

# European Journal of Inorganic Chemistry

Supporting Information

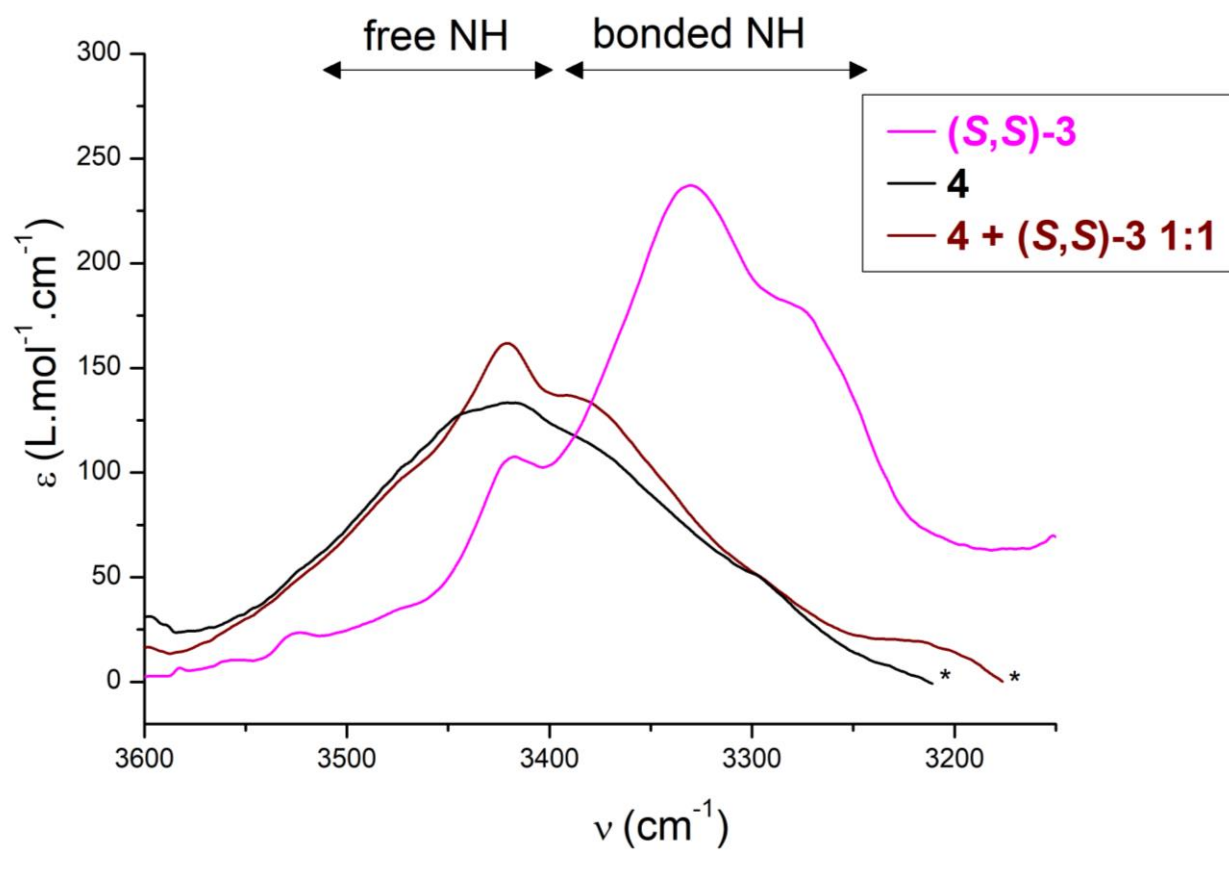
## **Cyclometallated Pt(II) Complexes Containing a Functionalized Bis-Urea Alkynyl Ligand: Probing Aggregation Mediated by Hydrogen Bonds *versus* Pt...Pt and $\pi$ – $\pi$ Interactions**

Gaëtan Basuyaux, Anissa Amar, Claire Troufflard, Abdou Boucekkine, Rémi Métivier,  
Matthieu Raynal,\* Jamal Moussa, Laurent Bouteiller, and Hani Amouri\*

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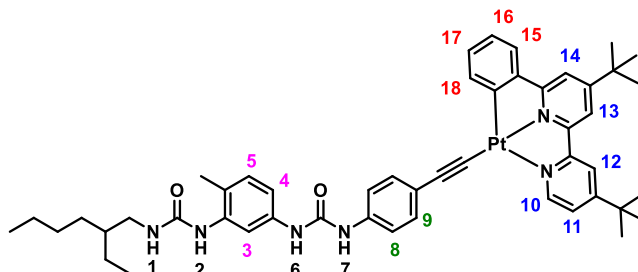
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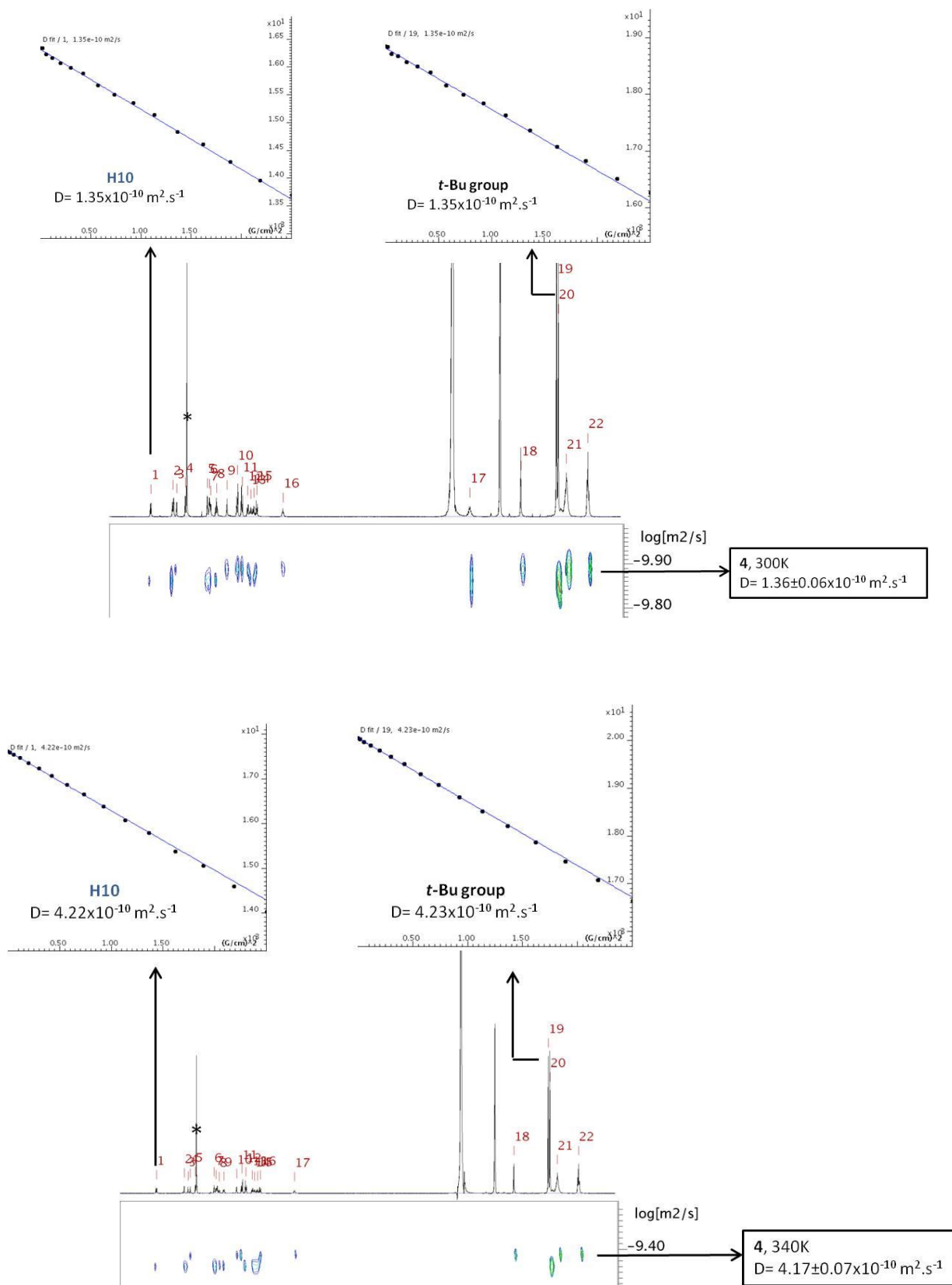


**Figure S1** FT-IR spectra of (S,S)-**3** ( $2 \times 10^{-3}$  M), cyclometallated Pt urea complex **4** ( $10^{-3}$  M) and **4** + (S,S)-**3** (1:1,  $2 \times 10^{-3}$  M) in CH<sub>2</sub>Cl<sub>2</sub>:THF (9:1 ratio). \* denotes saturation of the signal.

**Table S1** Temperature-induced chemical shift changes for **4** in DMSO- $d_6$  between 300 K and 360 K. Negative  $\Delta\delta$  and positive  $\Delta\delta$  values correspond to upfield and downfield shifted protons, respectively, upon decreasing the temperature. The aromatic C-H belonging to  $C\wedge N^{t-Bu}\wedge N^{t-Bu}$  that experience the larger shifts are underlined.

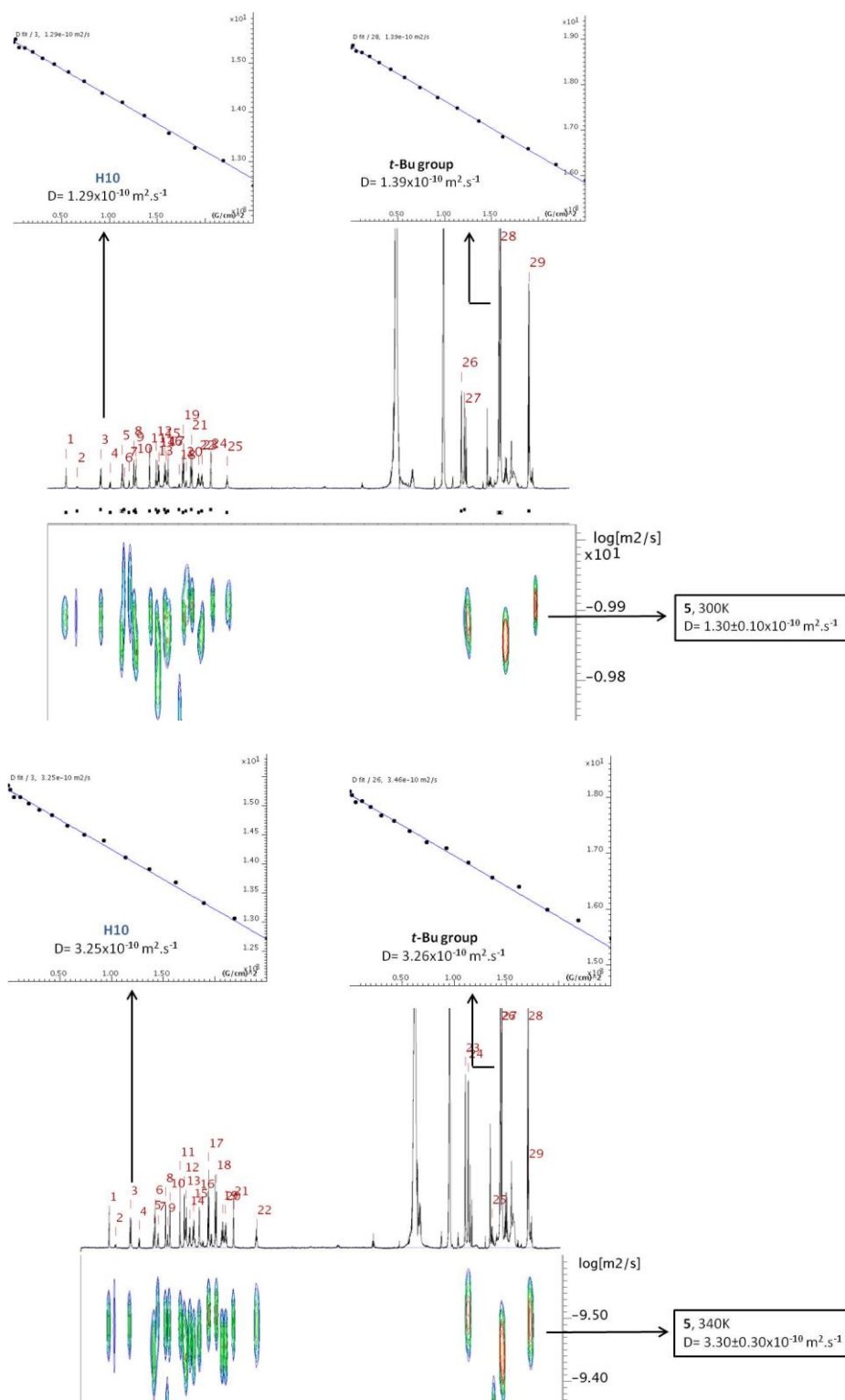


	<u>H10</u>	H6	<u>H12</u>	H7	<u>H13</u>	H14	H11
$\Delta\delta$ (ppb/K)	<b>-0.8</b>	4.2	<b>1.6</b>	2.7	<b>1.6</b>	0.6	0.3
	<b>H3</b>	<u><b>H15 or H18</b></u>	<u><b>H15 or H18</b></u>	H2	<b>H8</b>	<b>H9</b>	<b>H4</b>
$\Delta\delta$ (ppb/K)	1.1	<b>-0.8</b>	<b>0.8</b>	2.0	0.1	-0.1	0.2
	<b>H17</b>	<b>H16</b>	<b>H5</b>	H1			
$\Delta\delta$ (ppb/K)	0	0	-0.2	3.7			



**Figure S2** DOSY analyses of molecule **4** at 300 K (Top) and 340 K (Bottom) in DMSO-d<sub>6</sub>. Hydrodynamic radii according to the Stokes-Einstein equation for spherical objects ( $r_h = (k_B \times T) / (6 \times \pi \times \eta \times D)$ ):  $8.2 \pm 0.4 \text{ \AA}$  and  $5.4 \pm 0.1 \text{ \AA}$  at 300 K and 340 K, respectively.

\*= CHCl<sub>3</sub> inadvertently introduced in the sample.

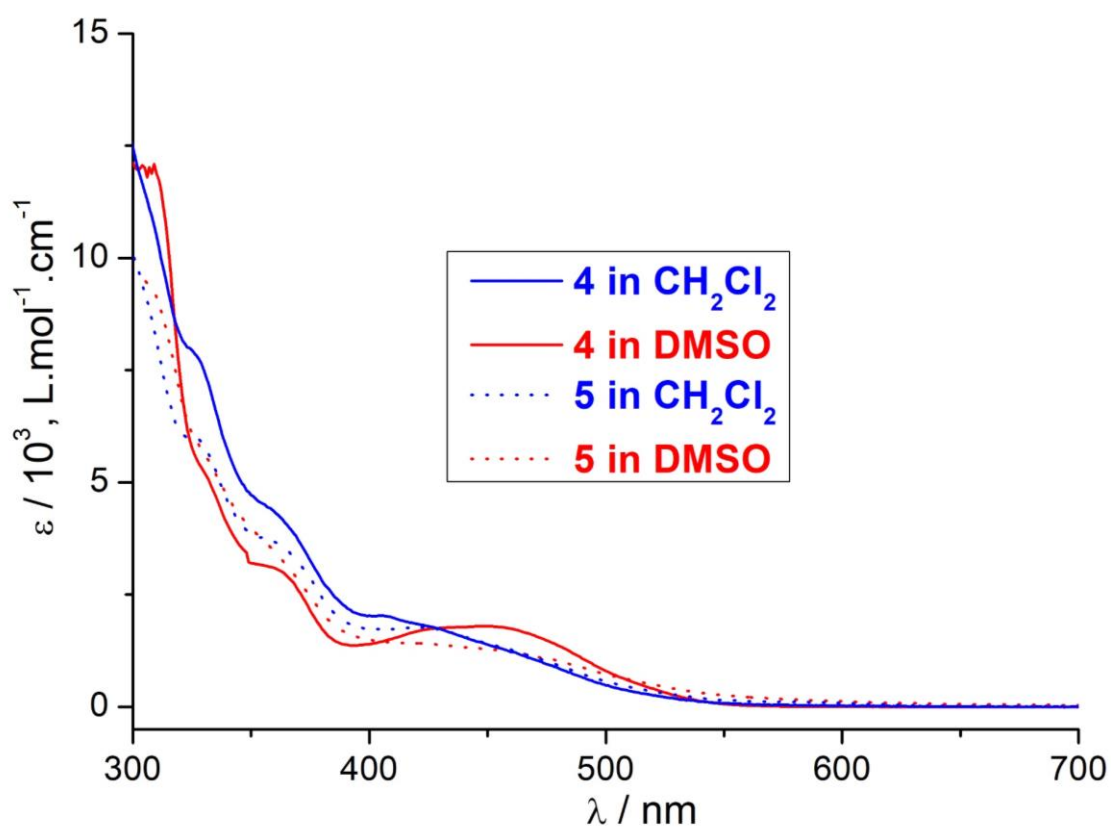


**Figure S3** DOSY analyses of molecule **5** at 300 K (Top) and 340 K (Bottom) in DMSO- $d_6$ . Hydrodynamic radii according to the Stokes-Einstein equation for spherical objects ( $r_h = (k_B \times T) / (6 \times \pi \times \eta \times D)$ ):  $8.5 \pm 0.6$  Å and  $6.8 \pm 0.5$  Å at 300 K and 340 K, respectively.

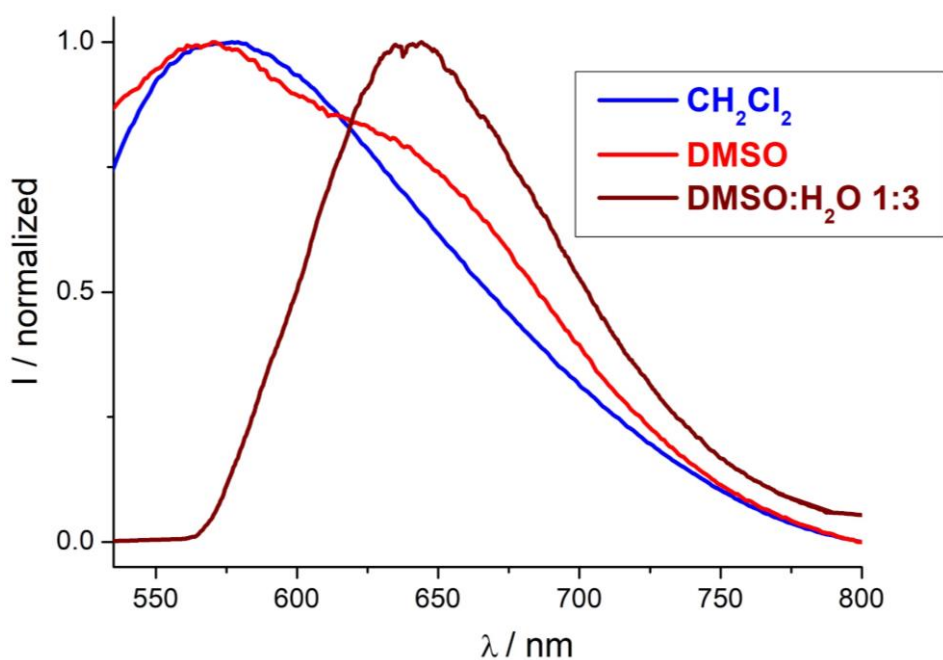
Used formula to compute the hydrodynamic radii, where  $r_{ij}$  is the distance between atoms  $i$  and  $j$ :

$$\frac{1}{R_{hyd}} = \frac{1}{N^2} \left\langle \sum_{i \neq j} \frac{1}{r_{ij}} \right\rangle$$

N: Number of atoms

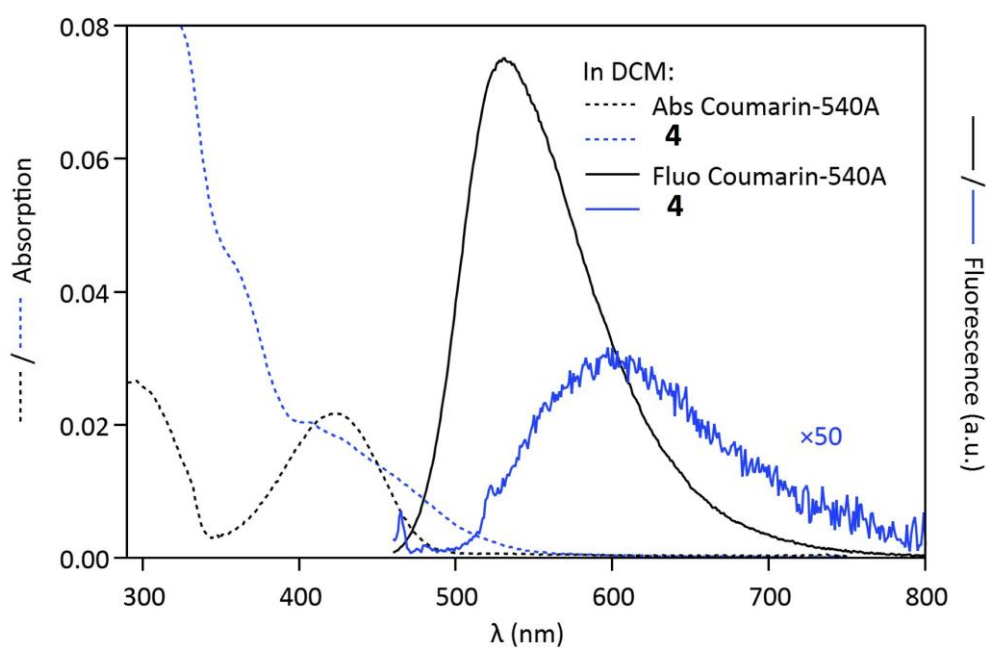


**Figure S4** UV-Vis absorption spectra of **4** and **5** in  $\text{CH}_2\text{Cl}_2$  ( $10^{-5}$  M) and in DMSO ( $10^{-4}$  M) at 293 K.

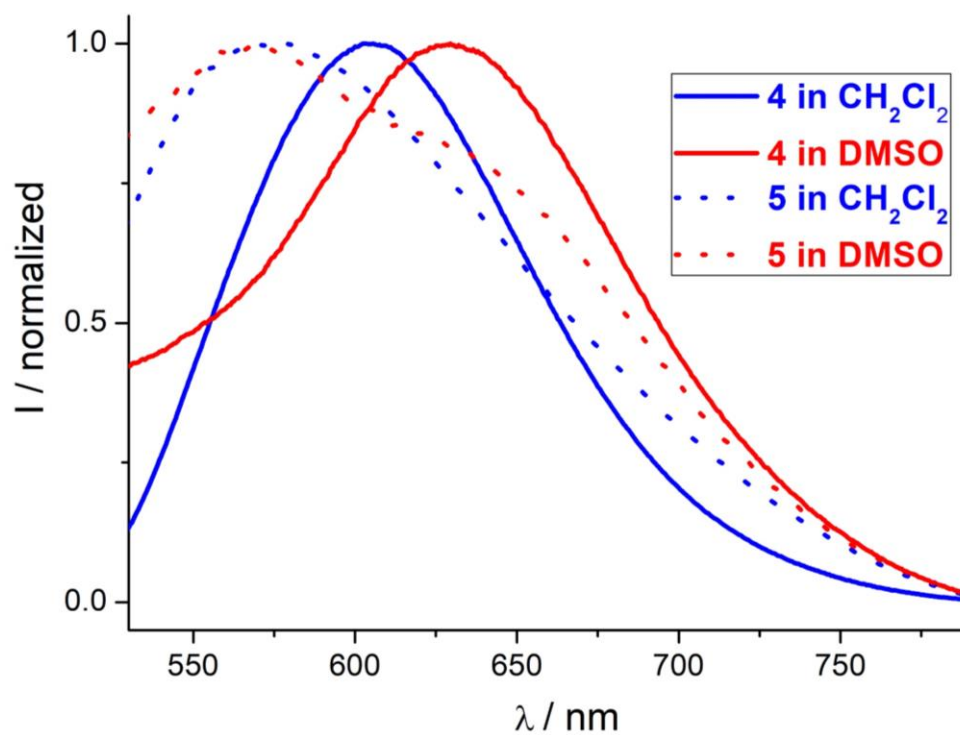


**Figure S5** Normalised emission spectra of **5** in  $\text{CH}_2\text{Cl}_2$  ( $10^{-5}$  M), in DMSO ( $10^{-4}$  M) and in DMSO:H<sub>2</sub>O 1:3 ( $10^{-3}$  M) at 293 K.  $\lambda_{\text{exc}} = 450$  nm.

a)

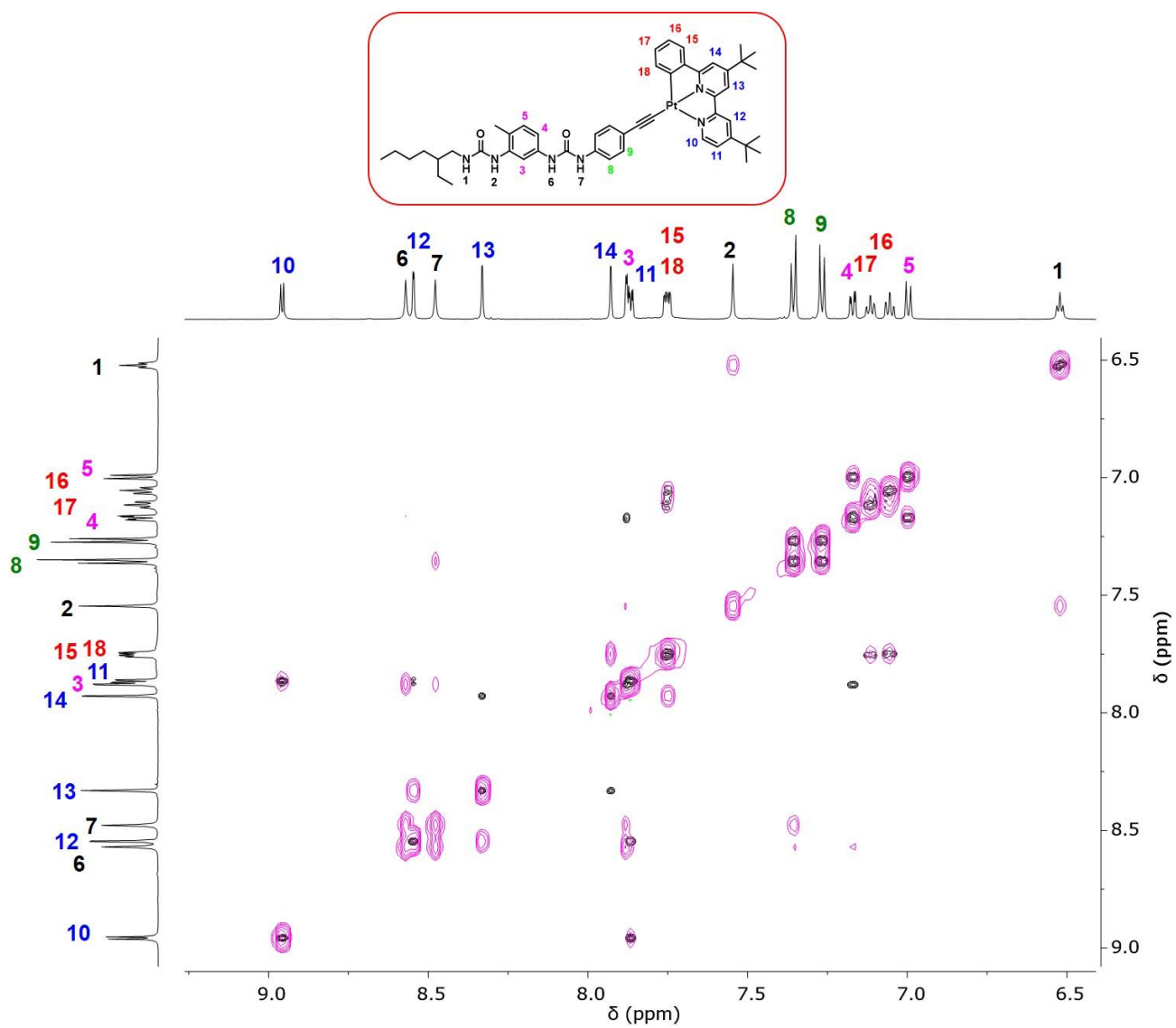


b)



**Figure S6** a) Phosphorescence of complex **4** in  $\text{CH}_2\text{Cl}_2$  with Coumarin-540A as reference for the determination of quantum yield. b) Normalised emission spectra of **4** and **5**.  $\lambda_{\text{exc}} = 450$  nm.



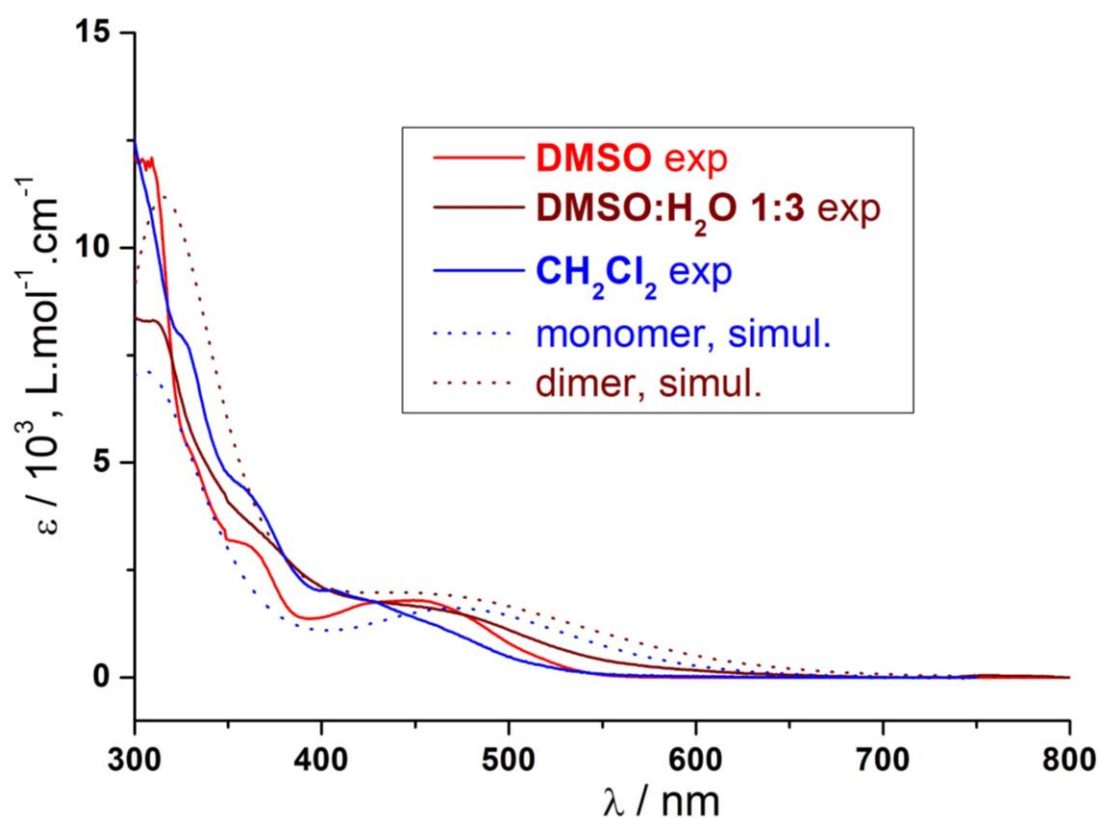


**Figure S7** Overlapped  $^1\text{H}$ - $^1\text{H}$  COSY (black contour) and  $^1\text{H}$ - $^1\text{H}$  NOESY (magenta contour) spectra of **4** in  $\text{DMSO-d}_6$  ( $6.1 \times 10^{-3}$  M).

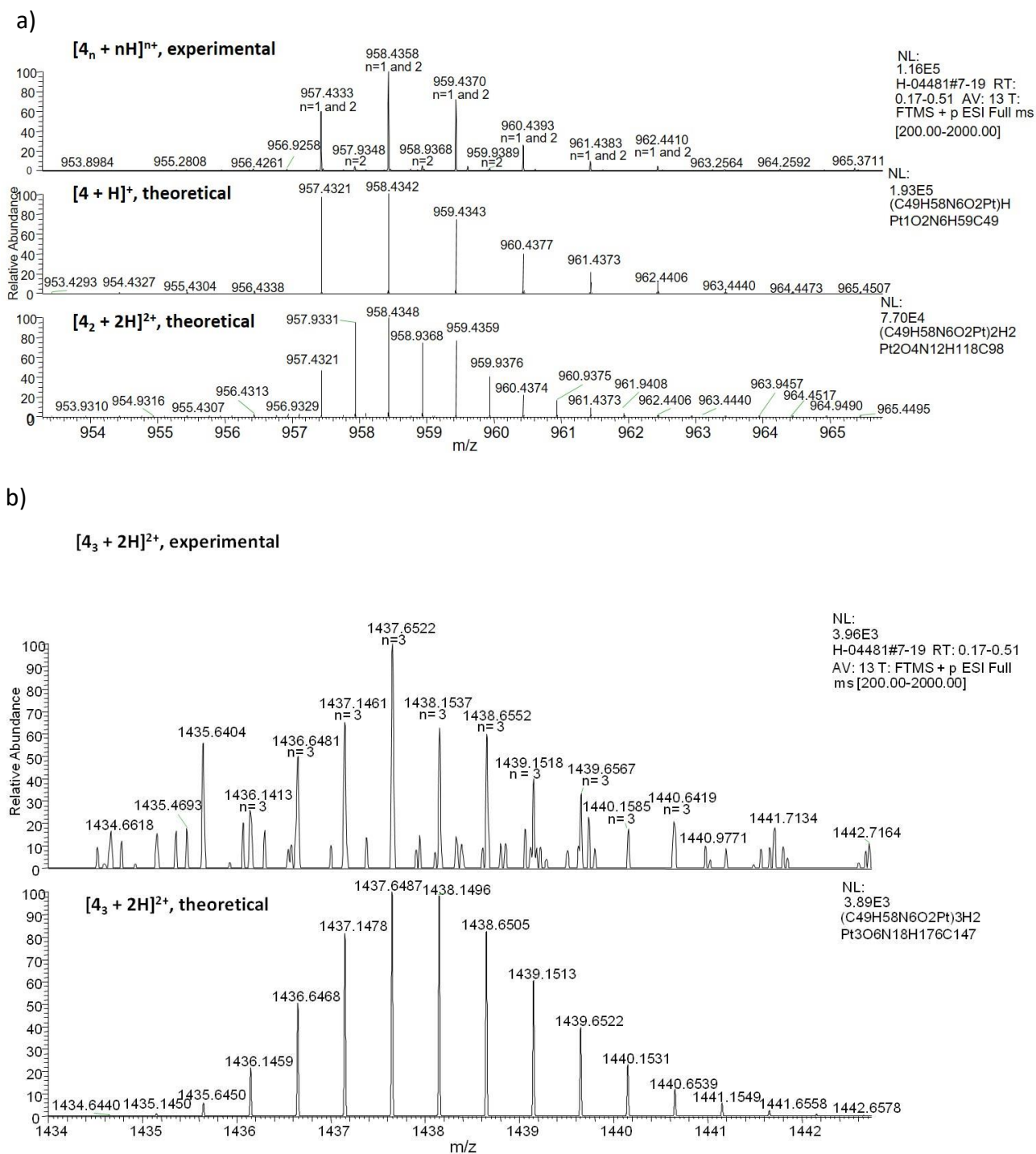
List of NOE contacts: **H6/H7**, **H6/H3**, **H12/H13**, **H7/H3**, **H7/H8**, **H14/H15**, **H2/H1**

**Table S2** Calculated wavelengths  $\lambda_{\text{calc}}$  (nm) and oscillator strengths ( $f$ ) at the PBE1PBE-GD3BJ/Lanl2dzp level in DMSO.

$\lambda_{\text{calc}}$ (nm)	$f$	Main transitions (weight)
Monomer <b>4</b>		
479	0.40	HOMO→LUMO(96%)
377	0.60	HOMO→LUMO+3(49%) HOMO-4 →LUMO(23%) HOMO-7→LUMO(19%)
Dimer <b>4<sub>2</sub></b>		
516	0.14	HOMO-1 →LUMO(81%)
446	0.17	HOMO-1→LUMO+4(60%) HOMO→LUMO+4(22%)



**Figure S8** Comparison of the experimental and simulated UV-Vis absorption spectra for complex **4**.



**Figure S9** HR-ESI analysis of molecule **4**. a) Zoom on the  $[4_n + nH]^{n+}$  species (detection of monomer and dimer). b) Zoom on the signal corresponding to the diprotonated trimer species.

## Cartesian coordinates of the optimized geometries

### Monomer 4 -DMSO

C	6.58844953	1.66467120	-0.11878990
C	7.46487363	0.50690511	0.17946212
C	7.40659761	-1.82994007	0.55181915
C	6.45452355	-2.95016316	0.61701615
C	5.07879745	-2.61299514	0.38918214
C	7.05988756	2.96329129	-0.27140091
C	8.84500875	0.53021111	0.34533213
C	8.79547770	-1.84924607	0.72405616
C	9.53748380	-0.66591597	0.62218316
C	2.89615628	-0.68302800	-0.19623391
C	4.41087438	2.37314924	-0.50845493
C	6.84633459	-4.26944626	0.88546218
C	5.88957651	-5.28695334	0.93568318
C	4.54027442	-4.97693432	0.71590516
C	4.14439438	-3.65883922	0.44719314
C	4.82490241	3.69450034	-0.67236994
C	6.18085753	4.02244837	-0.55523693
C	1.66943918	-0.61565900	-0.35130292
H	8.12576267	3.15003631	-0.16826290
H	9.38463677	1.46935319	0.25953513
H	9.28861576	-2.79312914	0.93565218
H	3.36262030	2.08896521	-0.59339193
H	7.89549865	-4.51124927	1.05748719
H	6.19266855	-6.31248042	1.14452420
H	3.79112337	-5.76900538	0.75495217
H	3.09026430	-3.43677821	0.28020913
H	4.07252034	4.44721340	-0.89043896
N	6.79562560	-0.65724199	0.28630313
N	5.26045045	1.37693117	-0.23912191
Pt	4.82126642	-0.68139900	0.04005511
C	6.71593955	5.43886149	-0.71853295
C	7.40643659	5.86348950	0.59121815
C	7.73722862	5.45631148	-1.87148004
C	5.60028447	6.43986555	-1.03416197
H	6.69720852	5.85712451	1.42931422
H	8.24426065	5.20297147	0.84805517
H	7.80316162	6.88181058	0.48474615
H	7.26831460	5.15215846	-2.81653611
H	8.13389163	6.47258056	-1.99662804
H	8.58594368	4.78768444	-1.68074002
H	6.03466046	7.44121062	-1.14645798
H	5.08396141	6.19567052	-1.97214004
H	4.85606640	6.49017756	-0.22825391
C	11.05227590	-0.63113397	0.79761417
C	11.69198896	-0.09400392	-0.49650893
C	11.39785494	0.30350611	1.97202926
C	11.63345693	-2.01825507	1.09027619

H	11.45099894	-0.74004397	-1.35101700
H	11.35414194	0.92311516	-0.73188095
H	12.78406803	-0.06630491	-0.38474292
H	10.94722691	-0.05680792	2.90620433
H	12.48702999	0.33890912	2.10771527
H	11.04898288	1.32910419	1.79856325
H	12.72141302	-1.93609306	1.20807720
H	11.23231893	-2.44428310	2.01972726
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C	-0.40015998	0.68034409	-0.76137695
C	-0.55332598	-1.70534709	-0.45246393
C	-1.78043809	0.74406009	-0.92145796
H	0.19016306	1.59429216	-0.82110895
C	-1.93832009	-1.65441609	-0.61135494
H	-0.07739894	-2.66852117	-0.26826291
C	-2.57109014	-0.41979600	-0.84867296
H	-2.25537213	1.70936716	-1.10497498
H	-2.53188813	-2.55991517	-0.55178293
N	-3.95319525	-0.26000100	-1.02117897
H	-4.24611528	0.69634108	-1.17902298
C	-4.93239032	-1.23287207	-0.99197696
O	-4.70870330	-2.42493216	-0.79110895
N	-6.18909740	-0.70965504	-1.21401799
H	-6.26137142	0.28629404	-1.38274300
C	-7.41705649	-1.39116110	-1.22808398
C	-8.55607358	-0.61417804	-1.49569100
C	-7.56166052	-2.76890320	-0.99929097
C	-9.83020867	-1.18997309	-1.50466801
H	-8.45136062	0.45319604	-1.68696802
C	-8.84390462	-3.32579025	-1.03489397
H	-6.69310544	-3.38397825	-0.79526095
C	-9.99654771	-2.57252220	-1.27338399
H	-8.94992761	-4.39588533	-0.85164696
C	-11.36535981	-3.19697625	-1.27672799
H	-12.04932484	-2.66088121	-0.60379794
H	-11.82289085	-3.18465525	-2.27804706
H	-11.31510781	-4.24281833	-0.95456796
N	-10.98886978	-0.42623904	-1.76499203
H	-11.65548986	-0.85829608	-2.39553508
C	-11.15075081	0.95209107	-1.74828003
O	-11.89263784	1.49915610	-2.57255309
N	-10.47758078	1.65869612	-0.79143895
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C	-12.52554193	1.03434506	1.40536321
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H	-12.46487488	-0.02013801	1.72108324

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H	-13.48223597	1.76044312	3.21594735
C	-15.02325711	1.04127806	1.89542925
H	-15.22591311	1.00761405	0.81386517
H	-14.95987607	-0.00574402	2.22944328
C	-16.17902219	1.73875010	2.61205131
H	-16.28136523	2.77954918	2.27388528
H	-17.13446028	1.22998506	2.42569830
H	-16.01511017	1.75728011	3.69878239
C	-10.59723474	1.42623410	3.01920634
H	-11.40901381	1.54111511	3.74995740
H	-10.27871671	0.37348603	3.08254335
C	-9.44403967	2.34465218	3.42709937
H	-8.56903964	2.23215417	2.77372832
H	-9.75347273	3.39847326	3.39093237
H	-9.11598068	2.12748916	4.45225345

#### Dimer 4<sub>2</sub>-DMSO

C	0.90248707	4.12869532	-1.28173510
C	2.09806416	3.27411525	-1.47398411
C	2.74256621	1.02830008	-1.85391214
C	2.15756616	-0.31025502	-2.02613716
C	0.72928405	-0.38529103	-1.92440015
C	0.95512707	5.51376240	-1.17996109
C	3.42384226	3.69044828	-1.49615011
C	4.08947331	1.40559711	-1.88086814
C	4.44977234	2.74698621	-1.70182213
C	-1.95512015	0.84054306	-1.56152612
C	-1.40969111	4.16650332	-1.06742708
C	2.92989823	-1.45481211	-2.25912217
C	2.31277918	-2.70314021	-2.38562818
C	0.91910307	-2.79845322	-2.27469318
C	0.14147601	-1.65281913	-2.04891616
C	-1.41842411	5.55535245	-0.95045407
C	-0.21911402	6.27171546	-1.02846308
C	-3.16208424	0.56790404	-1.57022812
H	1.92306115	6.00582046	-1.22962910
H	3.66109328	4.73947136	-1.34158810
H	4.84841237	0.64446205	-2.03392116
H	-2.33001018	3.58476027	-1.02943508
H	4.01531531	-1.38402611	-2.32936918
H	2.91514522	-3.59439627	-2.56069420
H	0.43441303	-3.77185929	-2.36550818
H	-0.94165907	-1.74418014	-1.96274715
H	-2.37462318	6.05260946	-0.81521506
N	1.80407314	1.97304215	-1.64908313
N	-0.28878702	3.46499426	-1.24742809
Pt	-0.09474001	1.37971010	-1.57299012
C	-0.16735701	7.79526661	-1.02514608
C	0.85508507	8.30665763	0.00265500
C	0.25946102	8.24755864	-2.43610419

C	-1.53157712	8.41254764	-0.69805106
H	0.54898904	8.04927164	1.02333708
H	1.85856614	7.89708862	-0.16306001
H	0.92462907	9.40022570	-0.06703001
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H	0.28966802	9.34459673	-2.48031519
H	1.25661809	7.86933560	-2.69591421
H	-1.43739211	9.50558171	-0.67137005
H	-2.28854717	8.16705665	-1.45435311
H	-1.90125215	8.08639861	0.28414002
C	5.90262047	3.21262125	-1.71391413
C	6.07777544	4.29364533	-2.79644722
C	6.24121649	3.80516329	-0.33336903
C	6.87653855	2.06729516	-2.00891315
H	5.83048244	3.89628230	-3.78991129
H	5.44404044	5.17027939	-2.61329520
H	7.12174854	4.63411035	-2.81382122
H	6.12084047	3.05140023	0.45568103
H	7.28397455	4.14974832	-0.32159002
H	5.60004142	4.66090736	-0.08638501
H	7.90276362	2.45683119	-2.01364515
H	6.82718652	1.27903710	-1.24738909
H	6.68835851	1.61651712	-2.99289323
C	-4.55947035	0.27728402	-1.55376212
C	-5.44463243	1.03826408	-0.76131006
C	-5.11463739	-0.76479406	-2.32230418
C	-6.80547851	0.75527206	-0.72234505
H	-5.04560938	1.86401014	-0.17559001
C	-6.47863451	-1.05422008	-2.29980618
H	-4.45693134	-1.36660610	-2.94937822
C	-7.34287155	-0.29990402	-1.48529111
H	-7.46288355	1.35787110	-0.09282301
H	-6.87640255	-1.86653514	-2.89767222
N	-8.72517667	-0.51803804	-1.38518710
H	-9.20938269	0.13486501	-0.78116406
C	-9.47004074	-1.51981812	-1.97324715
O	-8.99227968	-2.40521618	-2.67996521
N	-10.81294284	-1.41276811	-1.67412013
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C	-11.85862989	-2.24695617	-2.10191516
C	-13.15987400	-1.84570914	-1.75897314
C	-11.67679189	-3.43088226	-2.83579622
C	-14.26591409	-2.62364220	-2.11460516
H	-13.31659000	-0.92489607	-1.19751109
C	-12.79969098	-4.18103332	-3.19756824
H	-10.68005479	-3.75339129	-3.11306624
C	-14.10382006	-3.81664029	-2.84922622
H	-12.64868094	-5.10210739	-3.76219529
C	-15.29140715	-4.66048236	-3.22416225
H	-15.92240123	-4.86646837	-2.34830118
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H	-14.96875212	-5.61649444	-3.65108628

N	-15.57907521	-2.22841817	-1.77750914
H	-16.25460823	-2.26064617	-2.53312719
C	-16.01298124	-1.40281911	-0.75393606
O	-17.00483731	-0.68194205	-0.91810807
N	-15.31694715	-1.42194311	0.42514503
H	-15.75111518	-0.79746206	1.09687708
C	-14.65882110	-2.58037820	1.02464608
H	-13.82015708	-2.20298217	1.62345913
H	-14.22418708	-3.19917124	0.23443702
C	-15.58338118	-3.44651526	1.89318114
H	-16.11627822	-2.78110021	2.59393220
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C	-18.79116841	-5.48362739	0.87851607
H	-19.16758944	-4.75968936	0.13927601
H	-18.27177941	-6.26655048	0.30460202
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H	-20.52051955	-5.33013541	2.19511917
H	-20.67235257	-6.59159448	0.95535707
H	-19.61980648	-6.84920850	2.36136718
C	-14.73775313	-4.42978634	2.71725721
H	-15.39424720	-5.21030840	3.12395324
H	-14.03545306	-4.94544938	2.04283715
C	-13.97133307	-3.79007329	3.87631430
H	-13.24461099	-3.04083723	3.53523527
H	-14.66158611	-3.29140325	4.57102135
H	-13.41595202	-4.54873235	4.44357134
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C	-2.37381718	1.71249313	2.05543415
C	-1.04124708	3.66297028	2.19688417
C	0.36518203	4.08792131	2.17083417
C	1.32898410	3.04389923	1.98257915
C	-3.37898426	-0.60291605	1.92283115
C	-3.53712527	2.46063619	2.20689617
C	-2.18789417	4.45175434	2.33708518
C	-3.45690226	3.86185729	2.33996818
C	2.14217216	0.27722602	1.60243812
C	-0.85230806	-1.56262912	1.58806512
C	0.75950306	5.42429440	2.31359217
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C	3.06845623	4.75869036	2.06244016
C	2.67927321	3.41986826	1.92898115
C	-1.91847314	-2.46117319	1.60781712
C	-3.22385124	-1.99397615	1.79857414
C	3.22219425	-0.29025902	1.39806611
H	-4.36951833	-0.17284301	2.04950816
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H	-2.07847616	5.52735241	2.43582219

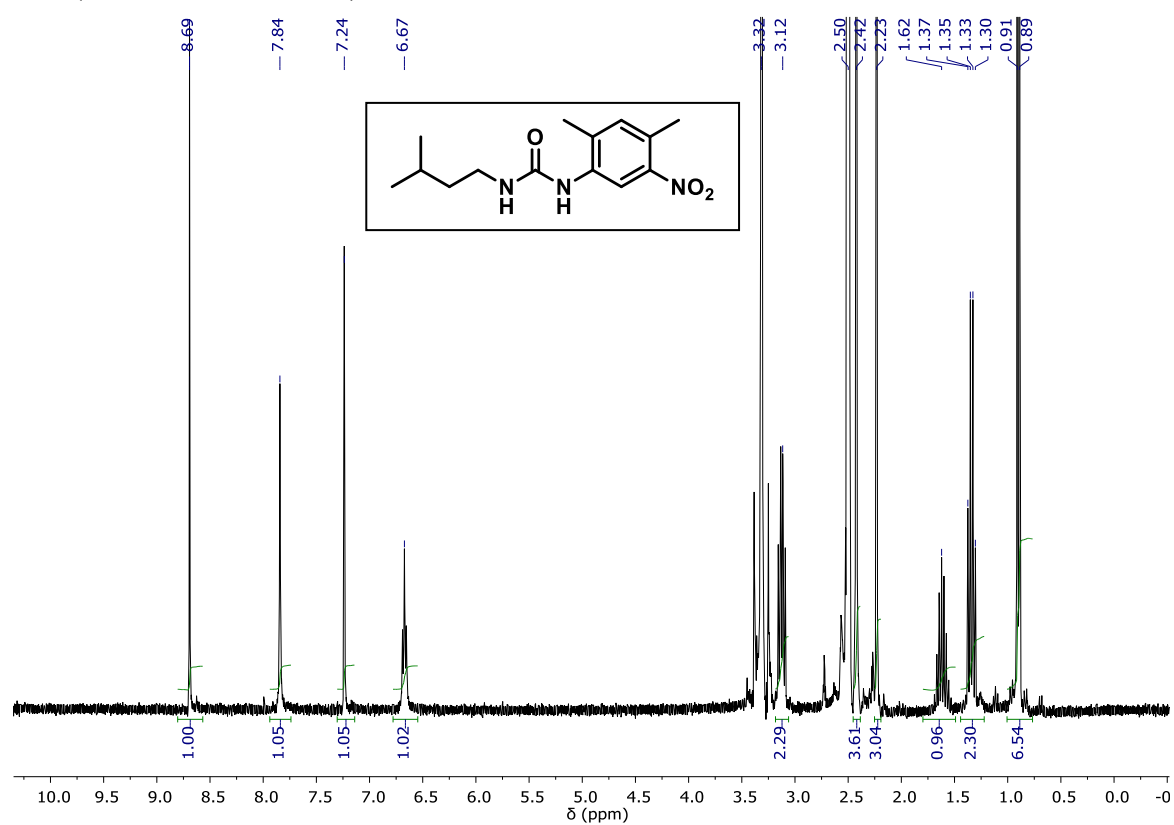


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H	-1.70305213	-3.51924627	1.48688811
N	-1.17757709	2.32966018	2.05139616
N	-1.01260608	-0.24666102	1.74893913
Pt	0.48770804	1.26178310	1.81556614
C	-4.43426834	-2.91390122	1.89337215
C	-5.13576739	-2.66998921	3.24277725
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H	-4.93521938	-2.79306221	-0.22990402
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H	-5.71911143	-1.54299212	0.74486806
H	-4.94033338	-5.01196238	1.89609414
H	-3.56664227	-4.63340935	0.84565406
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C	-5.64319042	4.42088934	1.26866010
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C	4.69281636	-2.10371016	0.54925204
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C	5.95489143	-2.56057119	0.17932201
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C	6.93359153	-0.46006804	0.88455007
H	5.55469241	0.98754707	1.66643813
C	7.09031456	-1.74300713	0.32949103
H	6.06149848	-3.56173827	-0.24180402
H	7.79853761	0.18083201	1.01908108
N	8.33234765	-2.27666718	-0.05034100
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C	9.48257875	-1.56966812	-0.34327203
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N	10.53320678	-2.40157018	-0.67027705
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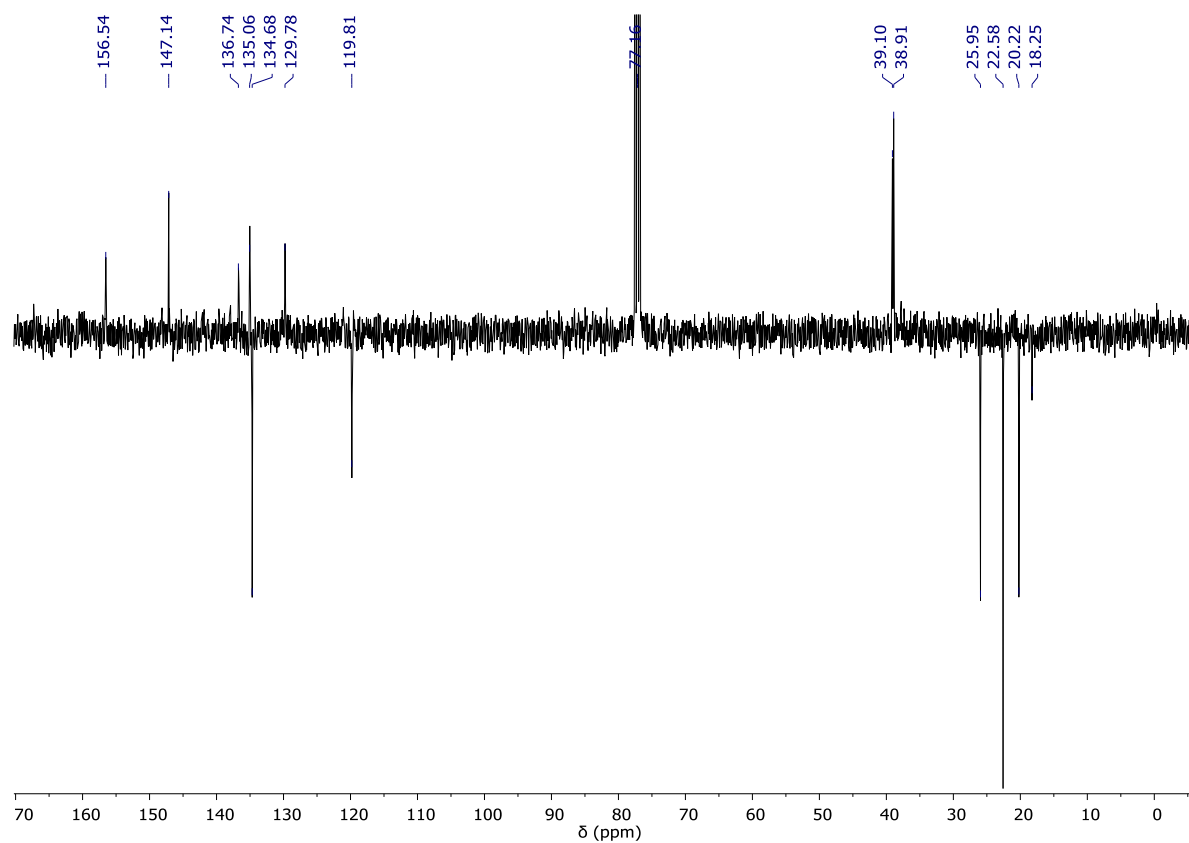
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C	13.52619705	-0.47911104	-1.79665314
H	11.51171690	0.10009001	-1.25844609
C	14.47707909	-1.49462211	-1.93652615
H	13.82416507	0.54782904	-2.01232515
C	15.89042924	-1.20609109	-2.36318118
H	16.61332325	-1.65391813	-1.66696313
H	16.11155421	-1.61343012	-3.36141226
H	16.07148723	-0.12633601	-2.40281919
N	15.00694216	-3.85000829	-1.80694514
H	15.53466221	-3.83189529	-2.67279520
C	15.02320515	-5.11321939	-1.23878110
O	15.47335819	-6.07357044	-1.87540814
N	14.53693613	-5.24824540	0.03382400
H	14.63074513	-6.20964350	0.34483903
C	14.63743610	-4.25828832	1.10383109
H	13.76571506	-4.39746634	1.75479313
H	14.55317012	-3.25504325	0.67600105
C	15.93235823	-4.35252033	1.92372415
H	15.99824323	-5.37329039	2.33905118
C	17.15814933	-4.11593431	1.03258808
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H	17.10482133	-3.09711924	0.61427005
C	18.50893744	-4.31597133	1.71974813
H	18.51791842	-5.29178043	2.23191517
H	18.66108844	-3.55573127	2.49964519
C	19.67817849	-4.25100933	0.73679706
H	19.54736947	-5.02695138	-0.03308800
H	19.65155352	-3.28573525	0.20766902
C	21.03613161	-4.42534534	1.41581511
H	21.09808260	-5.39510943	1.92956615
H	21.85878765	-4.37764734	0.68956205
H	21.20530963	-3.64052328	2.16648016
C	15.88411721	-3.36710026	3.10037924
H	16.83481129	-3.43255326	3.64529528
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C	14.74423915	-3.59961927	4.09493131
H	13.75954205	-3.37526026	3.66589228
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## NMR Spectra

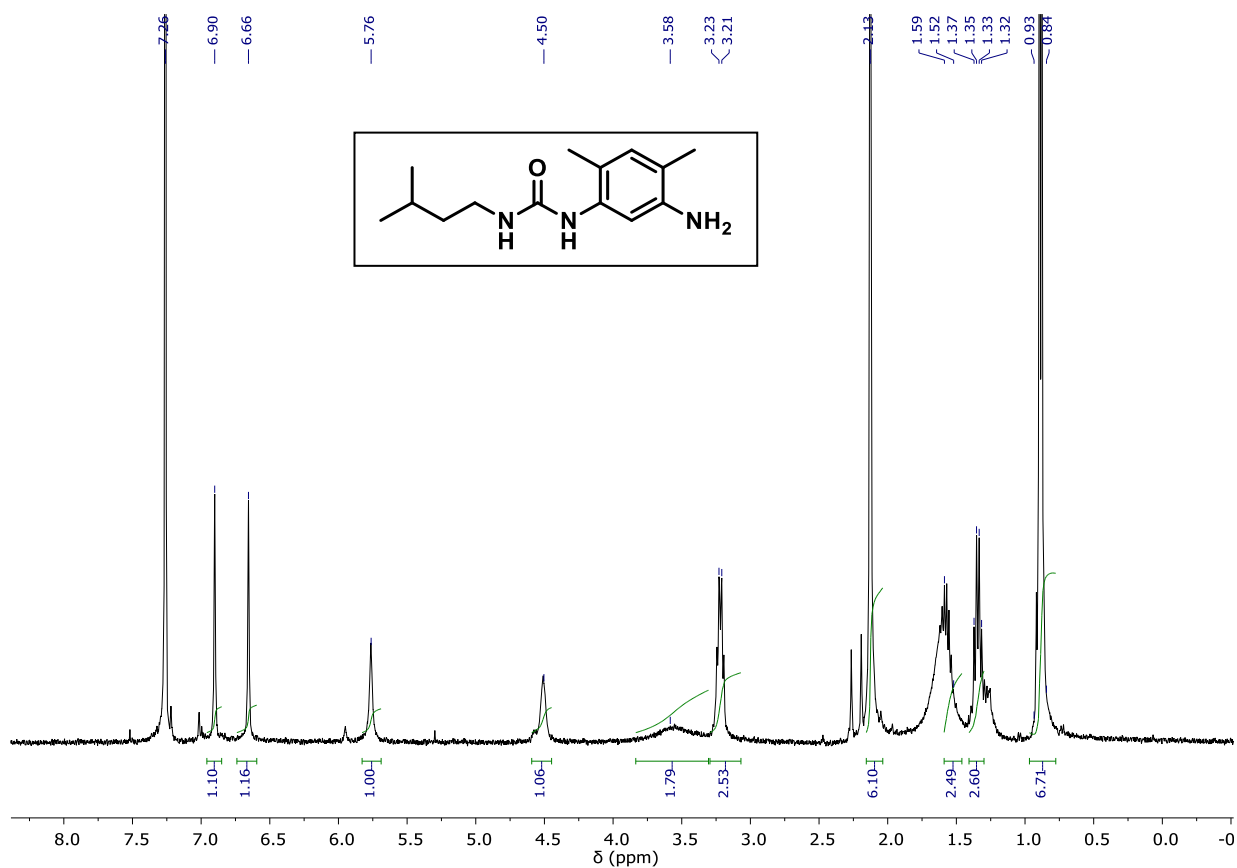
$^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ ) of *i*PentUXN.



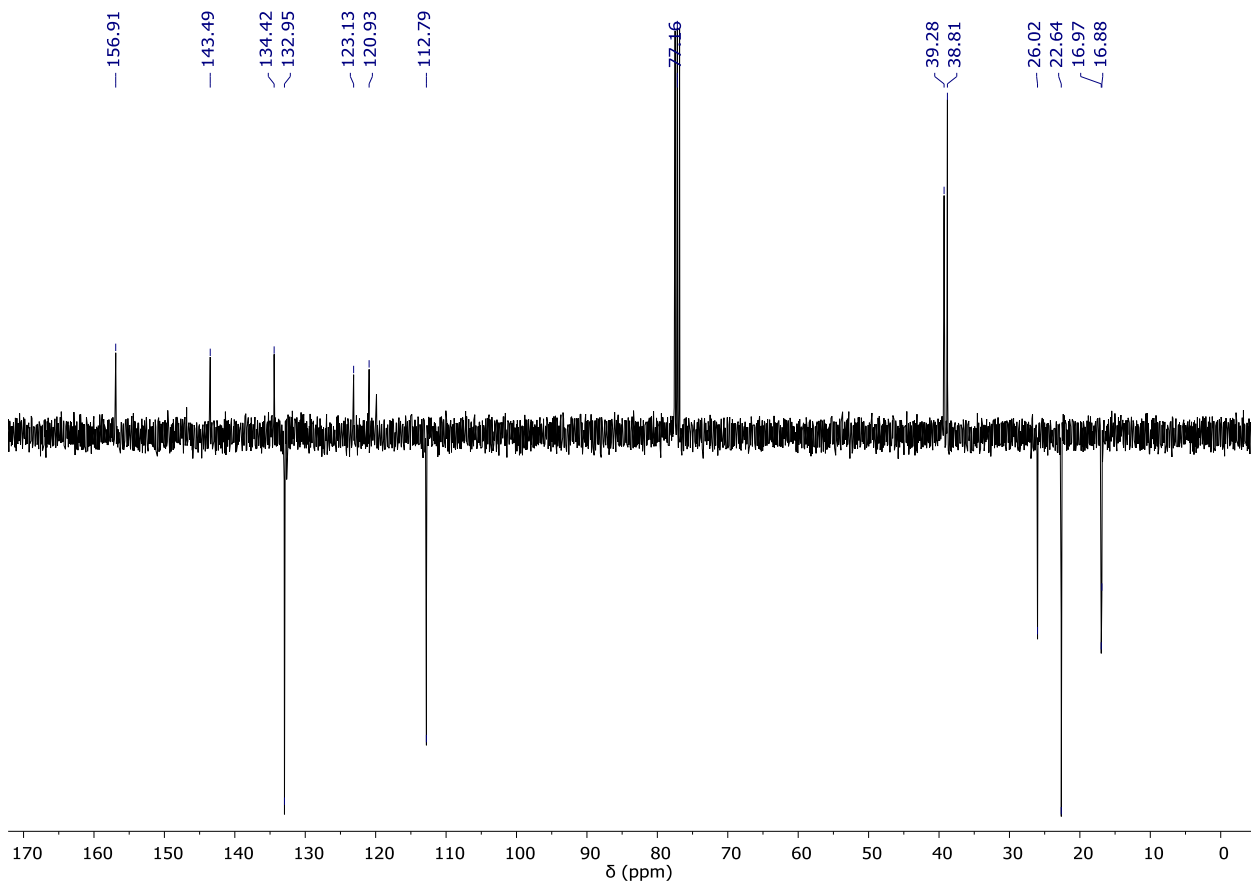
$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ , JMOD) of *i*PentUXN.



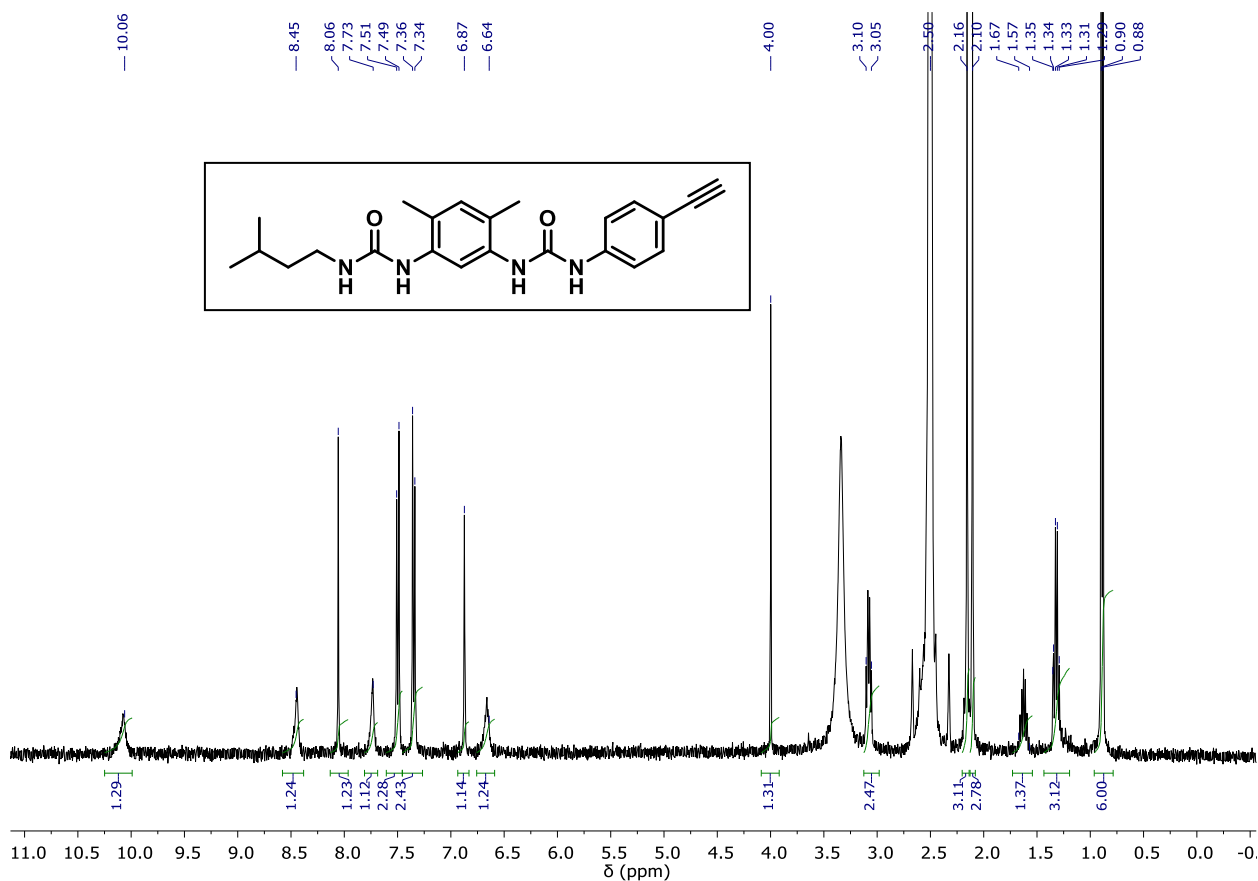
$^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ) of *i*PentUXA.



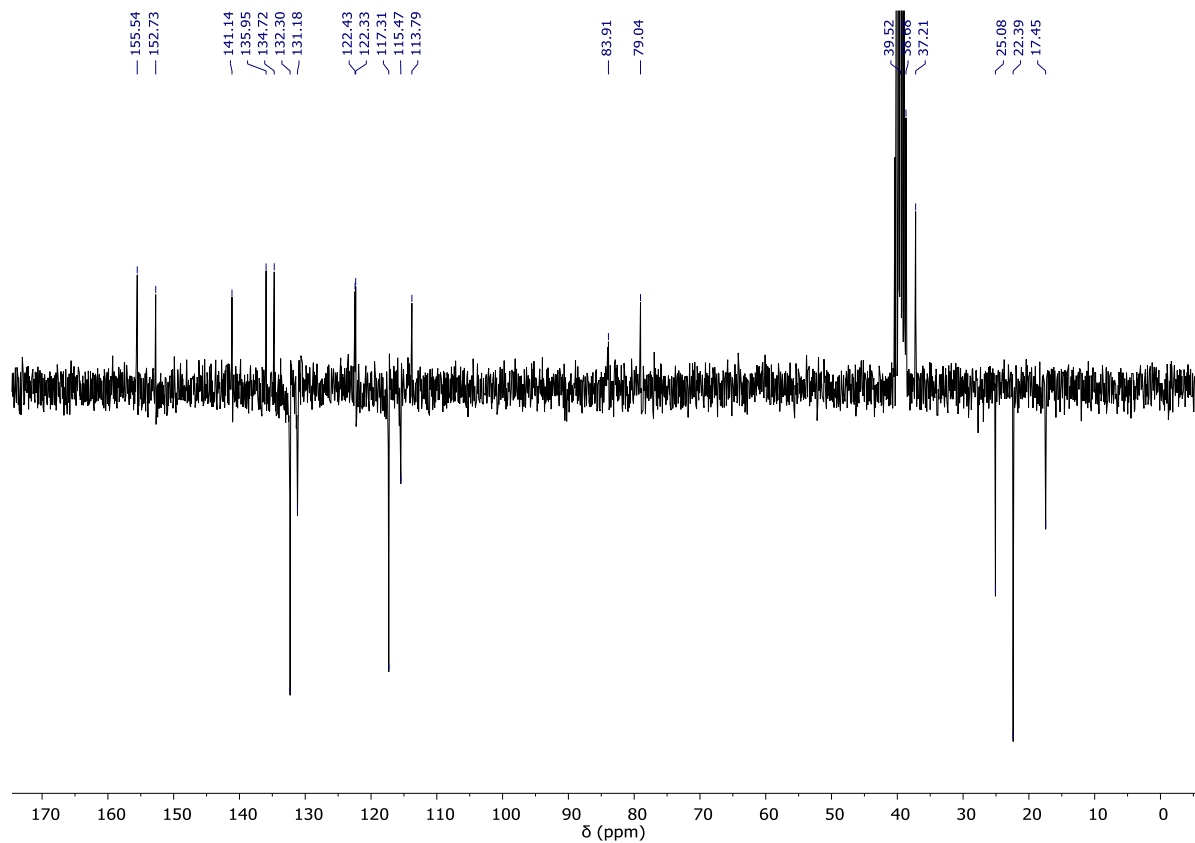
$^{13}\text{C}\{^1\text{H}\}$  NMR (101 MHz,  $\text{CDCl}_3$ , JMOD) of *i*PentUXA.



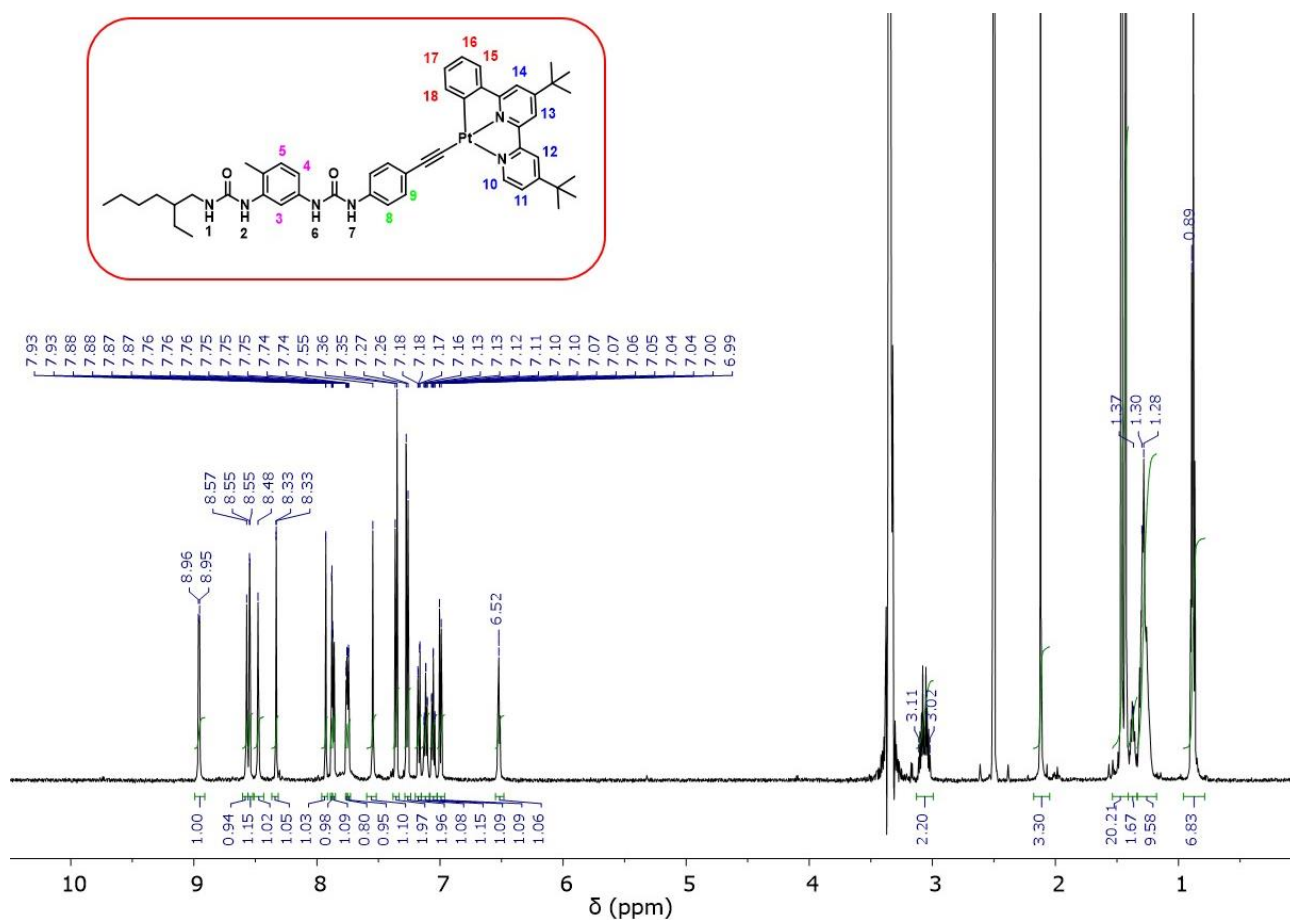
$^1\text{H}$  NMR (400 MHz, DMSO- $d_6$ ) of *i*PenXUE.



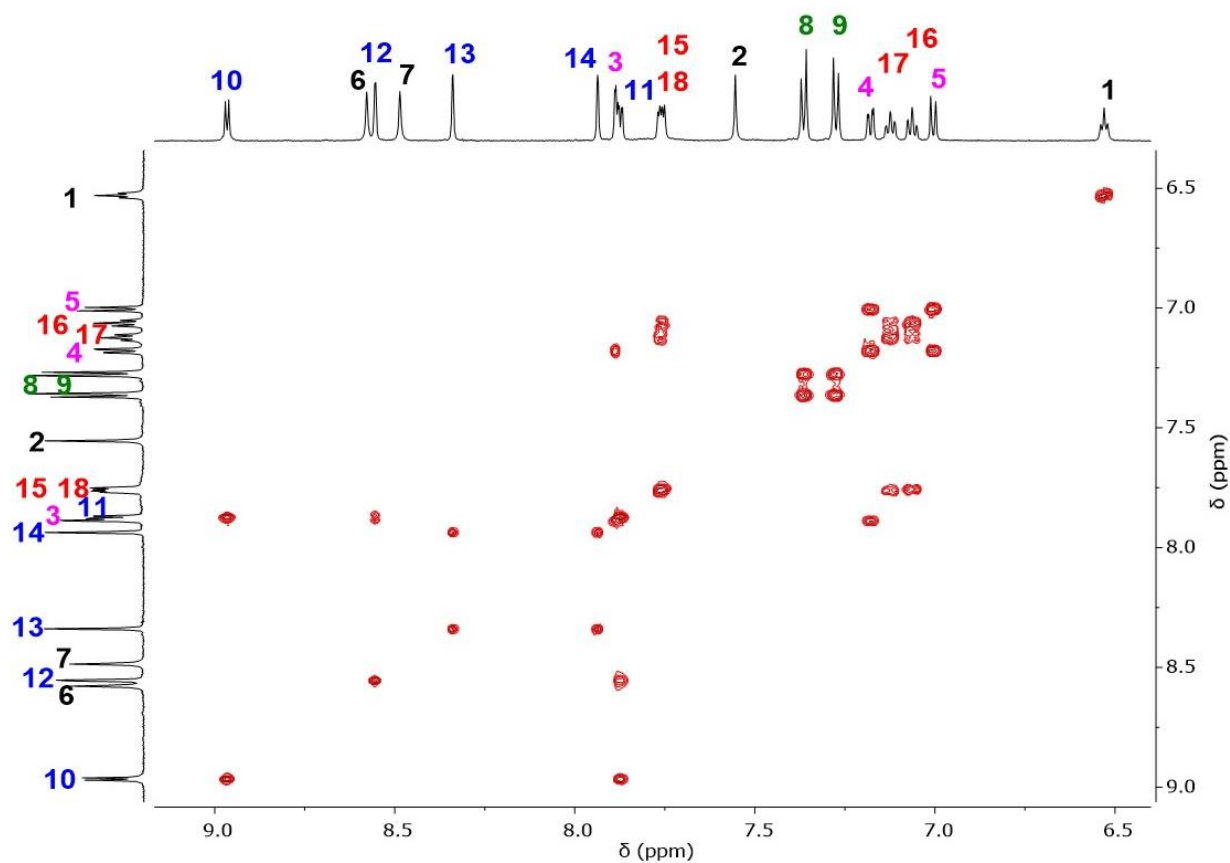
$^{13}\text{C}$  { $^1\text{H}$ } NMR (100 MHz, DMSO- $d_6$ , JMOD) of *i*PenXUE.



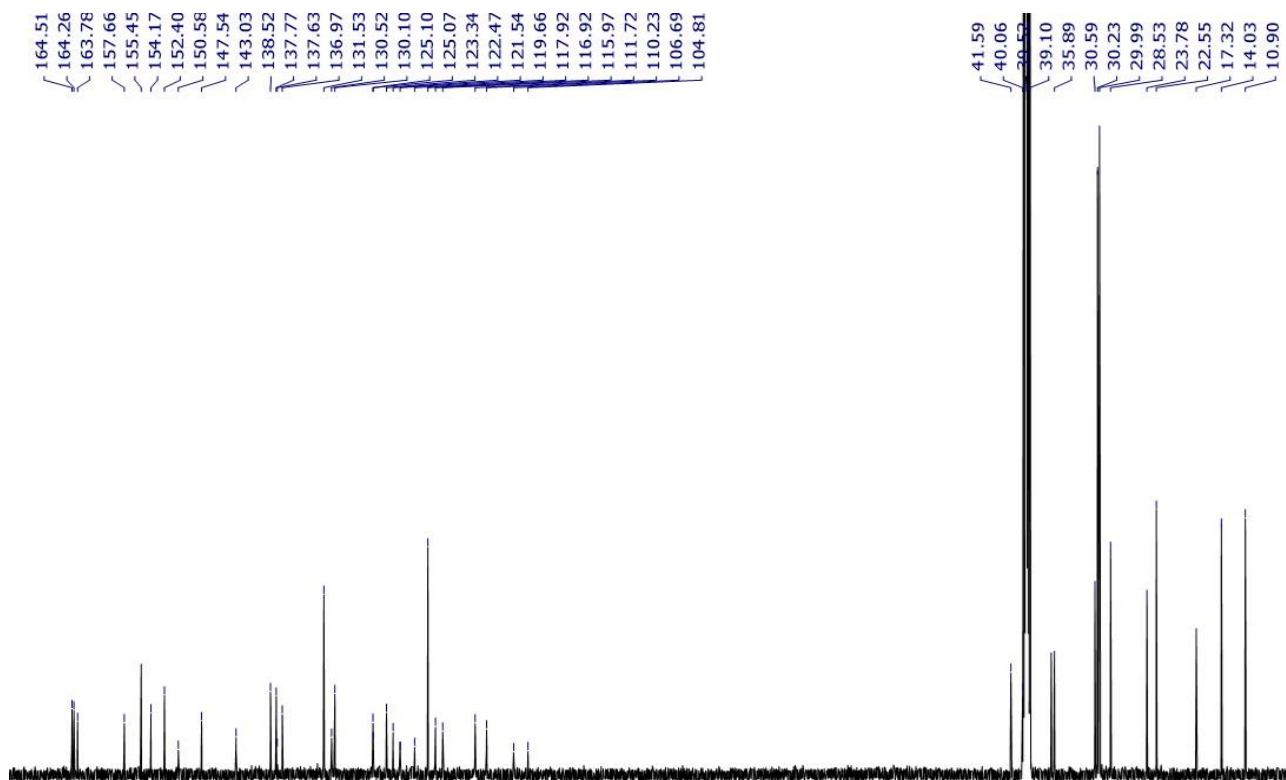
$^1\text{H}$  NMR (600 MHz,  $\text{DMSO-d}_6$ ) of compound 4. The signal present at  $\delta = 3.33$  ppm corresponds to water.



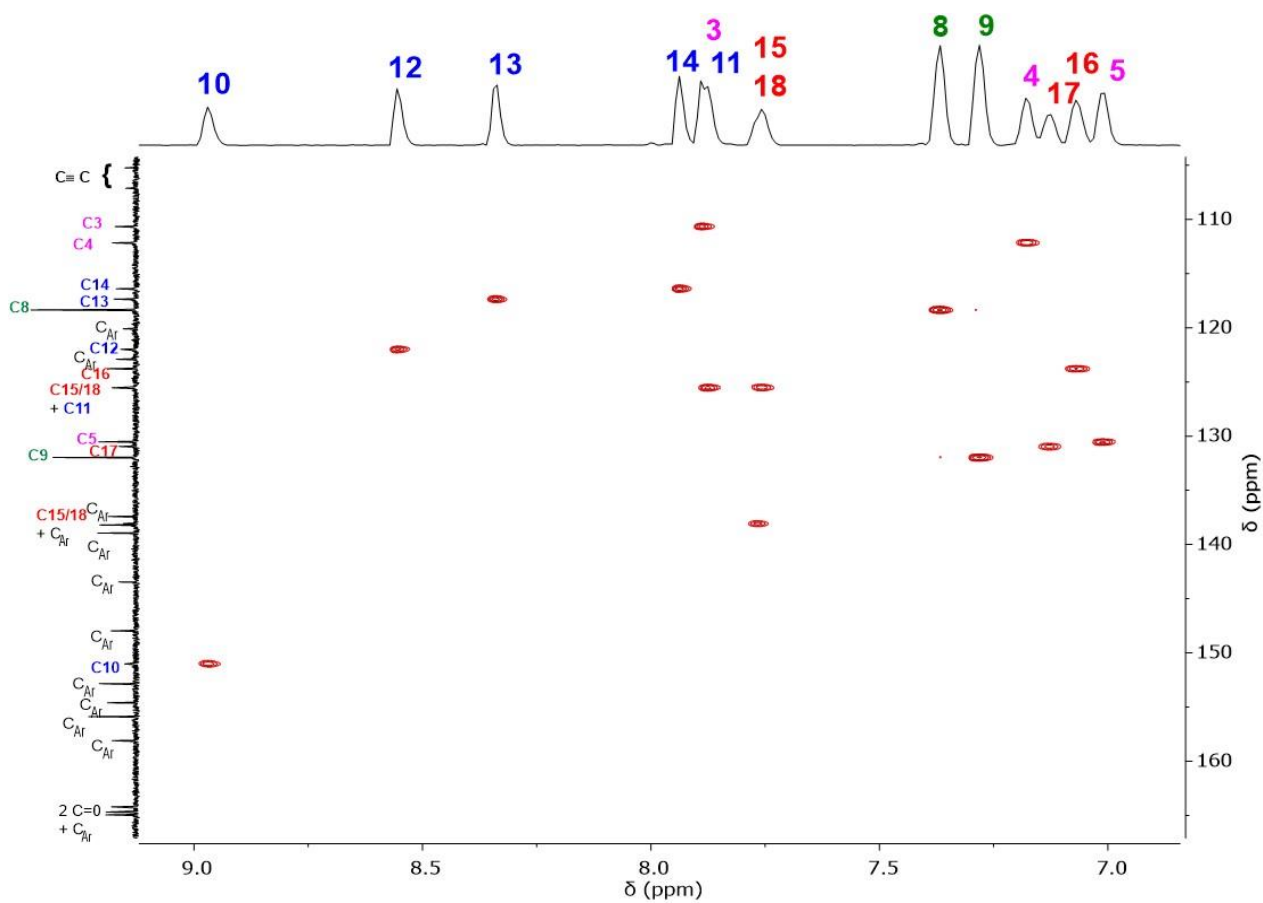
$^1\text{H}$ - $^1\text{H}$  COSY NMR (600 MHz,  $\text{DMSO-d}_6$ ) of compound 4. Region:  $\delta = 6.3$ – $9.2$  ppm.



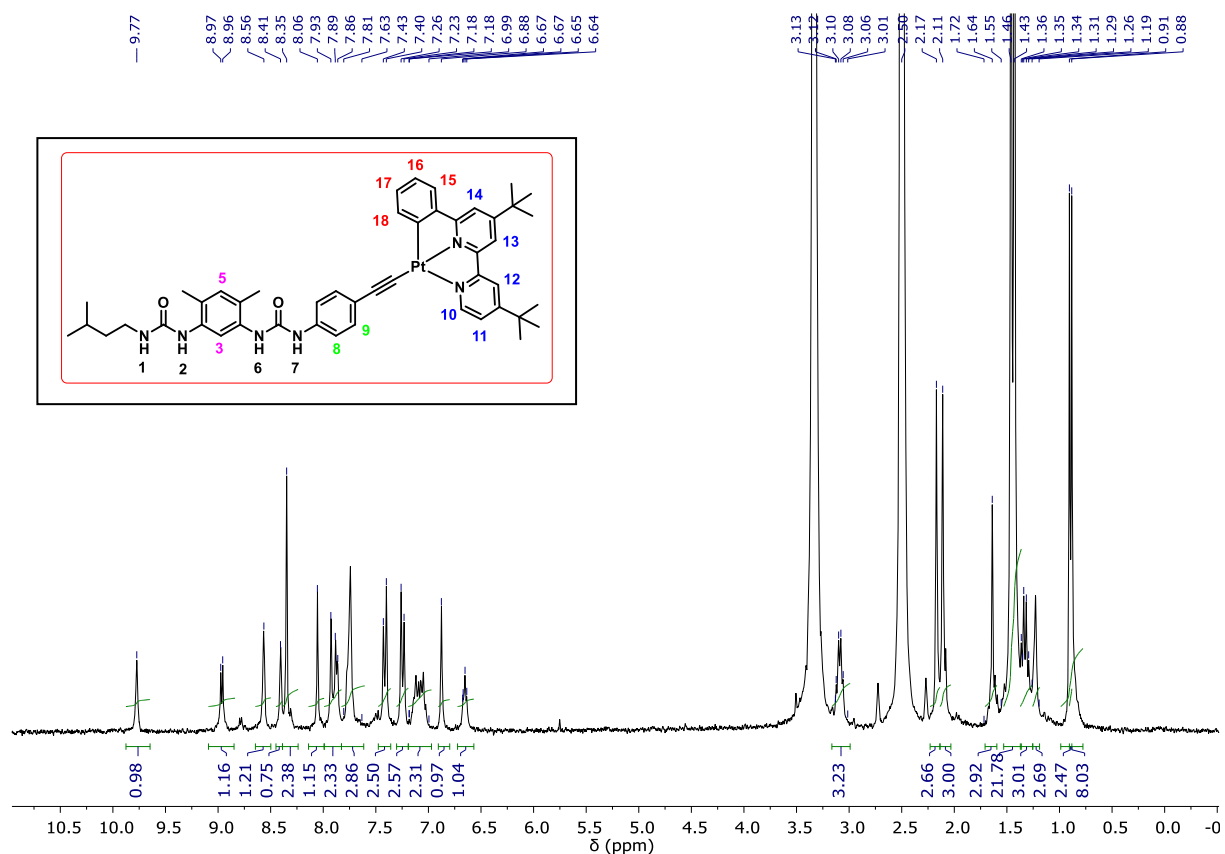
$^{13}\text{C}\{^1\text{H}\}$  NMR (202 MHz, DMSO- $d_6$ ) of compound 4.



$^1\text{H}-^{13}\text{C}$  HSQC NMR (DMSO- $d_6$ ) of compound 4.



$^1\text{H}$  NMR (300 MHz,  $\text{DMSO-d}_6$ ) of compound **5**. The signal present at  $\delta = 3.33$  ppm corresponds to water.



$^1\text{H}$ - $^1\text{H}$  COSY NMR (600 MHz,  $\text{DMSO-d}_6$ ) of compound **5**. Region:  $\delta = 6.2$ – $10.2$  ppm.

