

Supplementary Materials II – NMR and MS data

for

Photoluminescence of Ni(II), Pd(II), and Pt(II) complexes [M(Me₂dpb)Cl] obtained from C–H activation of 1,5-di(2-pyridyl)-2,4-dimethylbenzene (Me₂dpbH)

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Figure S024. 300 MHz ¹H NMR spectrum of [Pd₄(μ-κ²,κ²dpb)₂(μ-κ¹,κ¹-OAc)₄] with traces of [Pd(PyPhPy)Cl] in CD₂Cl₂.

Figure S025. EI-MS(+) of the product mixture of [Pd₄(μ-κ²,κ²dpb)₂(μ-κ¹,κ¹-OAc)₄] with traces of [Pd(PyPhPy)Cl].

Figure S026. 300 MHz ¹H NMR spectrum of [Pd₄(μ-κ²,κ²dpb)₂(μ-κ¹,κ¹-OPiv)₄] in DMSO-d₆.

Figure S027. UV-vis absorption spectra of [Pd₄(μ-κ²,κ²dpb)₂(μ-κ¹,κ¹-OAc)₄] and [Pd₄(μ-κ²,κ²dpb)₂(μ-κ¹,κ¹-OPiv)₄] in THF.

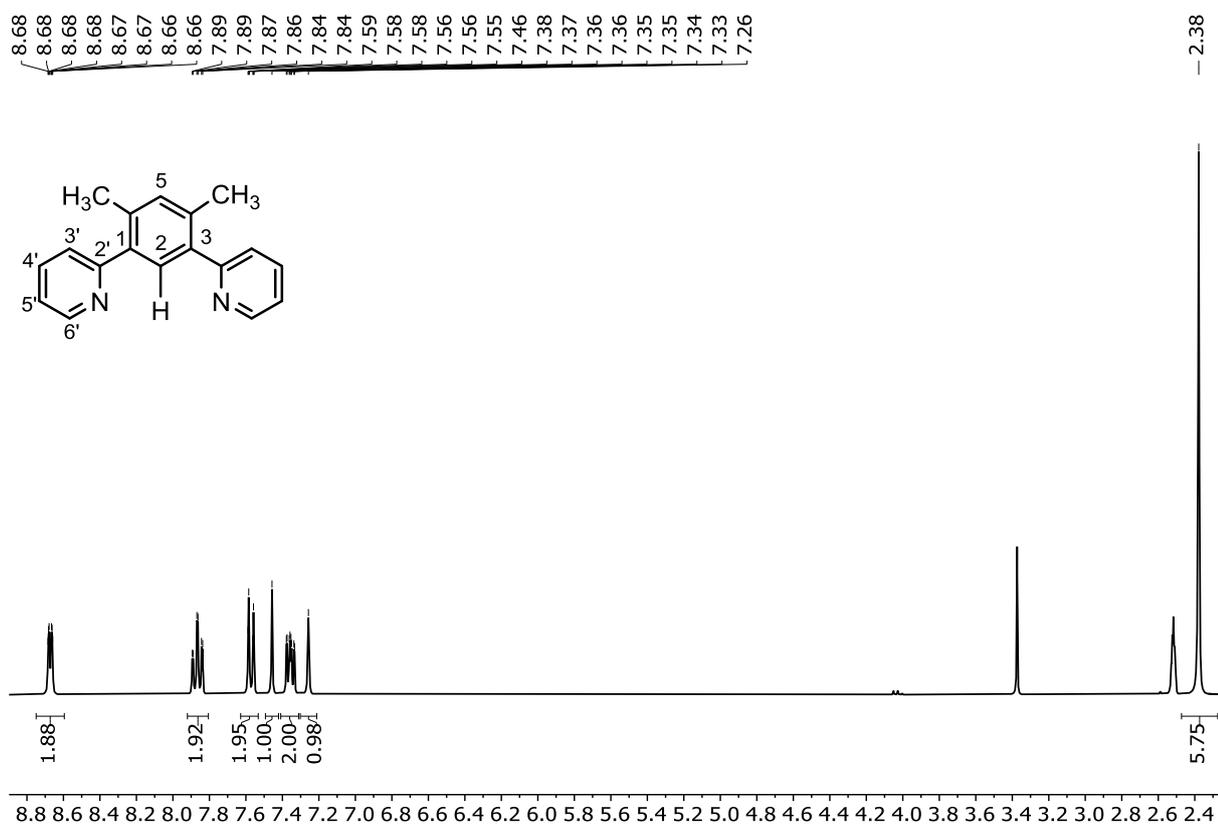


Figure. S001. 300 MHz ¹H-NMR spectrum of 1,5-di(2-pyridyl)-2,4-dimethylbenzene measured in DMSO-d₆ at RT.

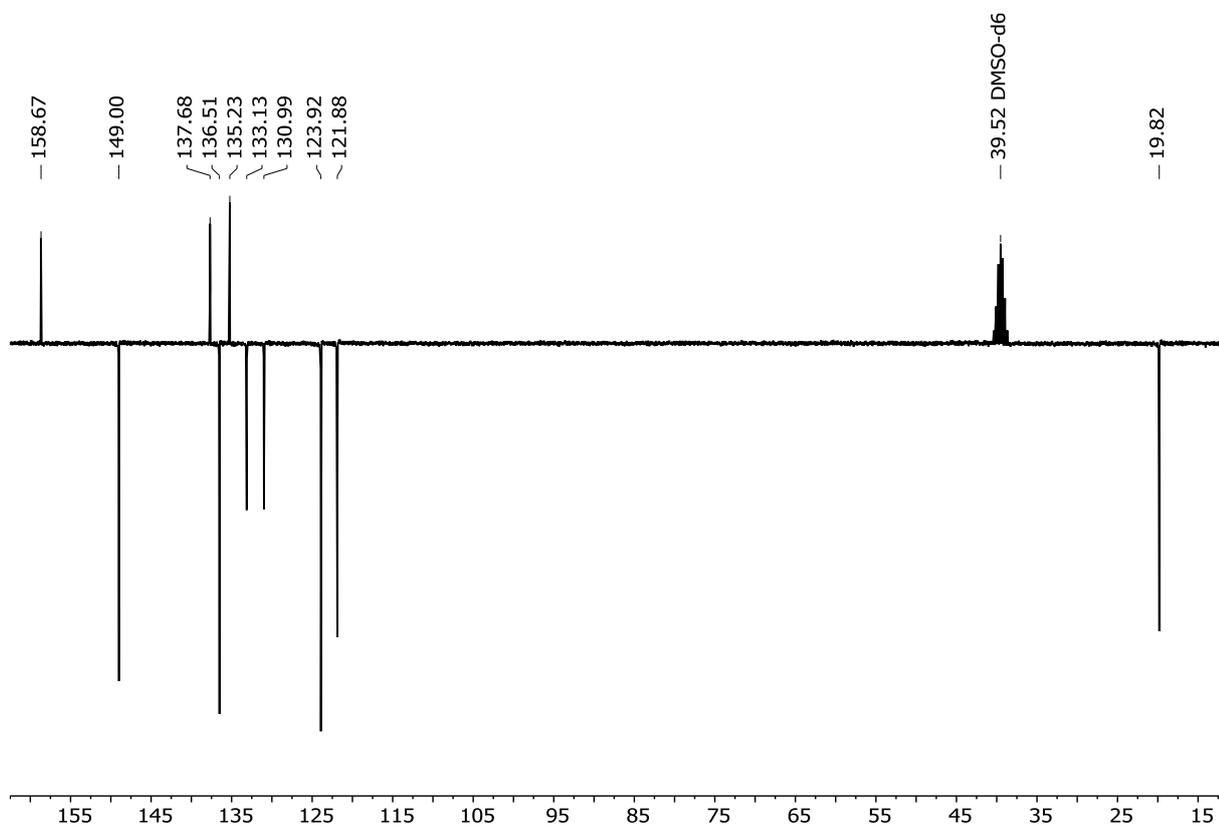


Figure. S002. 75 MHz ¹³C-NMR spectrum of 1,5-di(2-pyridyl)-2,4-dimethylbenzene measured in DMSO-d₆ at RT.



Figure. S003.H,C-HSQC spectrum of 1,5-di(2-pyridyl)-2,4-dimethylbenzene measured in DMSO-d₆ at RT.

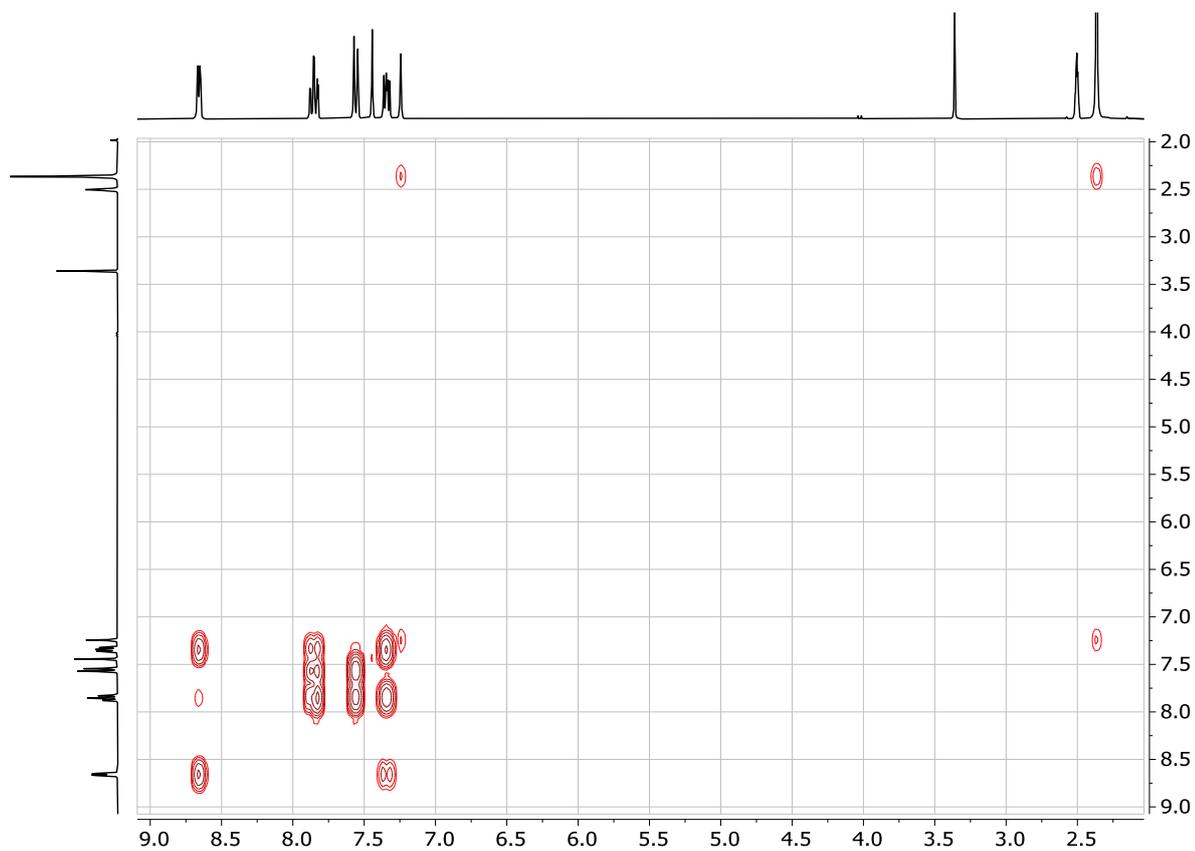


Figure. S004. H,H-COSY spectrum of 1,5-di(2-pyridyl)-2,4-dimethylbenzene measured in DMSO-d₆ at RT.

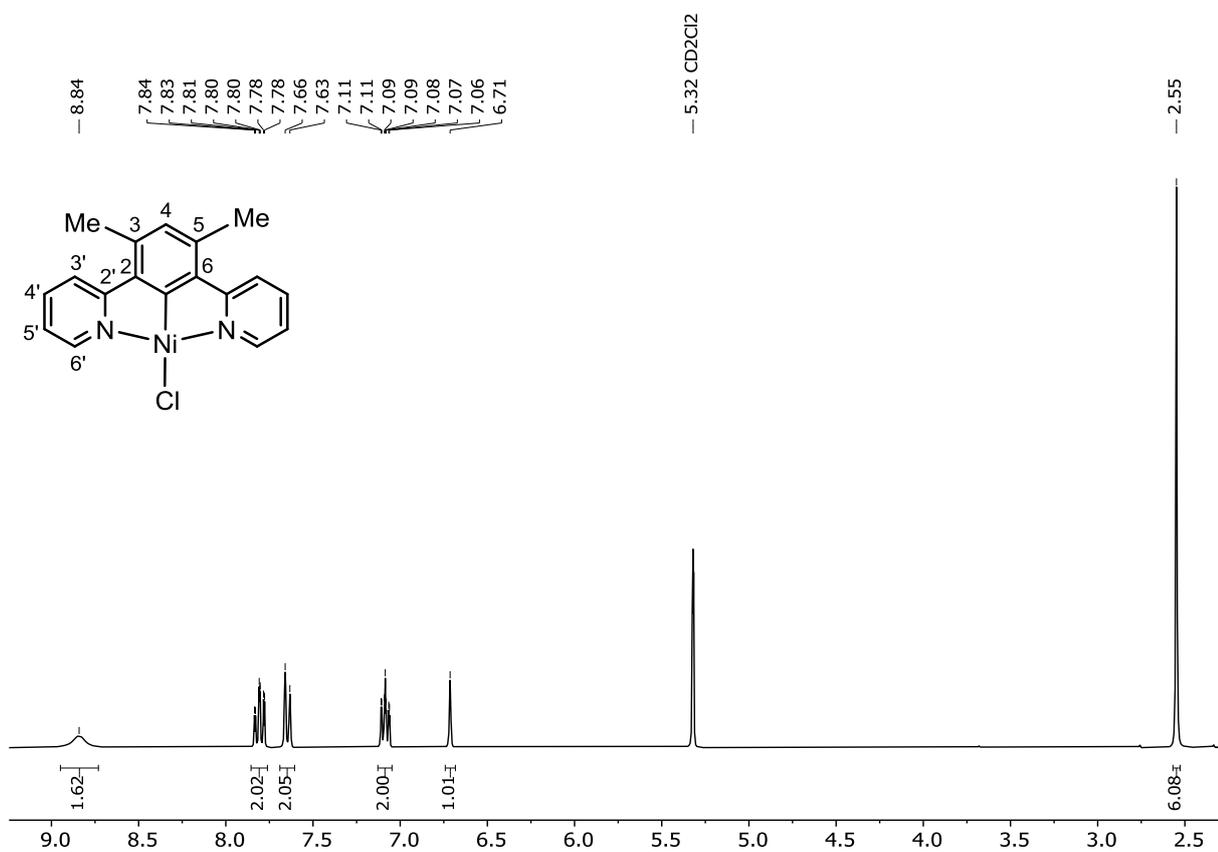


Figure. S005. 300 MHz ¹H-NMR spectrum of [Ni(Me₂dpb)Cl] measured in CD₂Cl₂ at RT.

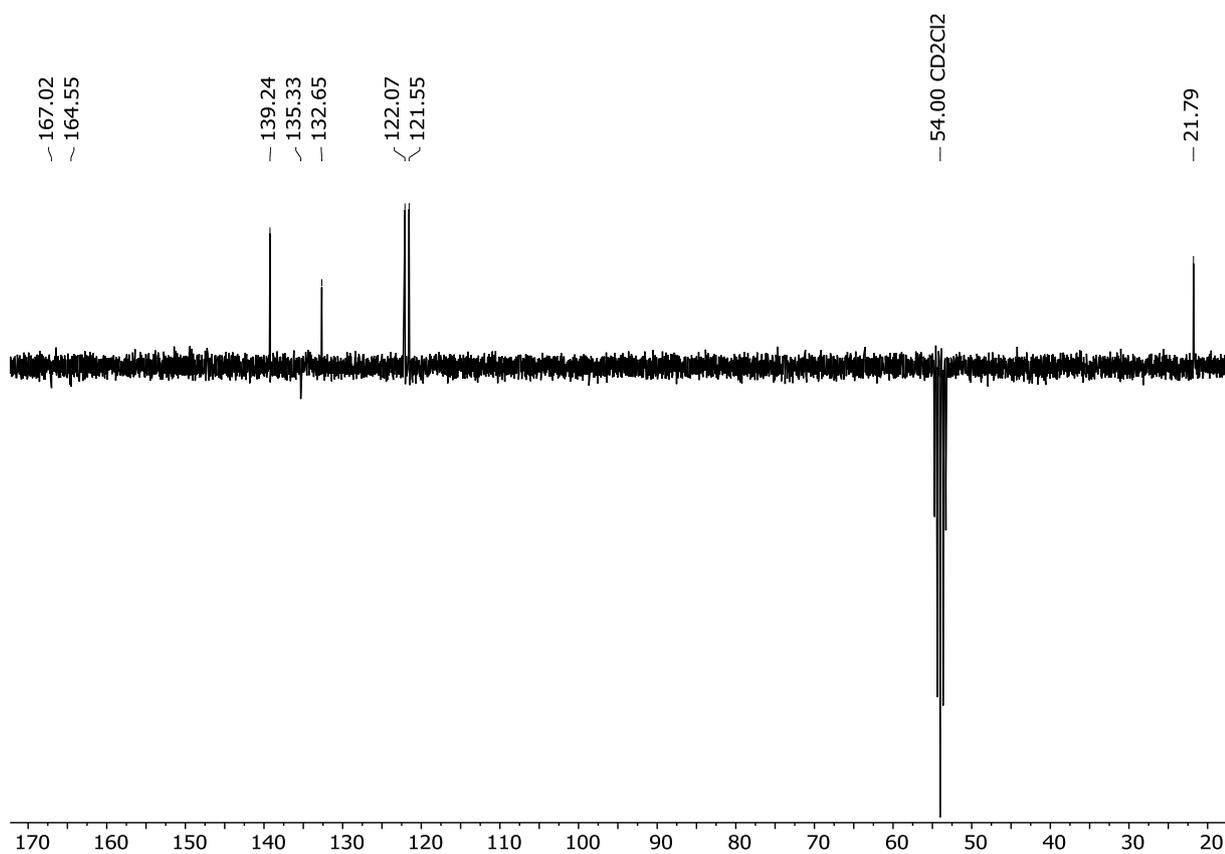


Figure. S006. 75 MHz ¹³C-NMR spectrum of [Ni(Me₂dpb)Cl] measured in CD₂Cl₂ at RT.

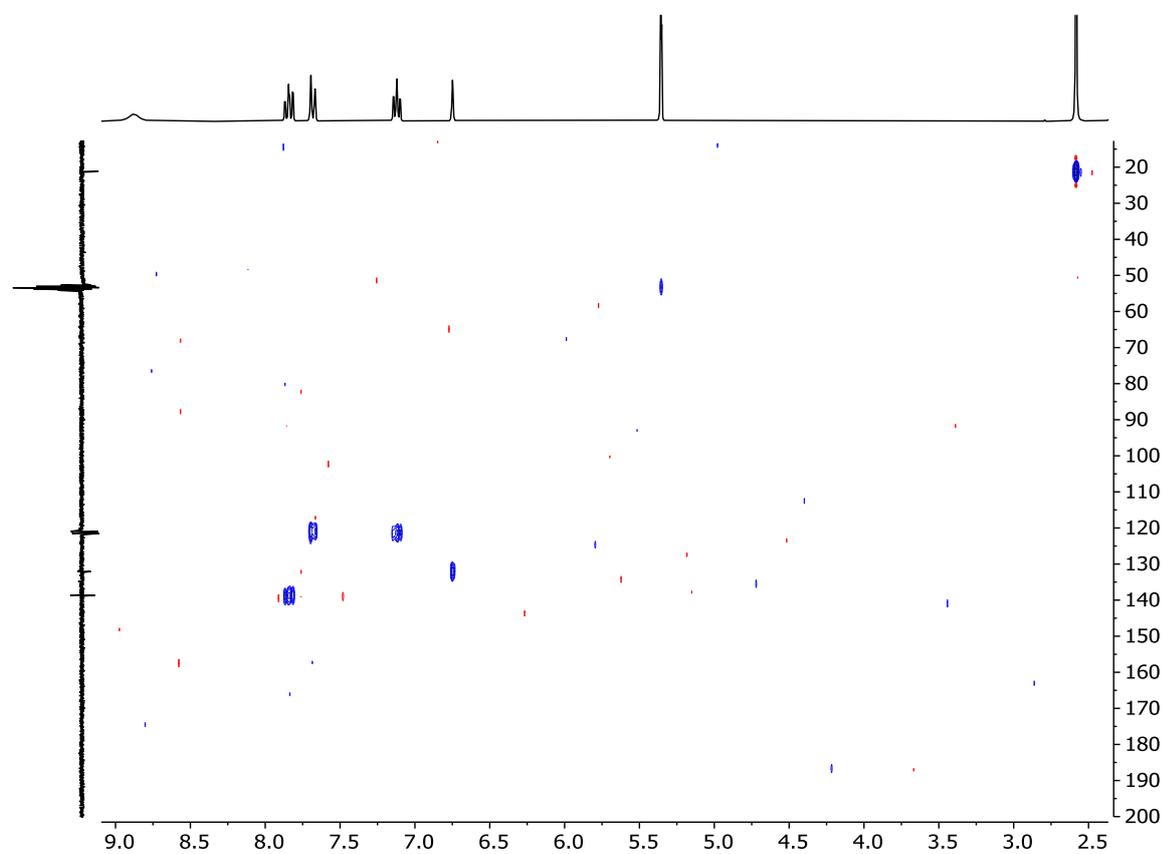


Figure. S007. ^1H , ^{13}C -HSQC spectrum of $[\text{Ni}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in CD_2Cl_2 at RT.

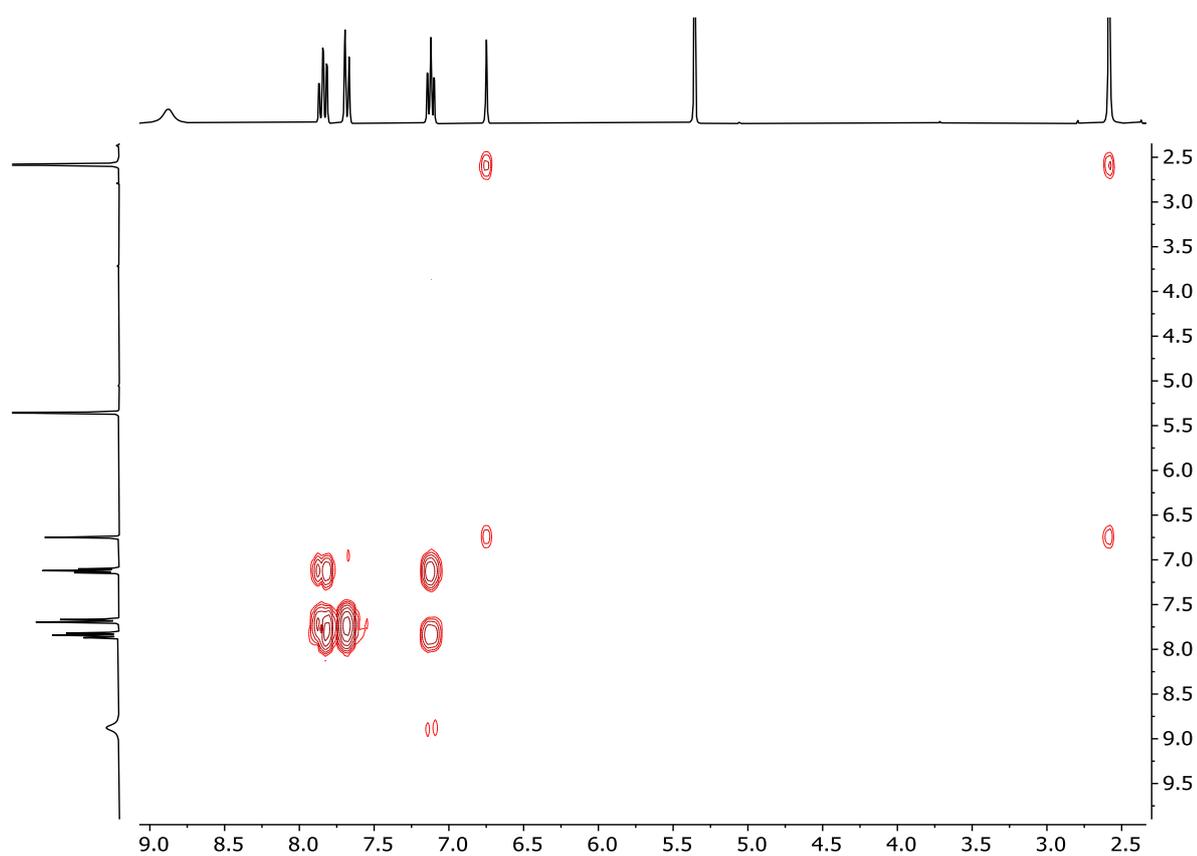


Figure. S008. ^1H , ^1H -COSY spectrum of $[\text{Ni}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in CD_2Cl_2 at RT.

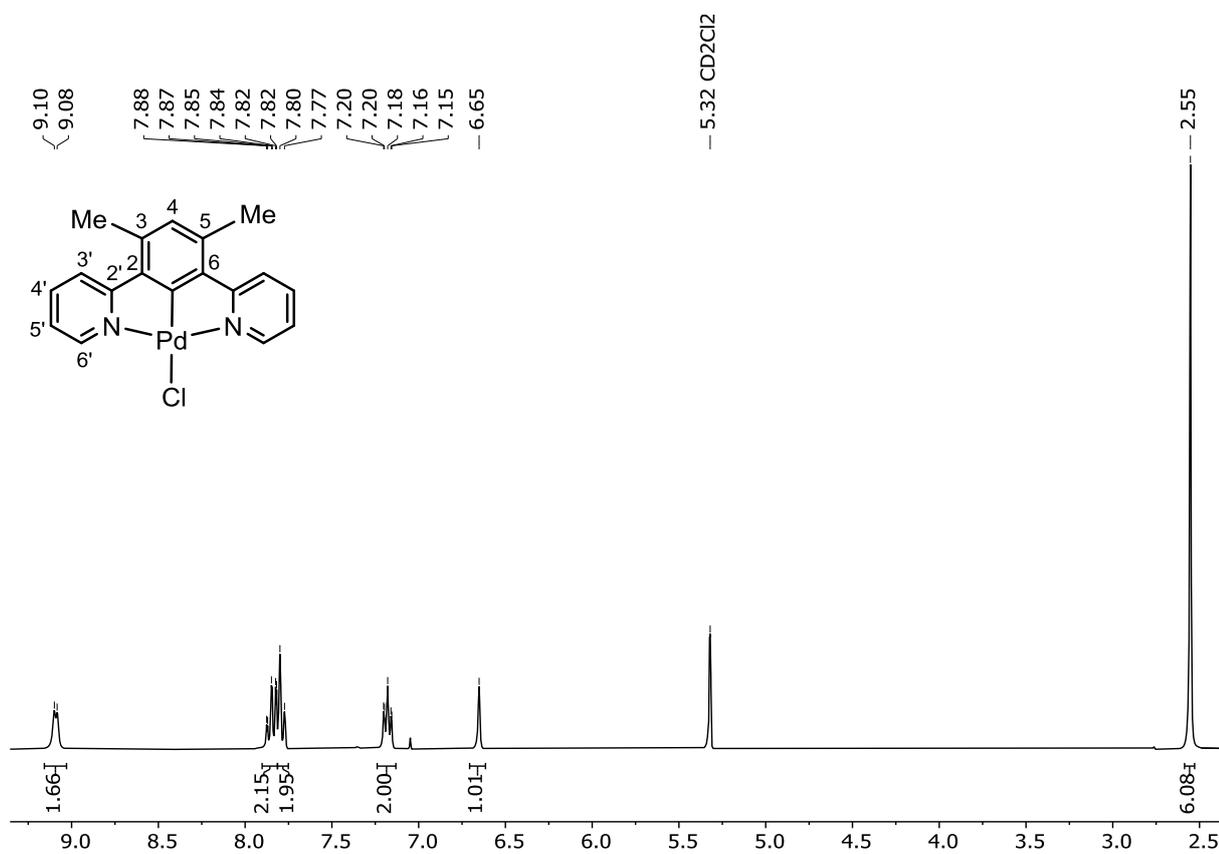


Figure. S009. 300 MHz ¹H-NMR spectrum of [Pd(Me₂dpb)Cl] measured in CD₂Cl₂ at RT.

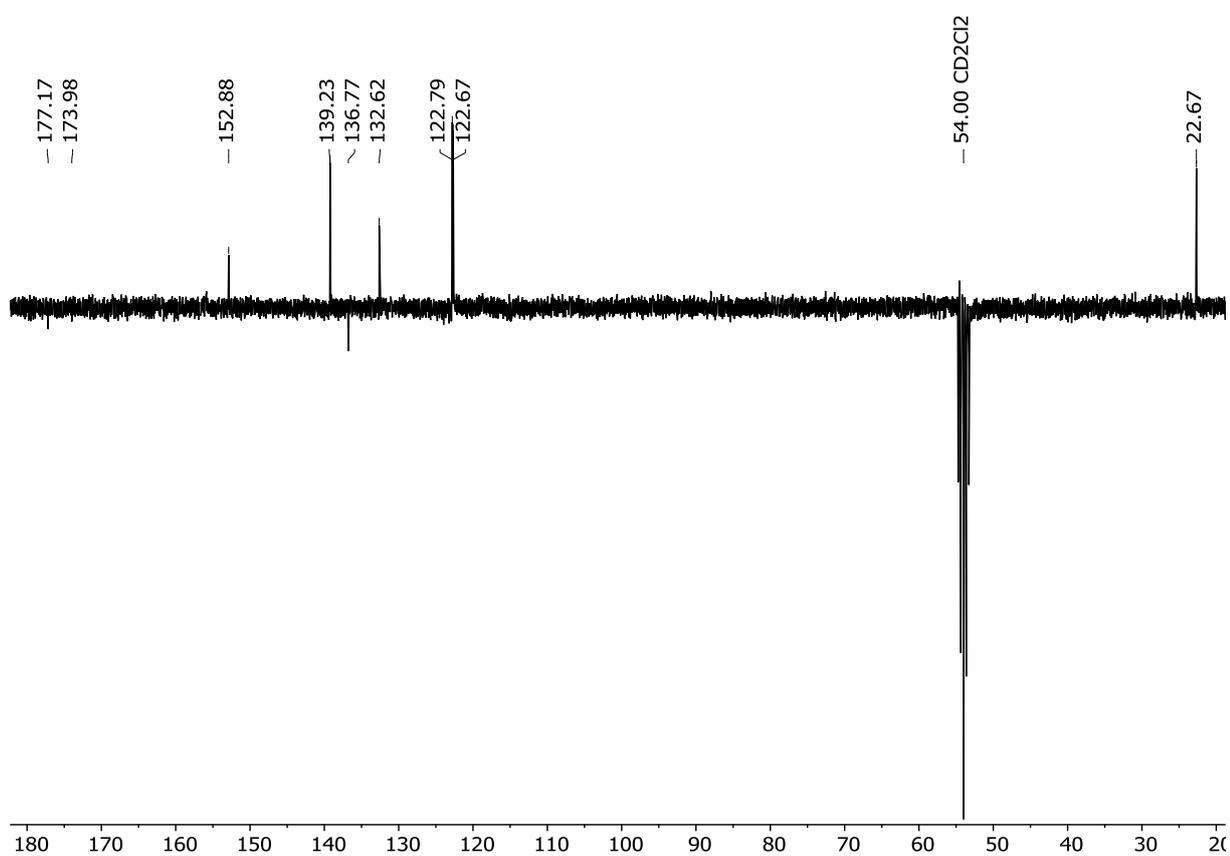


Figure. S010. 75 MHz ¹³C-NMR spectrum of [Pd(Me₂dpb)Cl] measured in CD₂Cl₂ at RT.

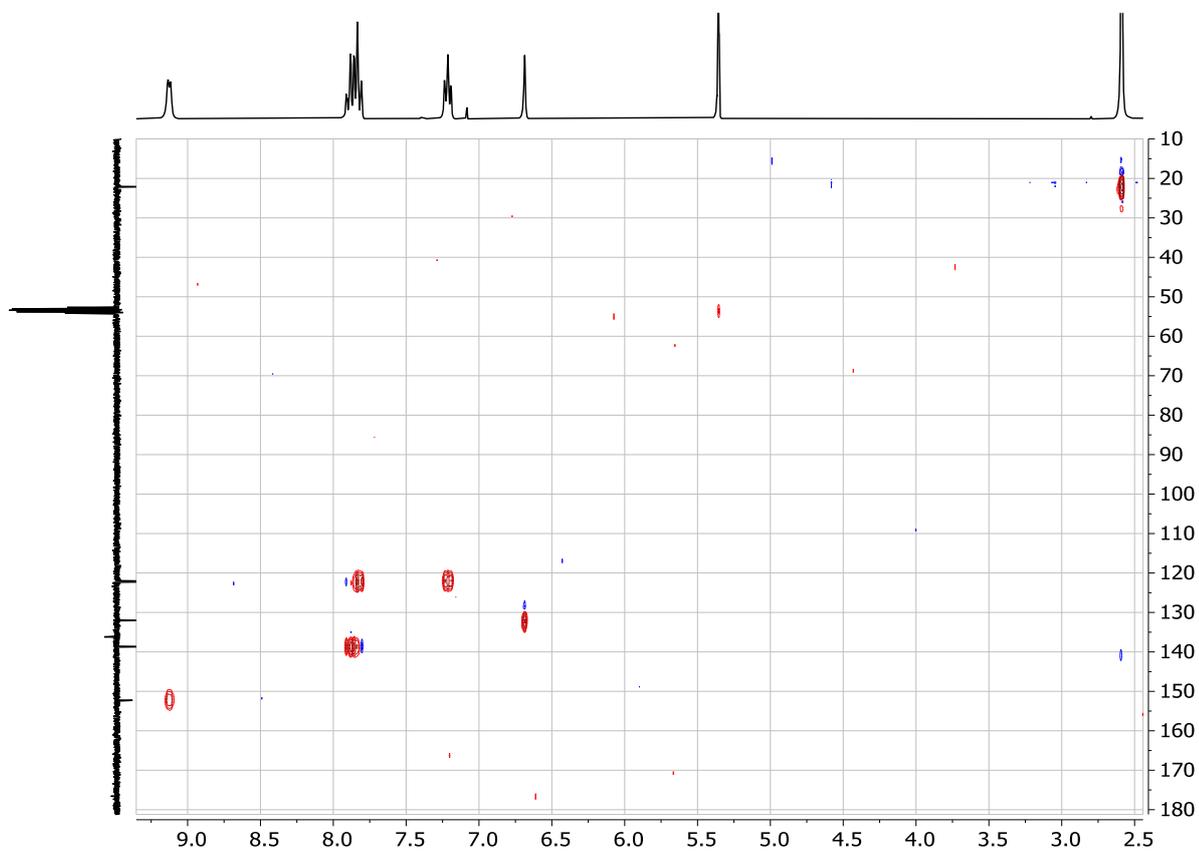


Figure. S011. $^1\text{H}, ^{13}\text{C}$ -HSQC spectrum of $[\text{Pd}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in CD_2Cl_2 at RT.

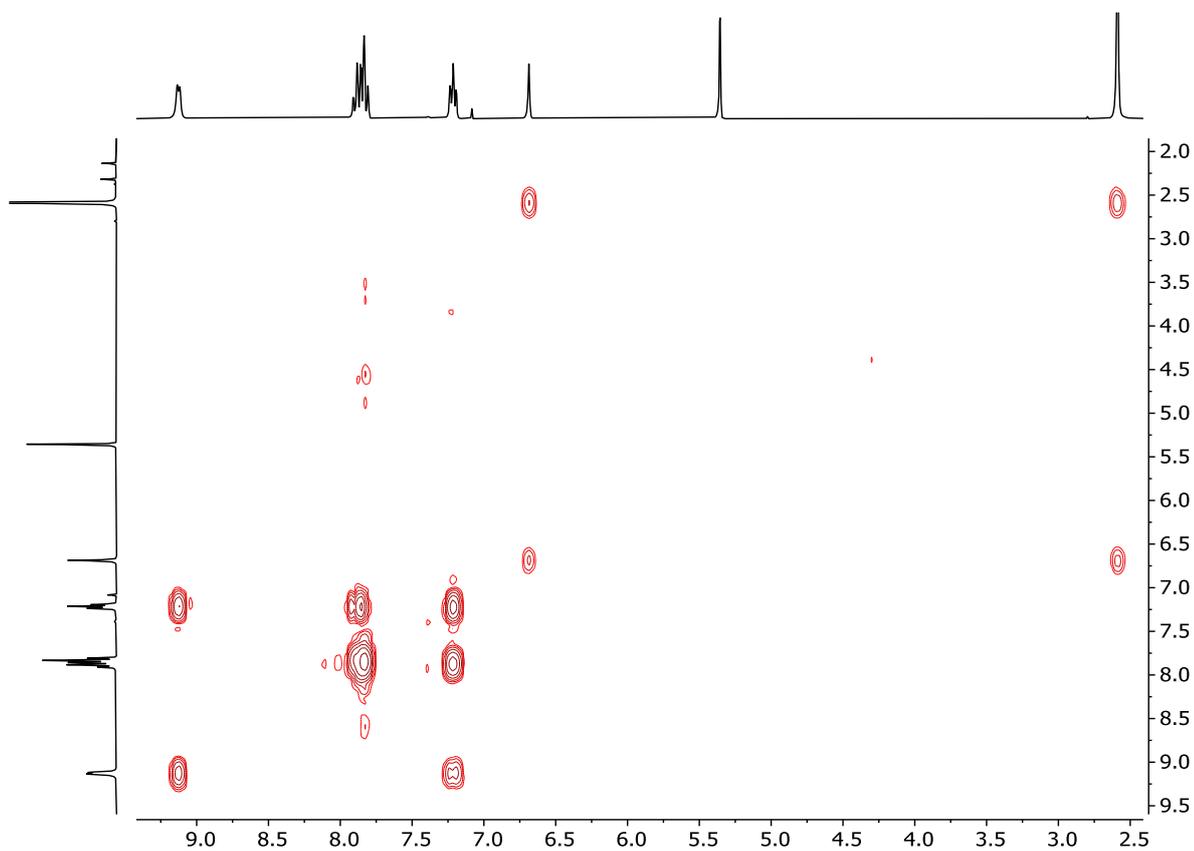


Figure. S012. $^1\text{H}, ^1\text{H}$ -COSY spectrum of $[\text{Pd}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in CD_2Cl_2 at RT.

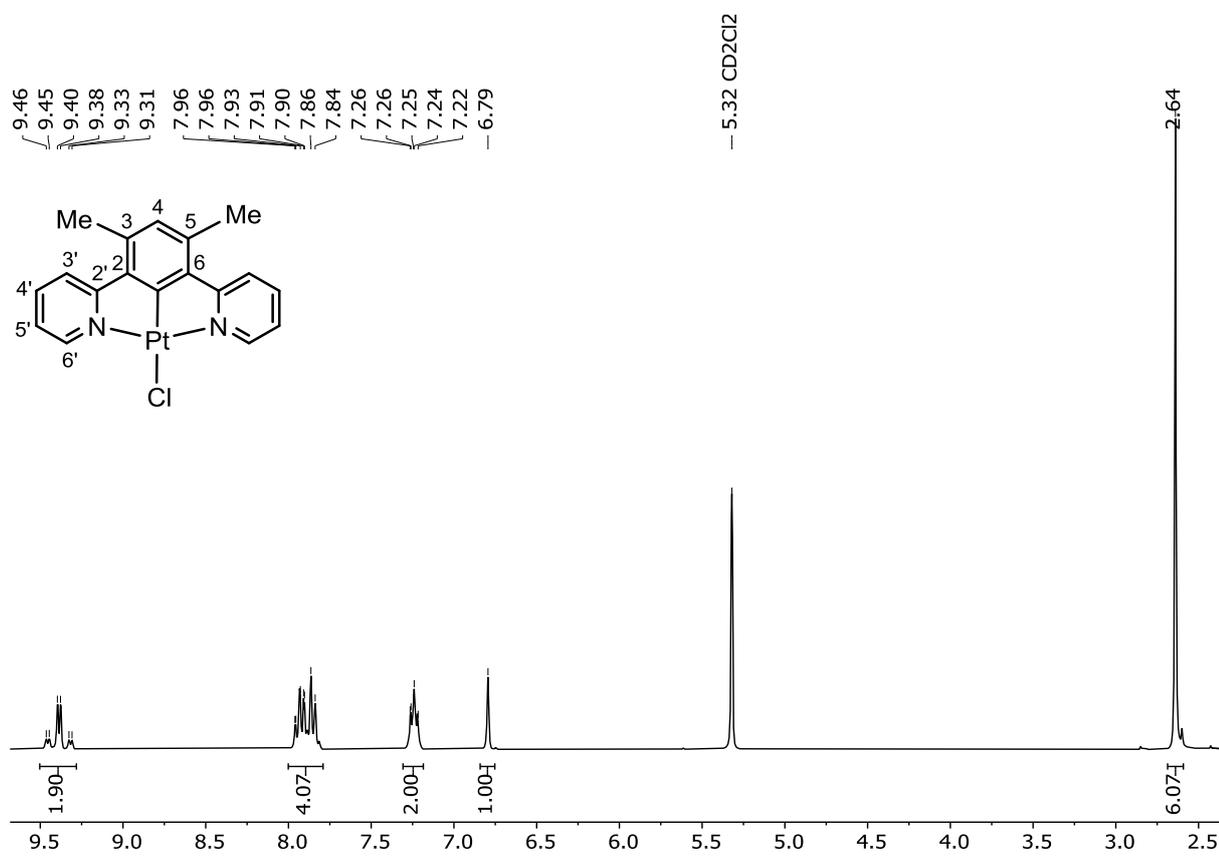


Figure. S013. 300 MHz ¹H-NMR spectrum of [Pt(Me₂dpb)Cl] measured in CD₂Cl₂ at RT.

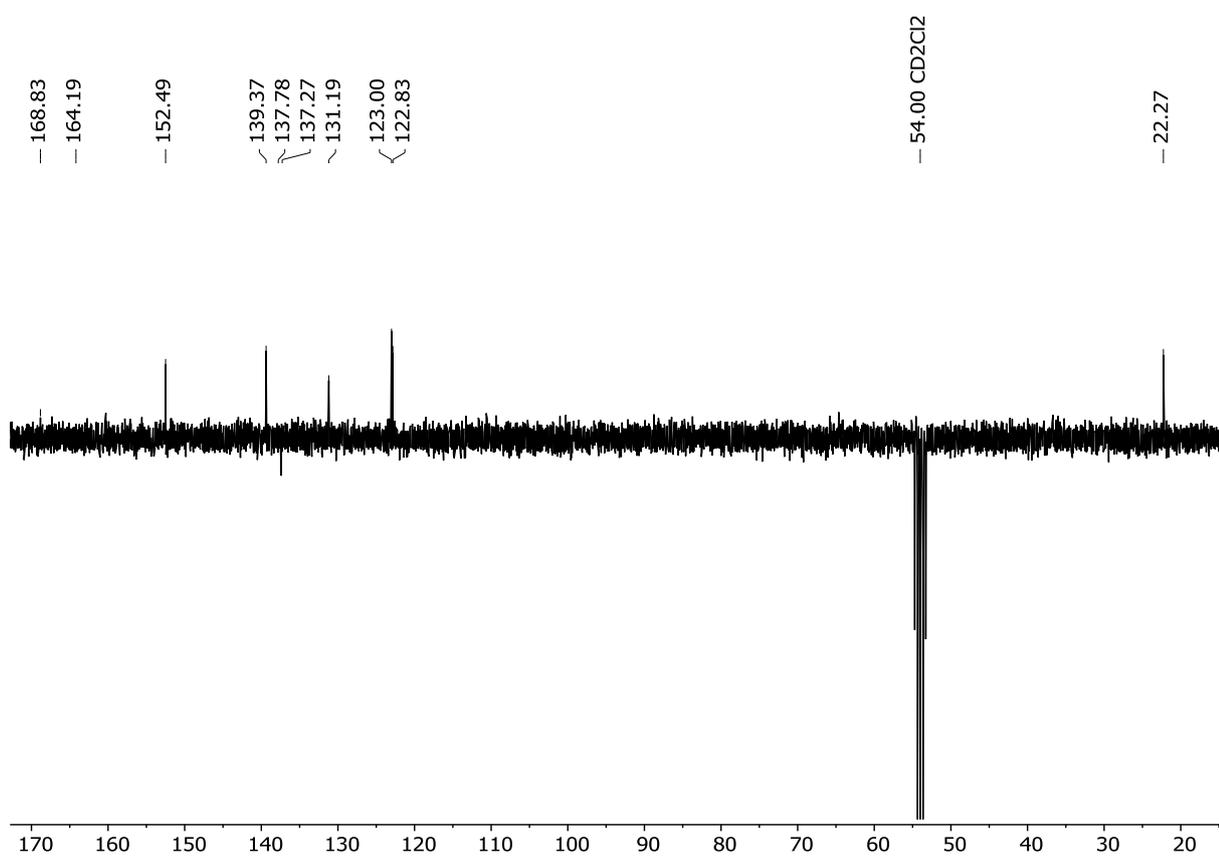


Figure. S014. 75 MHz ¹³C-NMR spectrum of [Pt(Me₂dpb)Cl] measured in CD₂Cl₂ at RT.

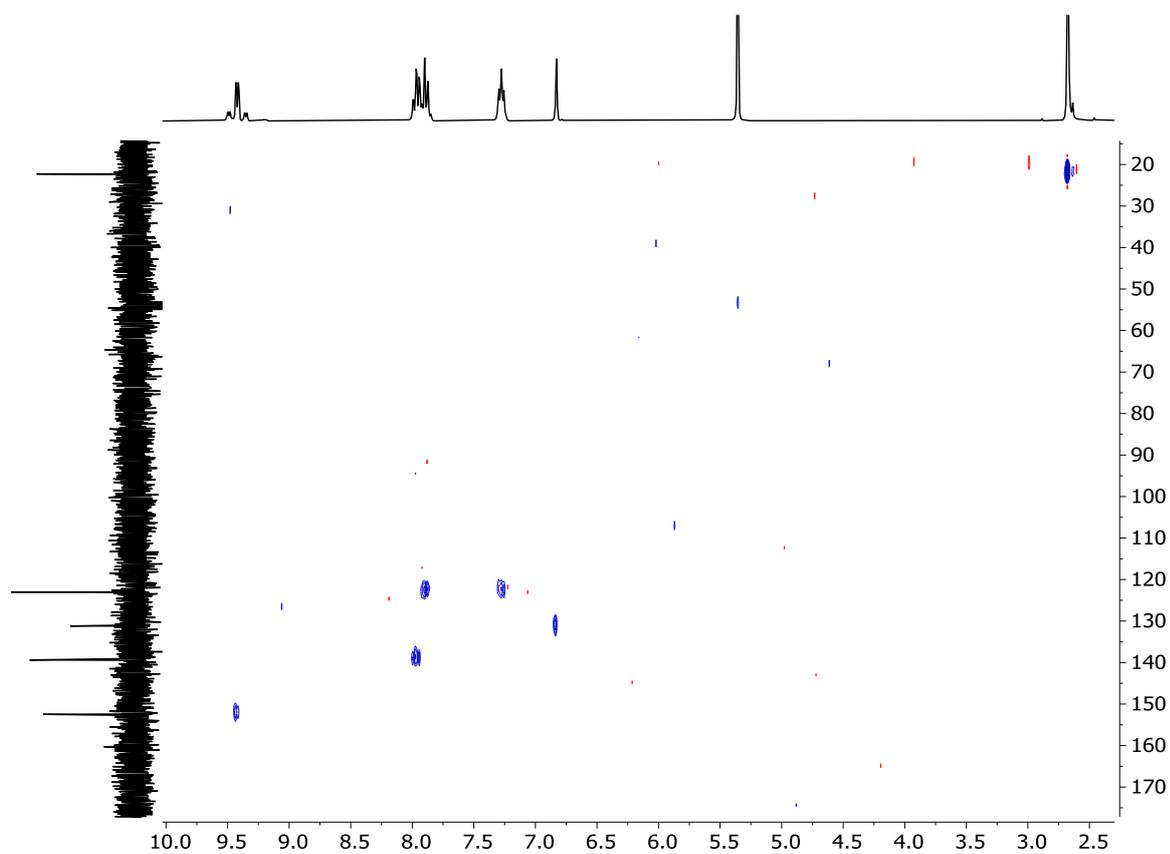


Figure. S015. $^1\text{H}, ^{13}\text{C}$ -HSQC spectrum of $[\text{Pt}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in CD_2Cl_2 at RT.

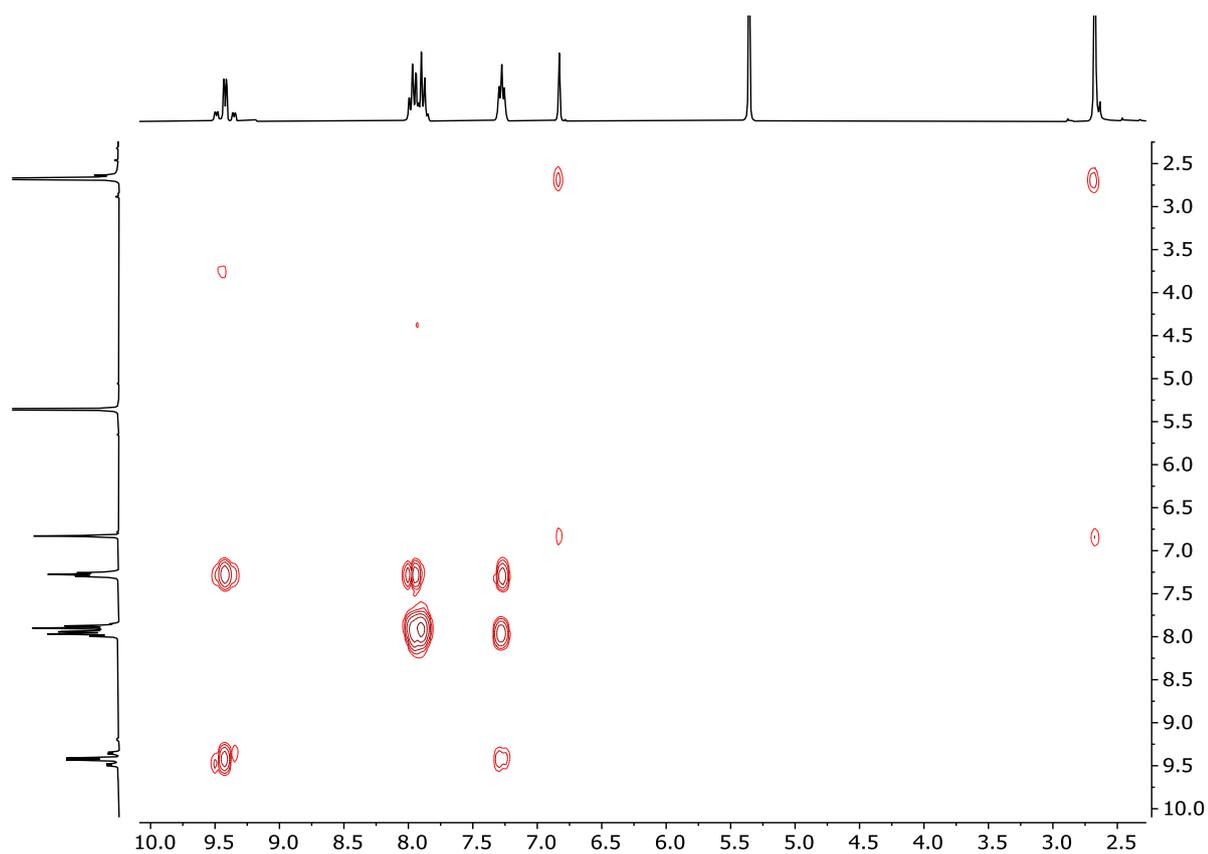
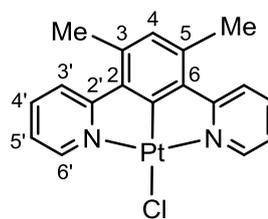


Figure. S016. $^1\text{H}, ^1\text{H}$ -COSY spectrum of $[\text{Pt}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in CD_2Cl_2 at RT.

— -3609.20



-3586 -3590 -3594 -3598 -3602 -3606 -3610 -3614 -3618 -3622 -3626 -3630 -3636

Figure. S017. $^{195}\text{Pt}\{^1\text{H}\}$ NMR spectrum of $[\text{Pt}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in DMSO-d_6 at RT.

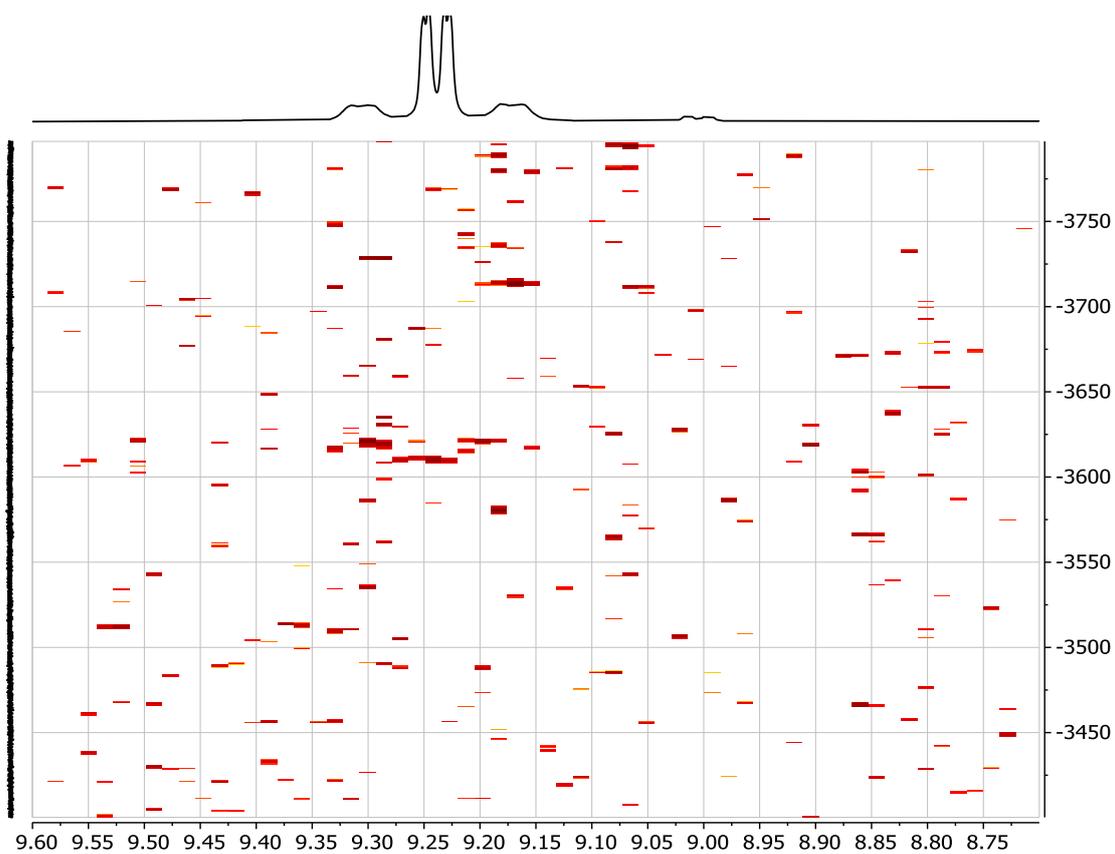


Figure. S018. $^1\text{H},\text{Pt}$ -HMBC spectrum of $[\text{Pt}(\text{Me}_2\text{dpb})\text{Cl}]$ measured in DMSO-d_6 at RT.

SPEC: k1e600 04-Dec-20 Elapse: 02:58.4 27
 Samp: ALK 06 C18H16N2 Start: 09:36:50 28
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet: 16 > 500
 Base: 259.2 Inten: 64081152 Masses: 16 > 500
 Norm: 259.2 RIC: 342794867 #peaks: 524
 Peak: 1000.00 mmu

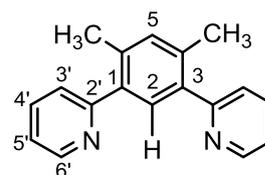
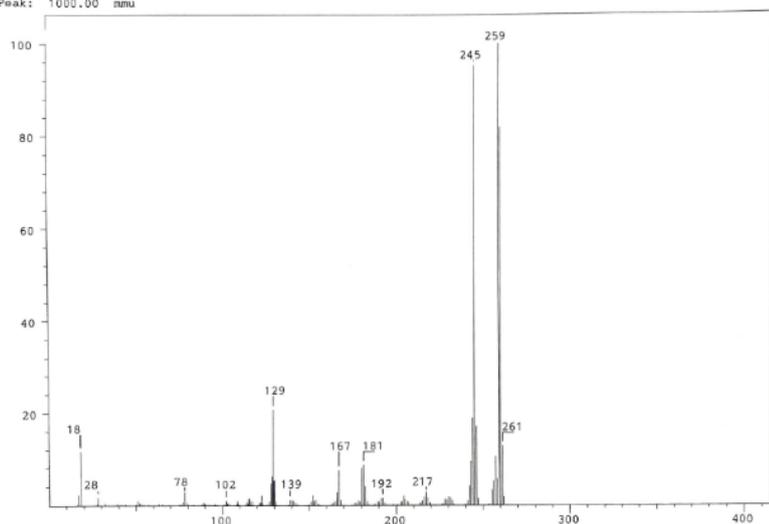
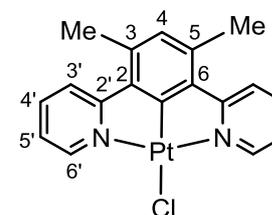
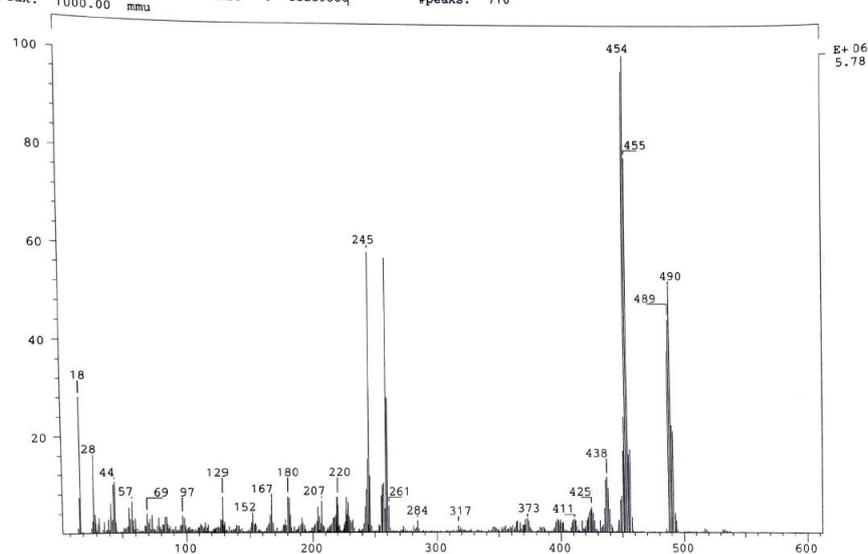


Figure. S019. MS-EI(+) of 1,5-di(2-pyridyl)-2,4-dimethylbenzene (Me₂dpbH).

SPEC: k1e4598 30-Nov-20 Elapse: 07:05.4 64
 Samp: ALK 13 C18H15N2PtCl Start: 09:56:21 65
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet: 16 > 600
 Base: 453.8 Inten: 5780745 Masses: 16 > 600
 Norm: 453.8 RIC: 83289886 #peaks: 710
 Peak: 1000.00 mmu



SPEC: k1e4598 30-Nov-20 Elapse: 05:08.3 46
 Samp: ALK 13 C18H15N2PtCl Start: 09:56:21 65
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet: 16 > 600
 Base: 18.2 Inten: 1586063 Masses: 16 > 600
 Norm: 453.8 RIC: 15687393 #peaks: 640
 Peak: 1000.00 mmu

SPEC: k1e4598 30-Nov-20 Elapse: 06:26.4 58
 Samp: ALK 13 C18H15N2PtCl Start: 09:56:21 65
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet: 16 > 600
 Base: 245.3 Inten: 4283544 Masses: 16 > 600
 Norm: 489.7 RIC: 69849582 #peaks: 715
 Peak: 1000.00 mmu

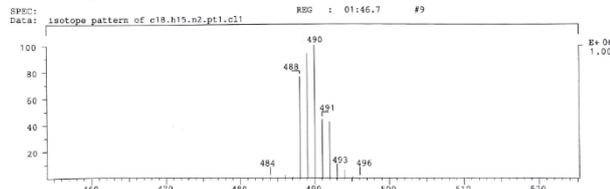
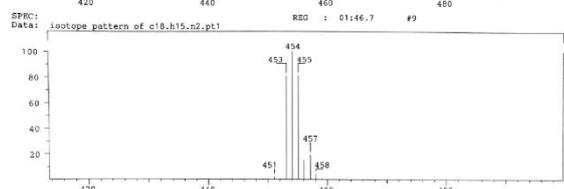
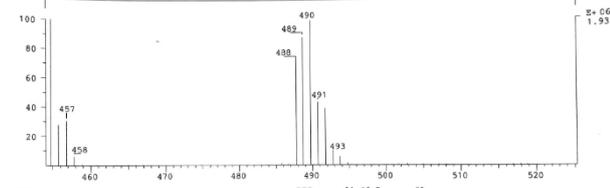
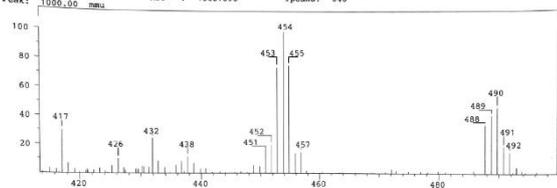
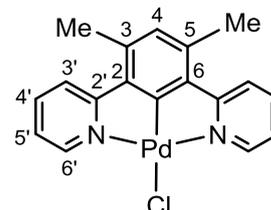
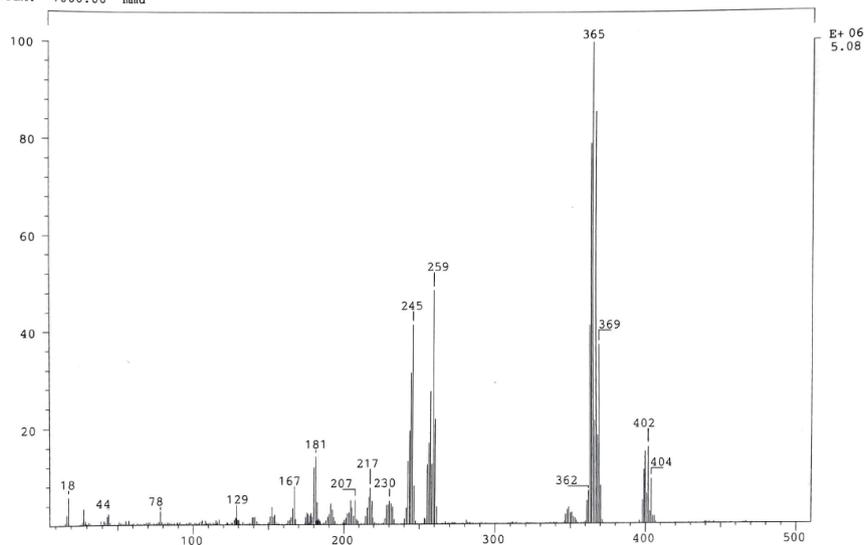


Figure. S020. MS-EI(+) of [Pt(Me₂dpb)Cl].

SPEC: kle4595 27-Nov-20 Elapse: 07:41.0 73
 Samp: ALK 09 C18H15N2PdCl Start : 09:20:21 74
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet :
 Base: 365.0 Inten : 5076796 Masse: 16 > 500
 Norm: 365.0 RIC : 50336045 #peaks: 544
 Peak: 1000.00 mmu



SPEC: kle4595 27-Nov-20 Elapse: 07:16.3 69
 Samp: ALK 09 C18H15N2PdCl Start : 09:20:21 74
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet :
 Base: 365.1 Inten : 1471843 Masse: 16 > 500
 Norm: 365.1 RIC : 16976970 #peaks: 418
 Peak: 1000.00 mmu

SPEC: kle4595 27-Nov-20 Elapse: 07:16.3 69
 Samp: ALK 09 C18H15N2PdCl Start : 09:20:21 74
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet :
 Base: 402.0 Inten : 1471843 Masse: 16 > 500
 Norm: 402.0 RIC : 16976970 #peaks: 418
 Peak: 1000.00 mmu

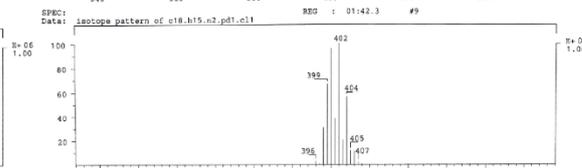
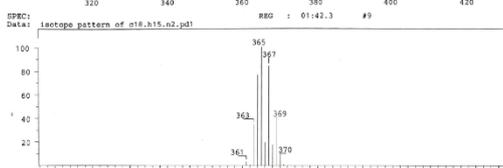
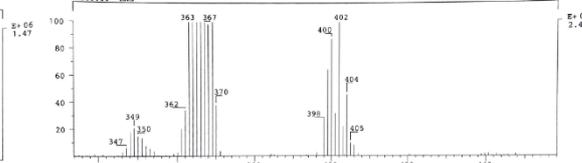
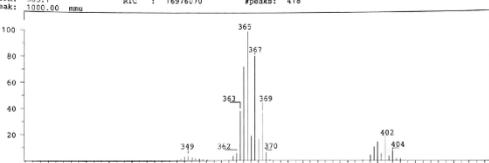


Figure. S021. MS-EI(+) of [Pd(Me2dpb)Cl].

SPEC: kle4593 26-Nov-20 Elapse: 05:26.7 51
 Samp: ALK 14 C18H15N2NiCl Start : 09:42:15 52
 Comm: EI pos 70eV
 Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
 Oper: A. Baum Client: L. Kletsch Inlet :
 Base: 317.2 Inten : 15718893 Masse: 16 > 500
 Norm: 317.2 RIC : 87888464 #peaks: 548
 Peak: 1000.00 mmu

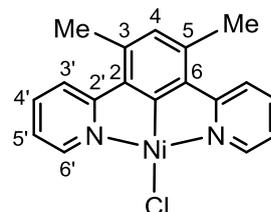
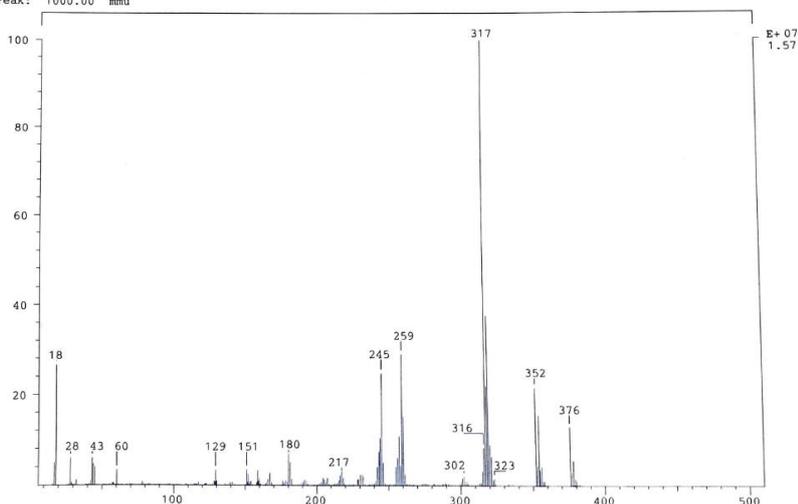


Figure. S022. MS-EI(+) of [Ni(Me2dpb)Cl]

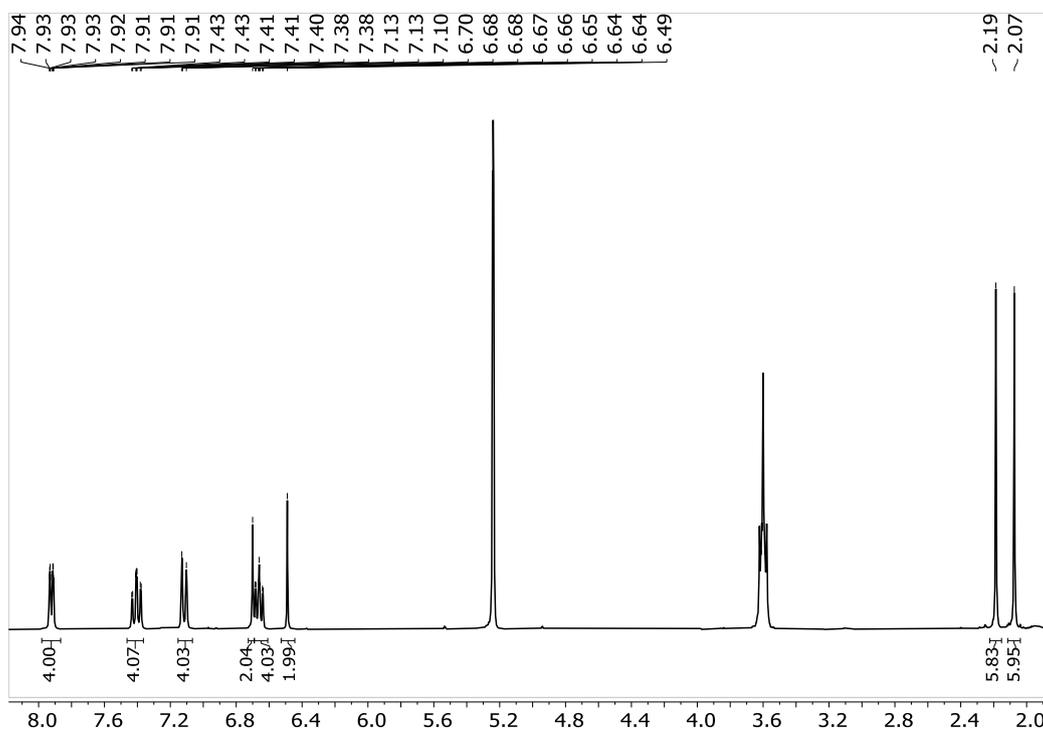


Figure S023. 300 MHz ^1H NMR spectrum of $[\text{Pd}_4(\mu\text{-}\kappa^2, \kappa^2\text{dpb})_2(\mu\text{-}\kappa^1, \kappa^1\text{-OAc})_4]$ in CD_2Cl_2 (synthetic route using PdCl_2).

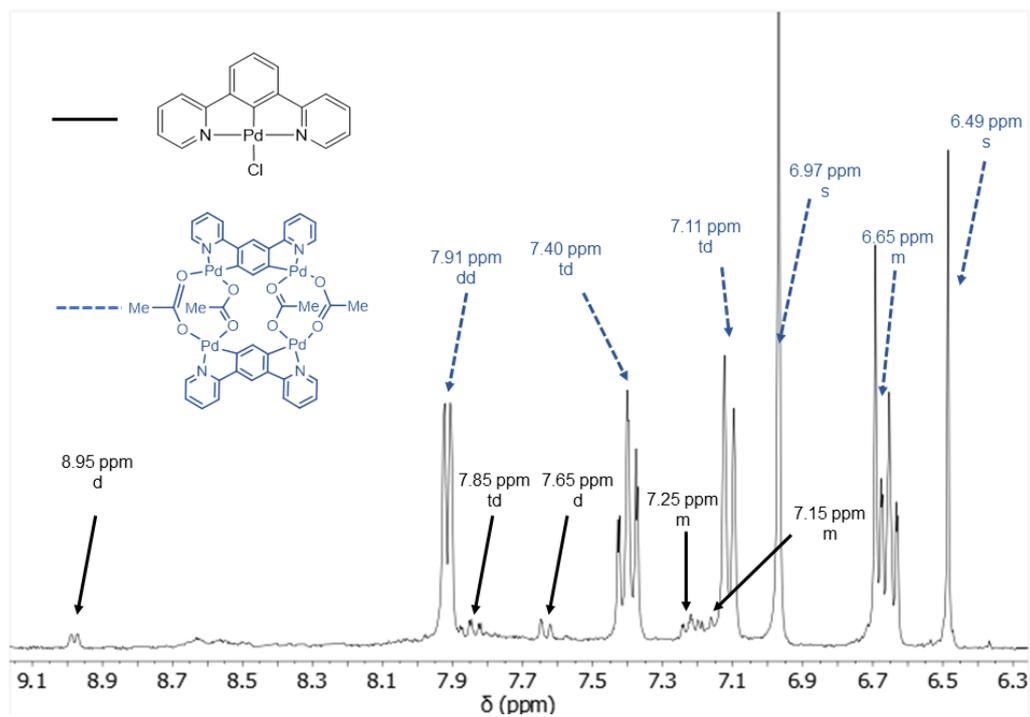


Figure S024. 300 MHz ^1H NMR spectrum of $[\text{Pd}_4(\mu\text{-}\kappa^2, \kappa^2\text{dpb})_2(\mu\text{-}\kappa^1, \kappa^1\text{-OAc})_4]$ with traces of $[\text{Pd}(\text{PyPhPy})\text{Cl}]$ in CD_2Cl_2 (synthetic route using $[(\text{COD})\text{PdCl}_2]$).

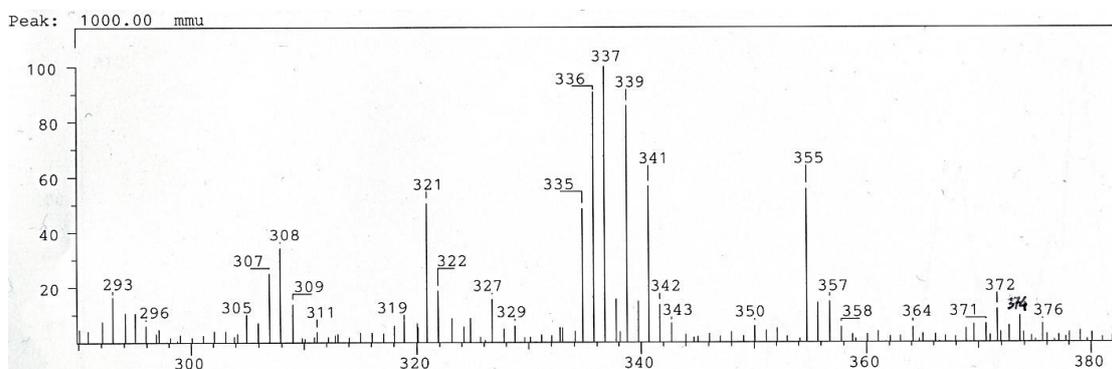


Figure S025. EI-MS(+) of the product mixture of $[\text{Pd}_4(\mu\text{-}\kappa^2, \kappa^2\text{dpb})_2(\mu\text{-}\kappa^1, \kappa^1\text{-OAc})_4]$ with traces of $[\text{Pd}(\text{dpb})\text{Cl}]$.

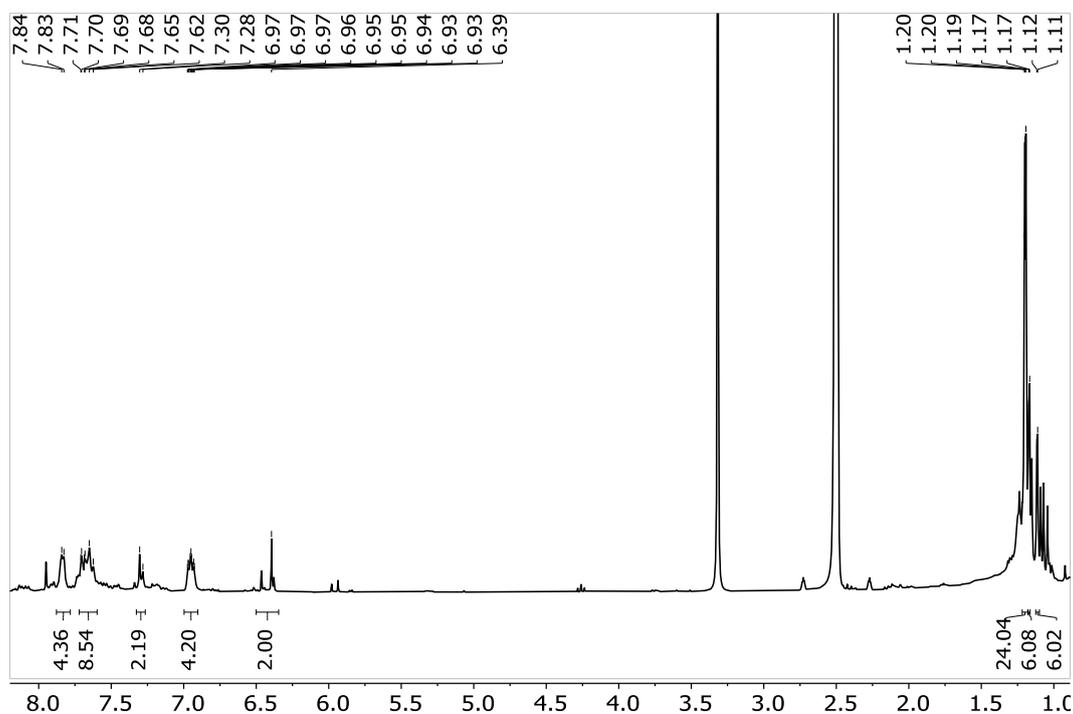


Figure S026. 300 MHz ^1H NMR spectrum of $[\text{Pd}_4(\mu\text{-}\kappa^2, \kappa^2\text{dpb})_2(\mu\text{-}\kappa^1, \kappa^1\text{-OPiv})_4]$ in DMSO-d_6 .

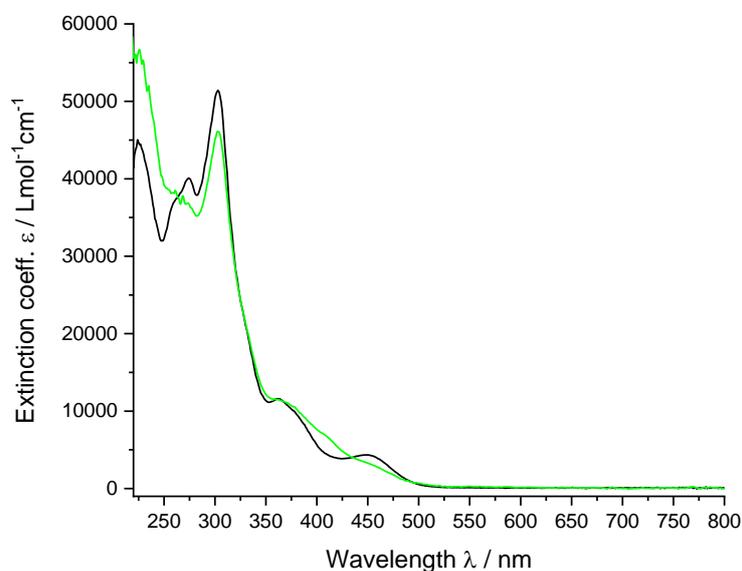


Figure S027. UV-vis absorption spectra of $[\text{Pd}_4(\mu\text{-}\kappa^2, \kappa^2\text{dpb})_2(\mu\text{-}\kappa^1, \kappa^1\text{-OAc})_4]$ (black) and $[\text{Pd}_4(\mu\text{-}\kappa^2, \kappa^2\text{dpb})_2(\mu\text{-}\kappa^1, \kappa^1\text{-OPiv})_4]$ (green) in THF.