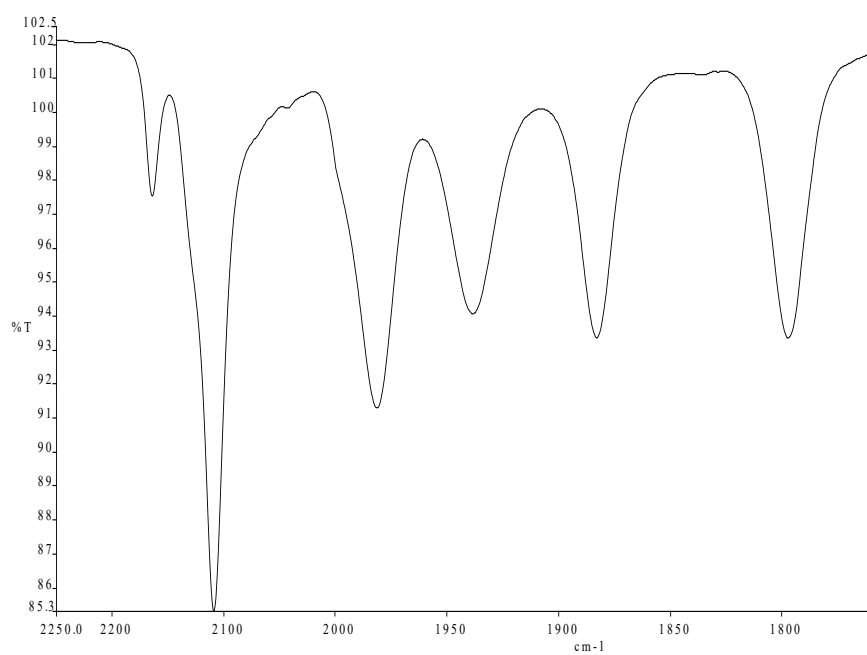


Figure S48. IR spectrum of compound **8b.1** in dichloromethane solution.

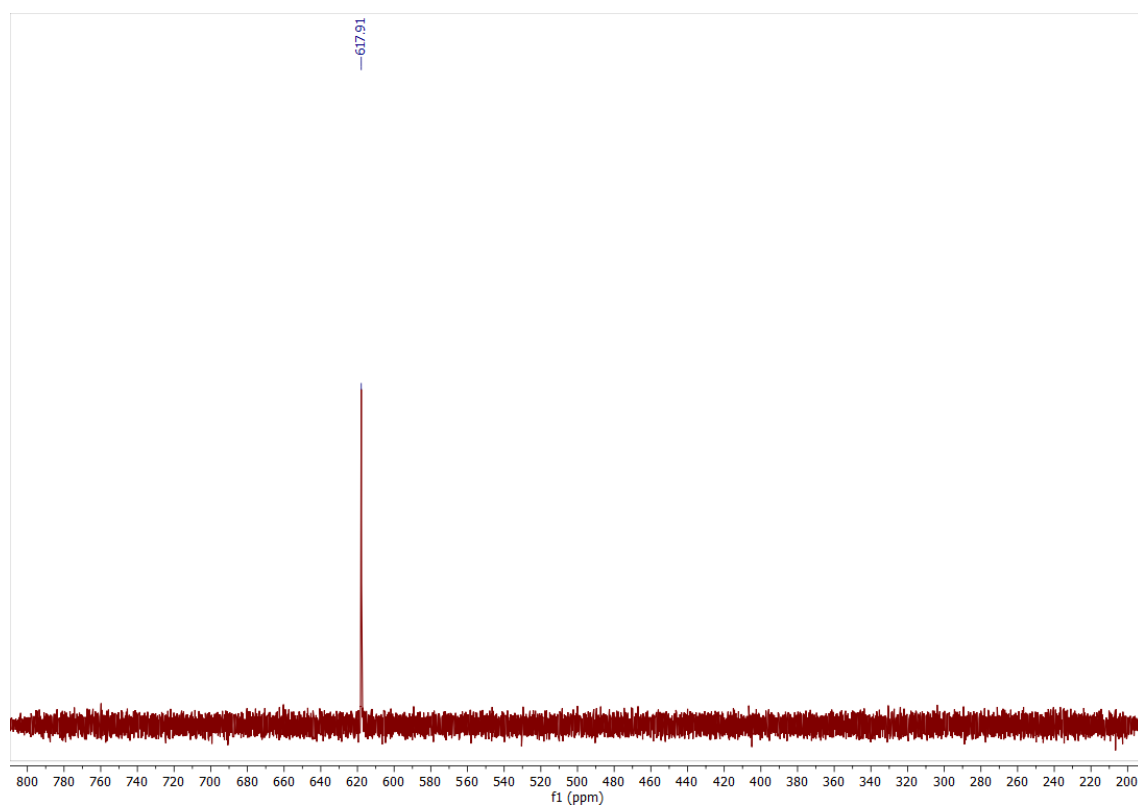


Figure S49. $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum of compound **8b.1** (CD_2Cl_2).

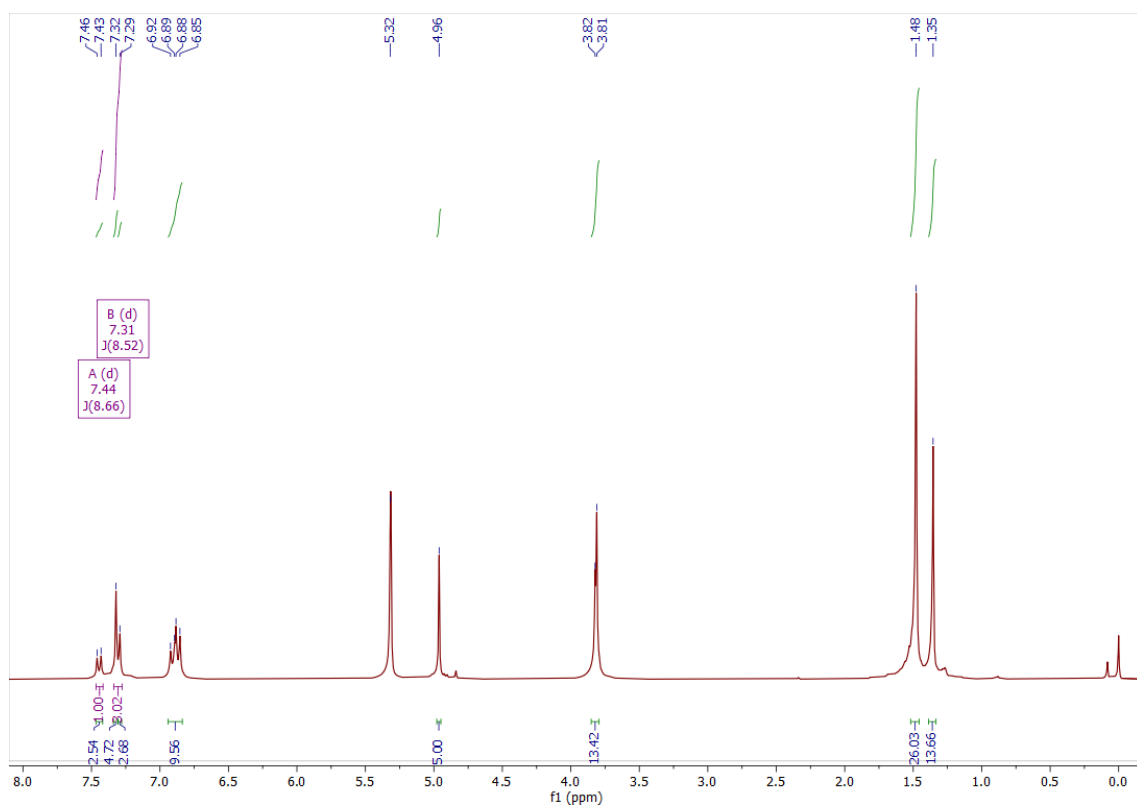


Figure S50. ¹H NMR spectrum of compound **8b.1** (CD₂Cl₂).

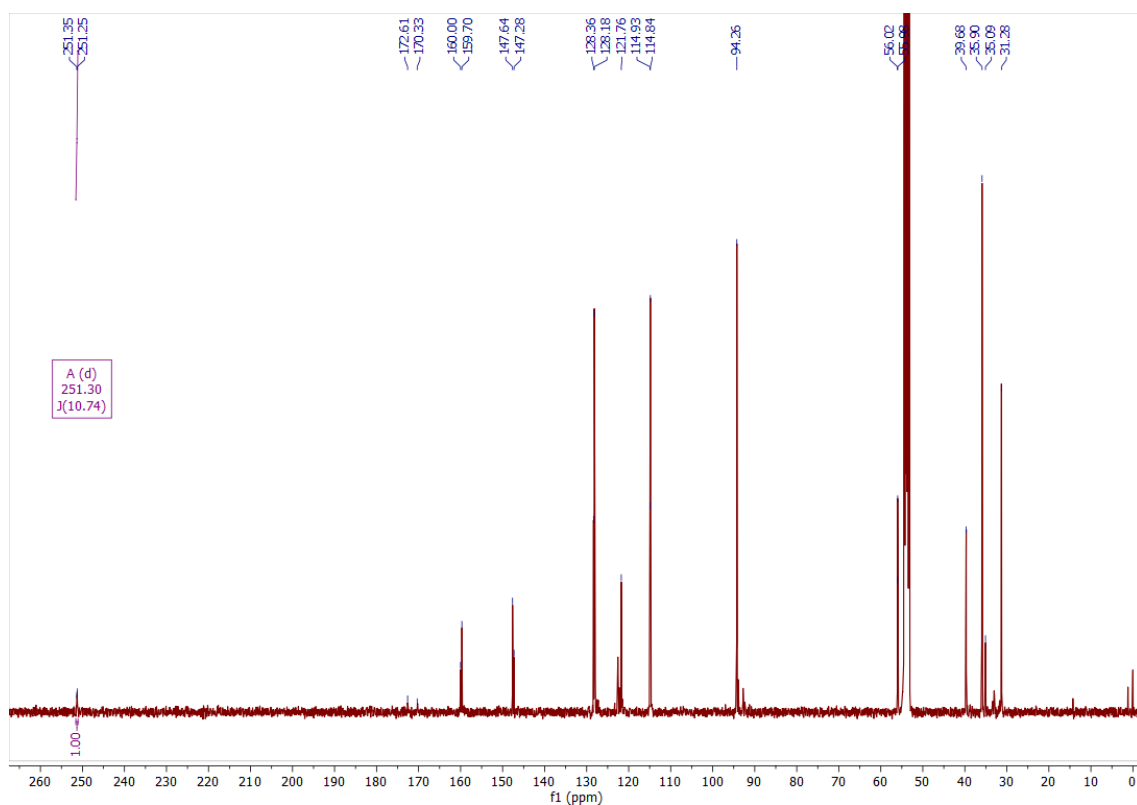


Figure S51. ¹³C{¹H} NMR spectrum of compound **8b.1** (CD₂Cl₂).