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Table S1. Selected bond distances and angles for **3**·4MeOH

Dy1—O1	2.284 (6)	Dy5—O17	2.216 (8)
Dy1—O2	2.295 (7)	Dy5—O31	2.287 (9)
Dy1—O13	2.384 (6)	Dy5—O22	2.290 (7)
Dy1—O9	2.394 (7)	Dy5—O20	2.300 (7)
Dy1—O3	2.442 (7)	Dy5—O1	2.326 (7)
Dy1—O21	2.466 (6)	Dy5—O15	2.381 (7)
Dy1—O6	2.478 (6)	Dy5—N4	2.496 (9)
Dy1—O15	2.491 (7)	Dy6—O5	2.191 (8)
Dy2—O23	2.211 (7)	Dy6—O18	2.321 (8)
Dy2—O10	2.337 (7)	Dy6—O20	2.338 (7)
Dy2—O24	2.346 (8)	Dy6—O12	2.351 (8)
Dy2—O26	2.374 (7)	Dy6—O3	2.396 (6)
Dy2—O21	2.405 (6)	Dy6—O1	2.412 (7)
Dy2—O2	2.417 (6)	Dy6—N1	2.473 (8)
Dy2—N6	2.475 (8)	Dy6—N5	2.509 (10)
Dy2—N7	2.482 (11)	Dy7—O29	2.151 (10)
Dy3—O11	2.225 (8)	Dy7—O25	2.267 (10)
Dy3—O14	2.274 (9)	Dy7—O19	2.283 (10)
Dy3—O7	2.351 (7)	Dy7—O28	2.330 (8)
Dy3—O16	2.359 (7)	Dy7—O30	2.343 (9)
Dy3—O9	2.408 (7)	Dy7—N10	2.464 (14)
Dy3—O13	2.444 (7)	Dy7—N8	2.492 (12)
Dy3—N9	2.482 (10)		
Dy3—N3	2.514 (9)		
Dy4—O8	2.192 (8)		
Dy4—O26	2.305 (7)		
Dy4—O27	2.326 (8)		
Dy4—O4	2.329 (7)		
Dy4—O2	2.342 (7)		
Dy4—O6	2.381 (7)		
Dy4—N2	2.483 (10)		
O1—Dy1—O2	91.7 (2)	O26—Dy2—O21	132.4 (2)
O1—Dy1—O13	112.1 (2)	O23—Dy2—O2	149.5 (3)
O2—Dy1—O13	139.6 (2)	O10—Dy2—O2	78.7 (2)
O1—Dy1—O9	138.1 (2)	O24—Dy2—O2	76.5 (2)

O2—Dy1—O9	112.3 (2)	O26—Dy2—O2	69.2 (2)
O13—Dy1—O9	71.5 (2)	O21—Dy2—O2	69.7 (2)
O1—Dy1—O3	71.0 (2)	O23—Dy2—N6	71.3 (3)
O2—Dy1—O3	81.4 (2)	O10—Dy2—N6	99.4 (3)
O13—Dy1—O3	76.6 (2)	O24—Dy2—N6	80.3 (3)
O9—Dy1—O3	143.5 (2)	O26—Dy2—N6	156.6 (3)
O1—Dy1—O21	80.4 (2)	O21—Dy2—N6	65.8 (2)
O2—Dy1—O21	70.6 (2)	O2—Dy2—N6	133.5 (2)
O13—Dy1—O21	142.8 (2)	O23—Dy2—N7	83.7 (3)
O9—Dy1—O21	76.3 (2)	O10—Dy2—N7	145.3 (3)
O3—Dy1—O21	139.1 (2)	O24—Dy2—N7	65.8 (3)
O1—Dy1—O6	145.2 (2)	O26—Dy2—N7	70.9 (3)
O2—Dy1—O6	68.7 (2)	O21—Dy2—N7	141.7 (3)
O13—Dy1—O6	73.6 (2)	O2—Dy2—N7	102.6 (3)
O9—Dy1—O6	76.7 (2)	N6—Dy2—N7	103.7 (3)
O3—Dy1—O6	77.7 (2)	O11—Dy3—O14	83.0 (3)
O21—Dy1—O6	116.6 (2)	O11—Dy3—O7	141.5 (3)
O1—Dy1—O15	67.6 (2)	O14—Dy3—O7	76.7 (3)
O2—Dy1—O15	144.0 (2)	O11—Dy3—O16	78.9 (3)
O13—Dy1—O15	76.4 (2)	O14—Dy3—O16	143.2 (3)
O9—Dy1—O15	73.4 (2)	O7—Dy3—O16	134.4 (2)
O3—Dy1—O15	115.9 (2)	O11—Dy3—O9	114.2 (3)
O21—Dy1—O15	76.9 (2)	O14—Dy3—O9	145.8 (3)
O6—Dy1—O15	142.9 (2)	O7—Dy3—O9	71.8 (2)
O23—Dy2—O10	79.6 (3)	O16—Dy3—O9	71.0 (2)
O23—Dy2—O24	131.7 (3)	O11—Dy3—O13	146.5 (2)
O10—Dy2—O24	144.5 (3)	O14—Dy3—O13	112.4 (3)
O23—Dy2—O26	85.5 (3)	O7—Dy3—O13	72.0 (2)
O10—Dy2—O26	77.6 (3)	O16—Dy3—O13	71.3 (2)
O24—Dy2—O26	115.7 (3)	O9—Dy3—O13	70.3 (2)
O23—Dy2—O21	122.5 (2)	O11—Dy3—N9	94.5 (3)
O10—Dy2—O21	71.8 (2)	O14—Dy3—N9	73.0 (4)

Symmetry codes: (i) *x*-1, *y*-1, *z*; (ii) *x*+1, *y*+1, *z*.

Table S2. Complete assignment of the ^{1}H and ^{13}C NMR spectra of **8** in $CDCl_3$ solution.

	2*a*		*b*		2*c*		2*d*		2*e*		aib	
	H	C	H	C	H	C	H	C	H	C	H	C
1	-	111.22	-	111.65	-	110.4	-	115.5	-	112.5		
1a	-	135.07	-	135.49	-	135.14	-	133.86	-	134.96		
2	-	167.8	-	165.95	-	167.2	-	163.3	-	167.9		
4a	-	126.4	-	126.8	-	125.8	-	127.5	-	126.0		
3	6.265	125.1	6.966	125.0	5.064	125.33	6.697	124.35	5.967	125.7		
4	7.394	135.4	7.792	135.3	7.027	134.6	7.234	132.9	7.074	133.5		
5	7.618	129.03	7.715	129.13	7.425	128.6	7.536	128.5	7.525	128.5		
6	7.25	121.8	7.25	121.8	7.05	120.9	7.27	121.8	7.13	120.5		
7	7.491	127.3	7.477	127.3	7.252	126.63	7.424	126.34	7.362	126.34		
8	8.061	118.3	8.003	118.6	7.718	118.0	7.654	120.6	7.802	119.0		
9	9.404[a] 9.384[a]	161.59	9.27	159.47	8.96	159.71	8.881[a] 8.848[a]	158.41	8.84	158.84		
11	-	68.8	-	69.23	-	67.3	-	68.45	-	65.9	-	57.28
12	1.93[a] 1.913[a]	27.22	1.795	24.6	1.559[a] 1.519[a]	29.2[a] 29.2[a]	1.572[a] 1.298[a]	23.2[a] 22.82[a]	1.594	26.04	1.554	29.0
12’	1.509[a] 1.494[a]	30.09	1.518	31.84	1.363[a] 1.347[a]	28.5	0.94[a] 0.862[a]	30.02[a] 30.02[a]	1.052	28.77	1.468	29.0
13	-	184.32	-	184.5	-	184.4	-	186.7	-	187.0		184.9

[a] For these nuclei two different types of the corresponding *a*-*e* modes were observed.

Table S3. Inter-ligand NOEs observed experimentally in the 2D ^{1}H-^{1}H NOESY spectrum of 8 in CDCl$_3$ solution, and the respective proton-proton distances ($r_{H\text{-}H}$) from the X-ray crystal structure of the Dy complex.

H	a		b		c		d		e		aib	
	NOE	r (Å)	NOE	r (Å)	NOE	r (Å)	NOE	r (Å)	NOE	r (Å)	NOE	r (Å)
3	12b	3.53			12e	3.90	9a1					
	aib 12	3.32					12e	3.29				
6							9c	4.24				
9	3d	4.44	12d2	4.25	12’e	4.34						
					12’d1	3.59						
					12’d2	3.33						
12	3d	2.96	9d	4.41		-			3d	3.29	3a	3.52
	4d	4.54	3a	3.53					3c	3.90		
12’	-			-		-	3c	5.1	9c	4.19	8c	4.11
									3c	4.34		

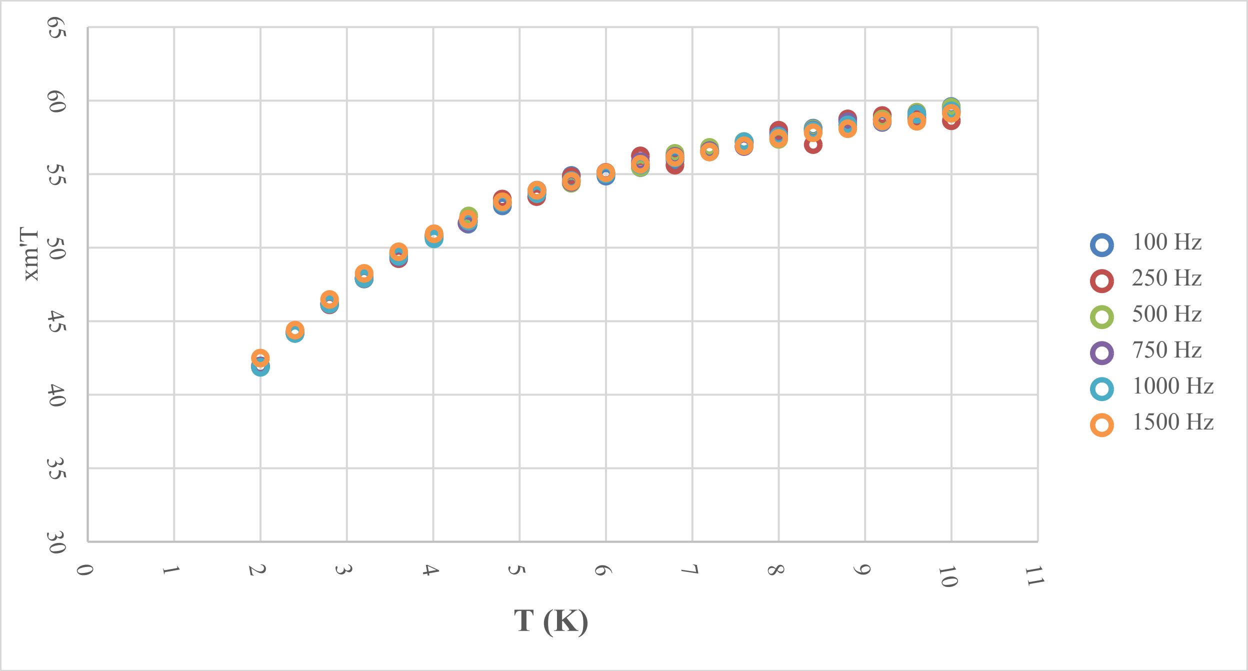


Figure S1. Plot of the in-phase ($\chi_{M'}T$) signals vs. temperature for **2**·4MeOH ([Tb_7]) in a 2.5 G field oscillating at the indicated frequencies.

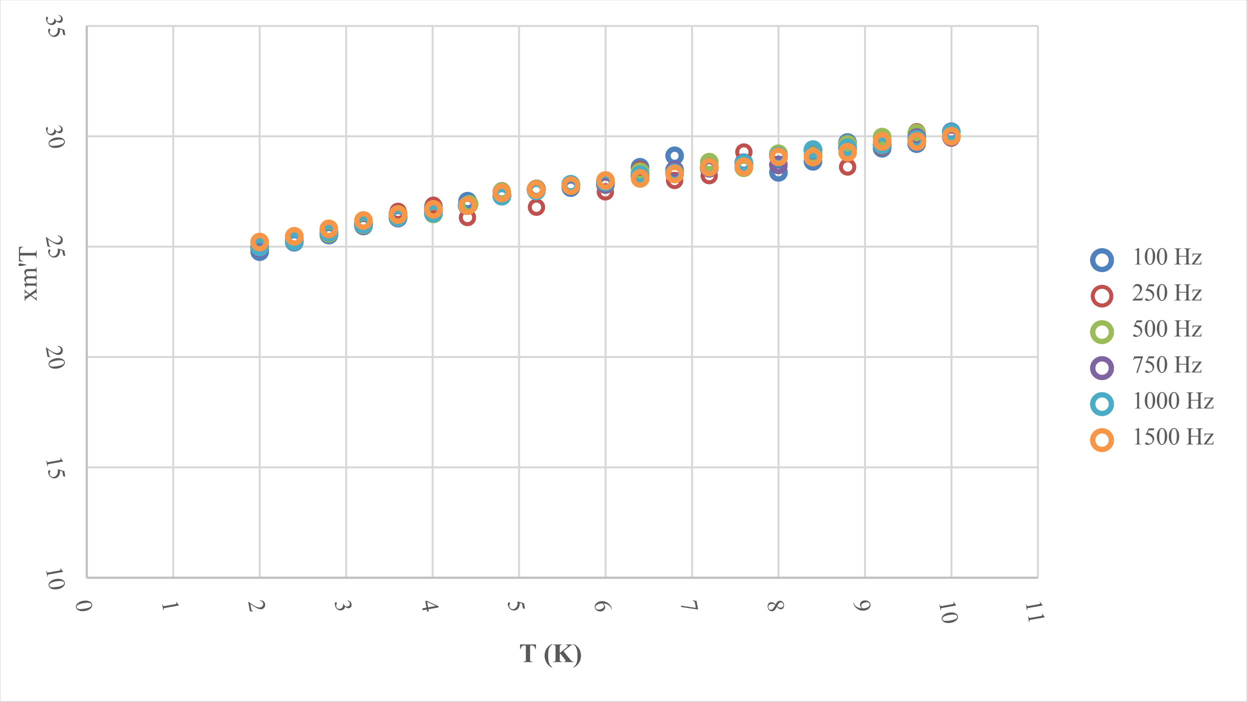


Figure S2. Plot of the in-phase ($\chi_{M'}T$) signals vs. temperature for **5**·4MeOH ([Er_7]) in a 2.5 G field oscillating at the indicated frequencies.

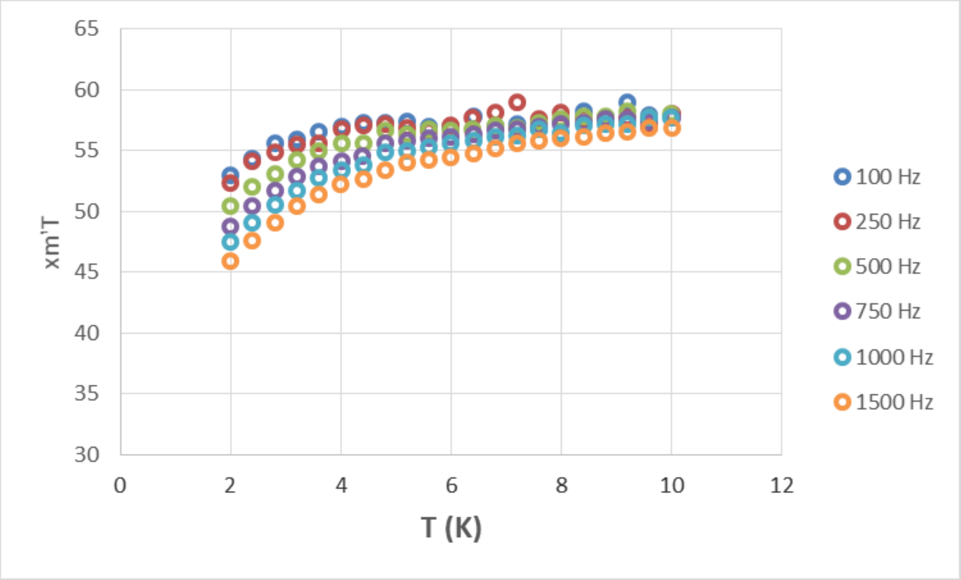
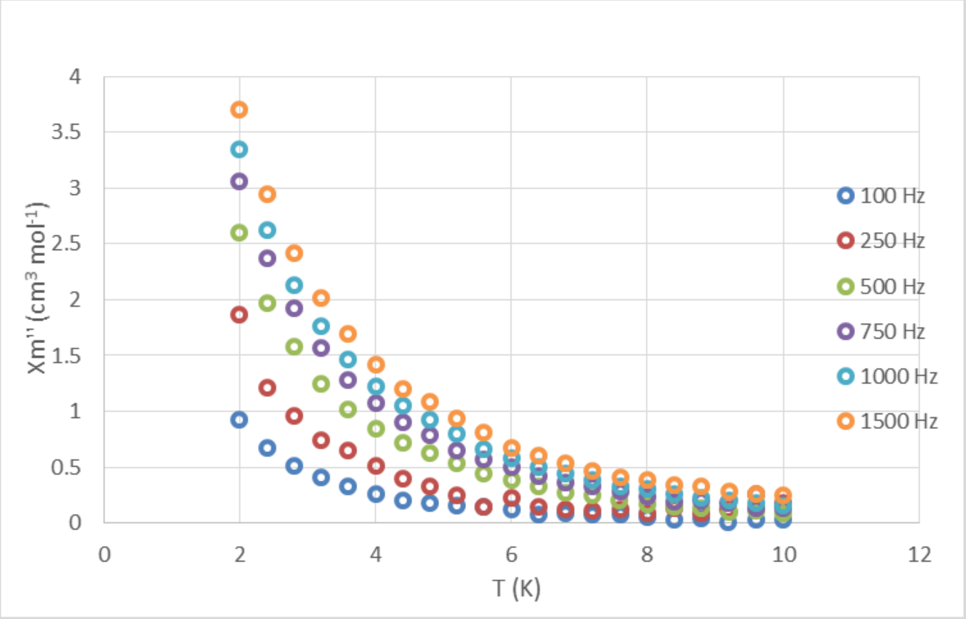


Figure S3. Plot of the in-phase ($\chi_{M'}$T) signals vs. temperature for **3**·4MeOH ([Dy$_7$]) in a 2.5 G field oscillating at the indicated frequencies.



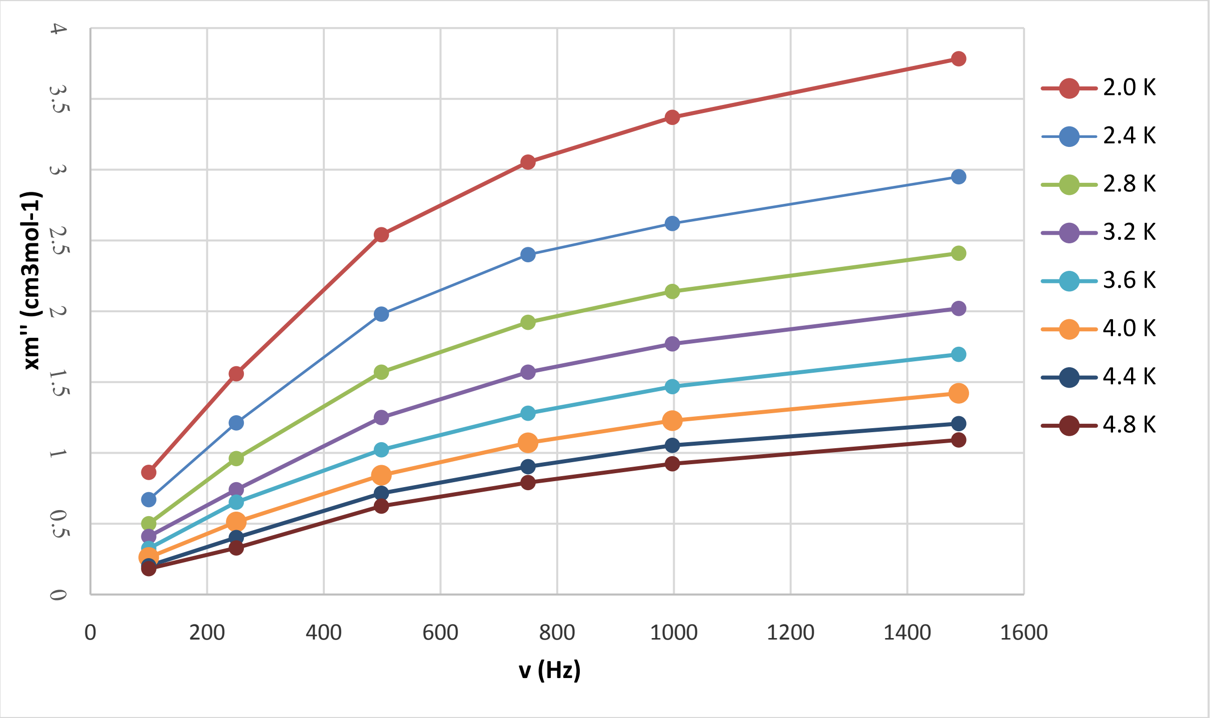


Figure S4. Plot of the out-of-phase (χ_M'') signals vs. temperature (top) and frequency (bottom) for **3**·4MeOH ([Dy_7]) in a 2.5 G field oscillating at the indicated frequencies.

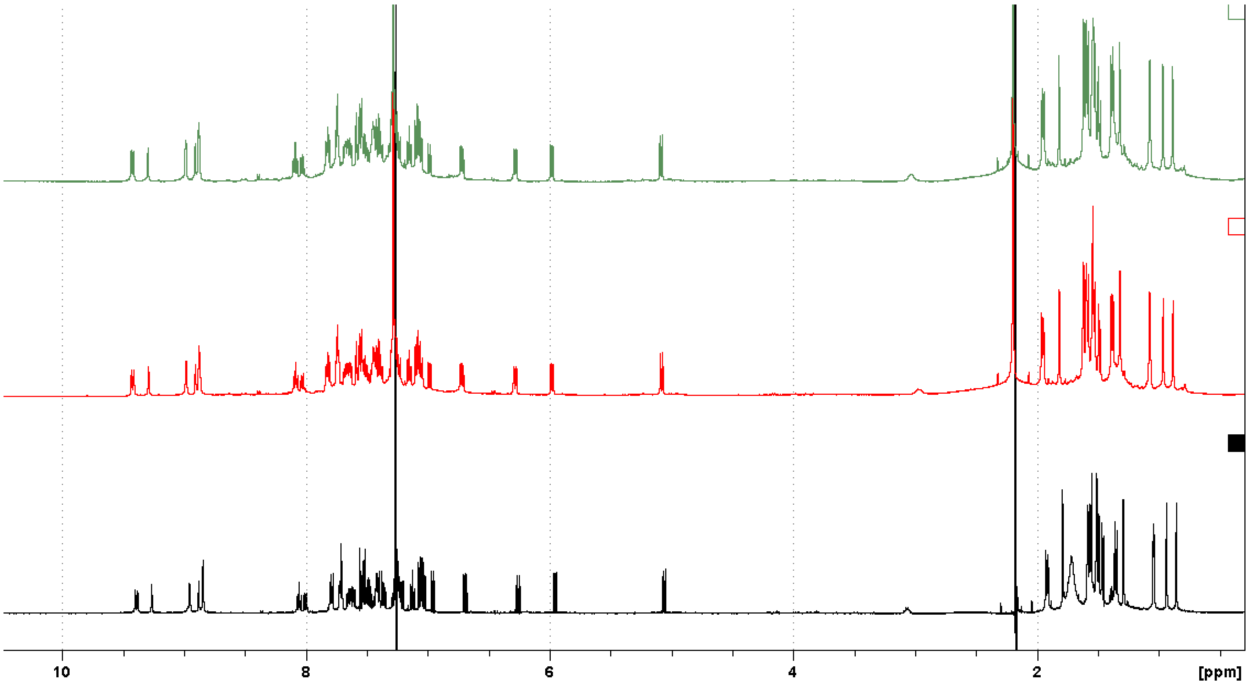


Figure S5. 500 MHz ^{1}H 1D NMR spectrum of **8** in $CDCl_3$ solution at 0 (bottom), 5 (middle) and 14 (top) days after solution preparation and storage at ambient temperature and light.

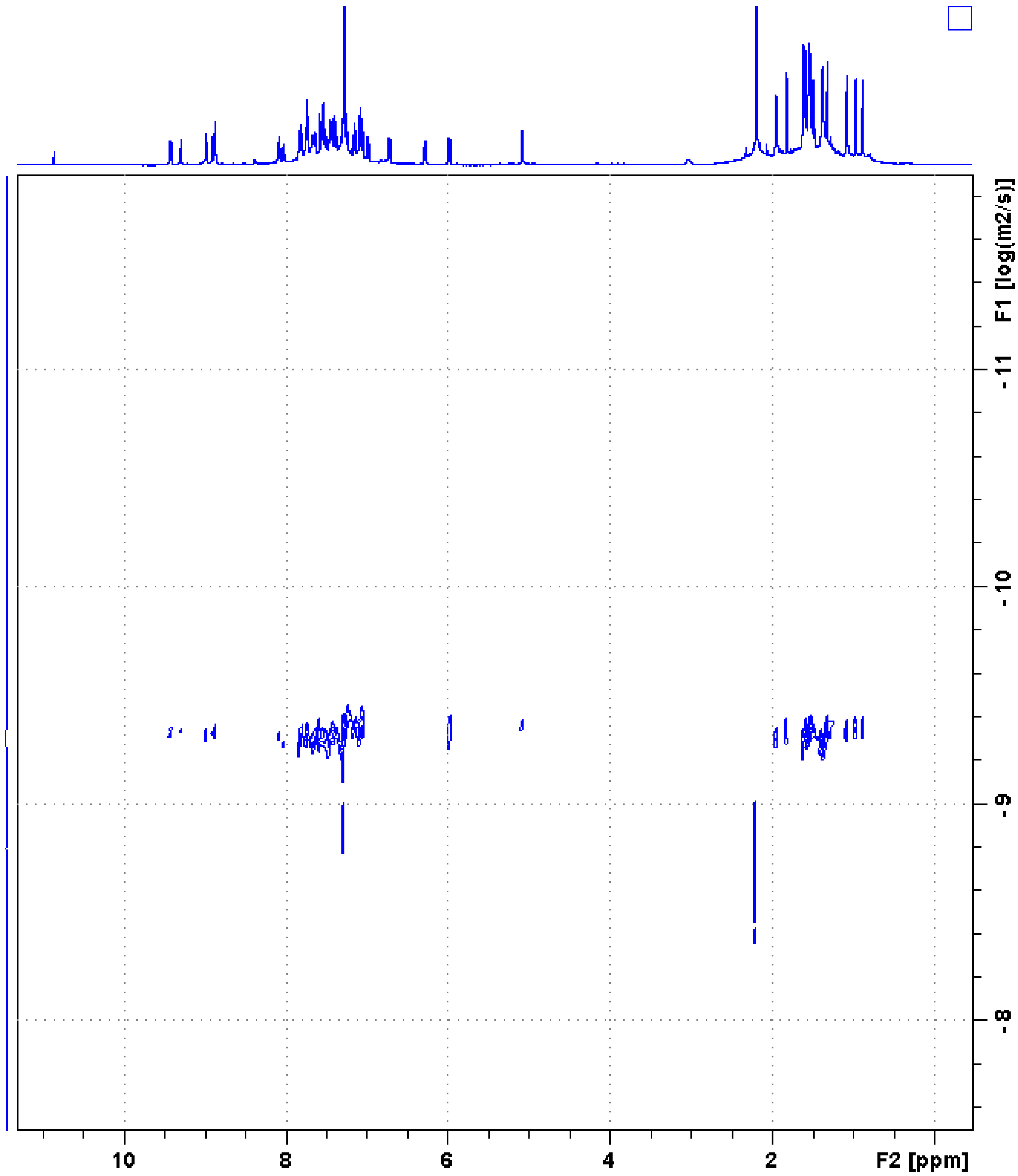


Figure S6. gDOSY ^1H 2D NMR spectrum of **8** in $CDCl_3$ solution for diffusion measurement using a stimulated echo, at 500 MHz. Similar results were obtained by performing the experiment on a DPX-300 NMR spectrometer. A viscosity value of 0.537 mPa s at 298K was used for the solvent in the Stokes-Einstein equation (CRC Handbook of Chemistry and Physics, 85th ed.). The self-diffusion coefficient D of **8** in $CDCl_3$ solution was calculated as ~ 4.0 x 10^{-10} m^2/s.

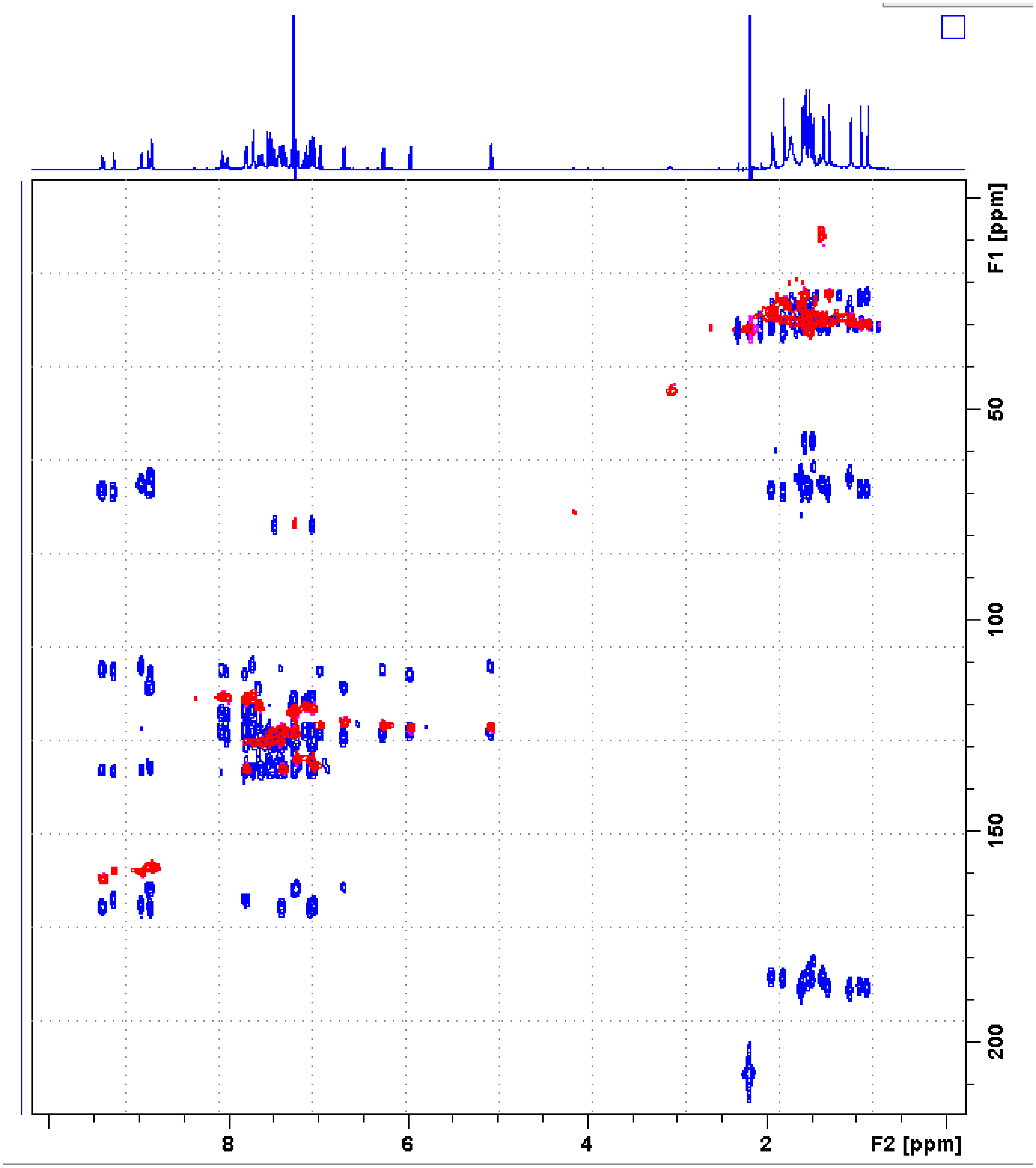


Figure S7. gHSQC (red, one bond C-H correlations) and gHMBC (blue, multiple bond correlations-long range) ^1H-^{13}C heteronuclear 2D NMR spectra of **8** in $CDCl_3$ solution at 500 MHz.

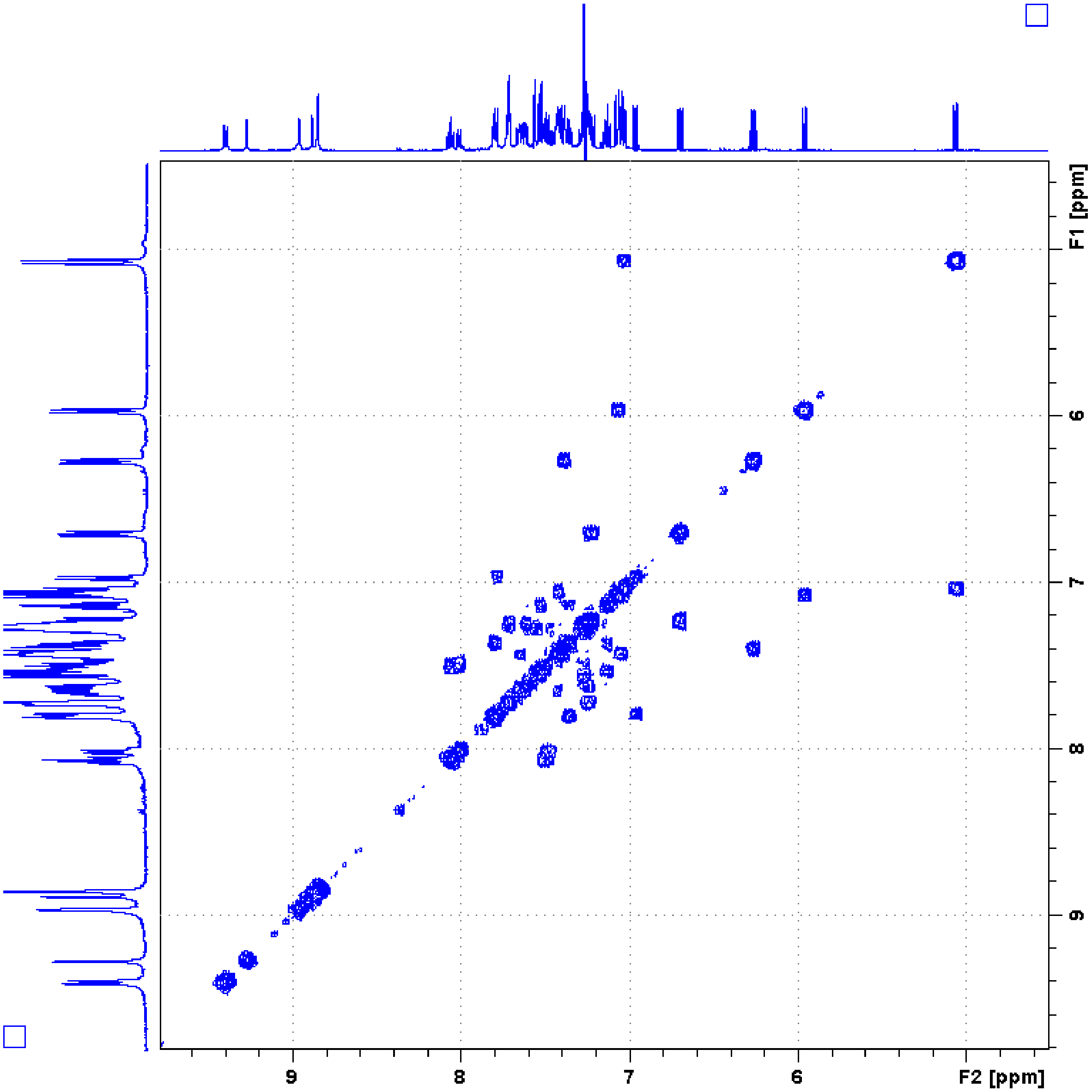


Figure S8. Aromatic region of the gCOSY homonuclear ^{1}H-^{1}H 2D NMR spectrum of **8** in $CDCl_3$ solution at 500 MHz.

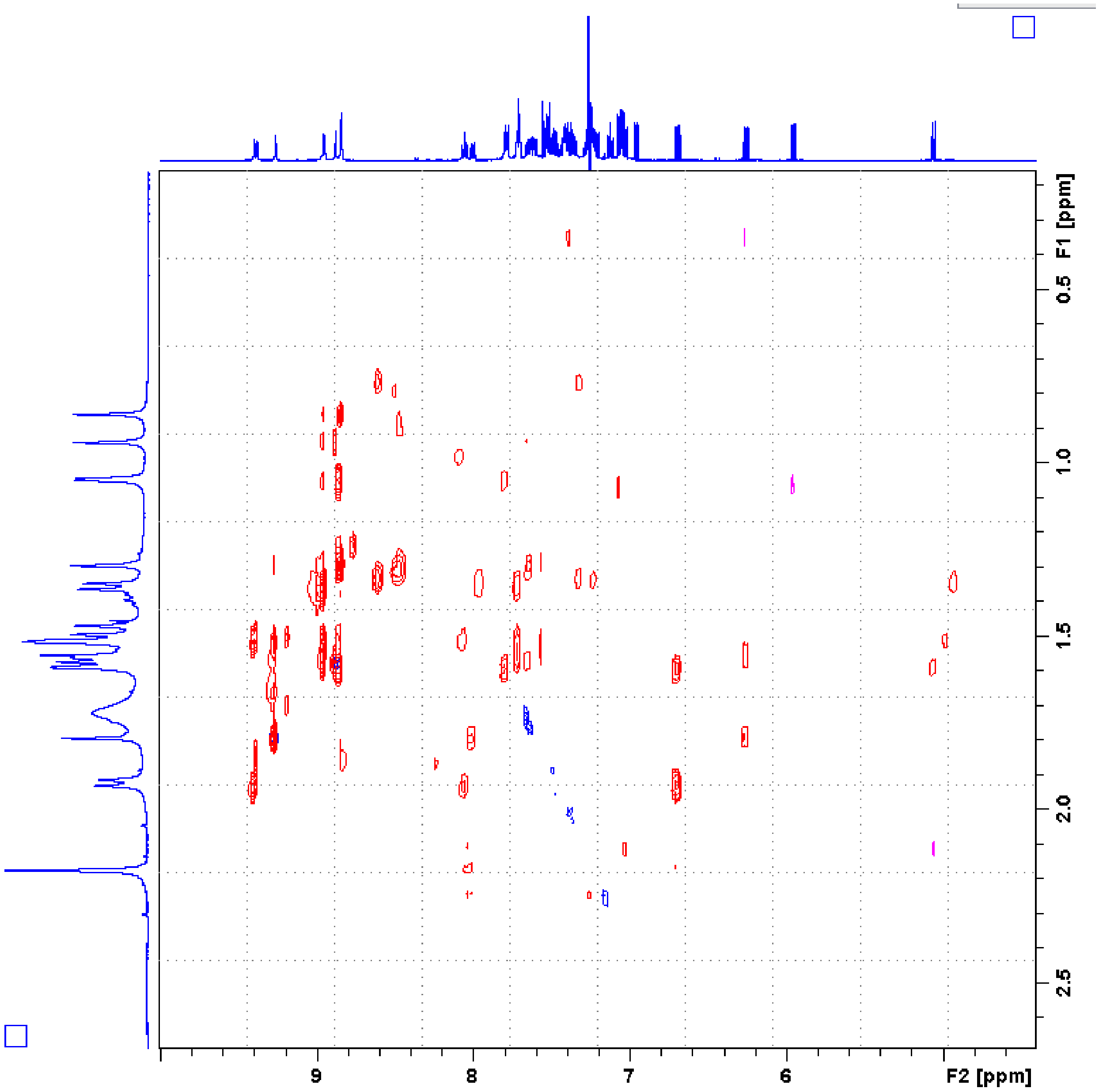


Figure S9. gNOESY ^{1}H-^{1}H 2D NMR spectrum of **8** in $CDCl_3$ solution at 500 MHz; the spectral region displaying the intra- and inter-ligand (L’) NOEs between protons H-12/12’ and aromatic protons 1-9 of structures *a*-*e* is shown. A mixing time of 250 ms was used.

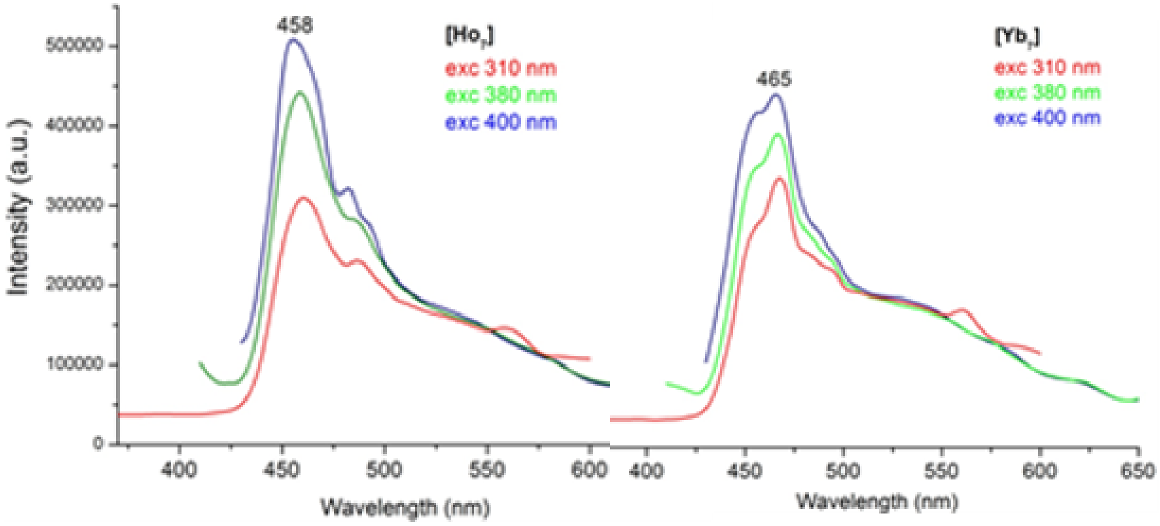


Figure S10. Solid-state room temperature emission spectra for 4 and 7 with various excitation wavelengths

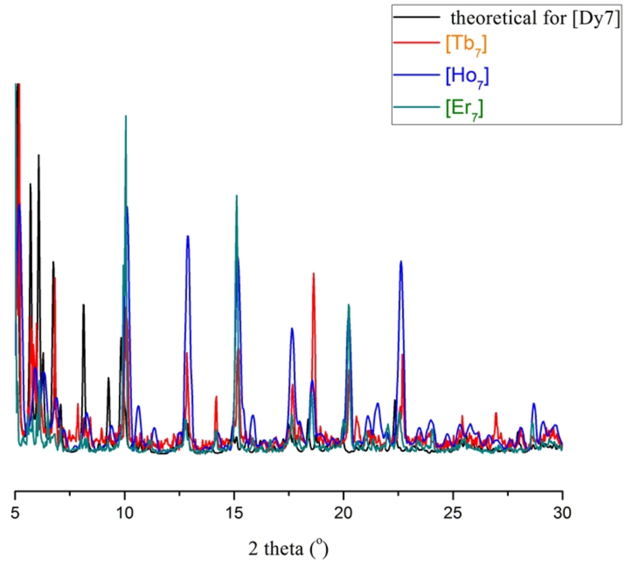


Figure S11. Powder XRD diagram comparison between complexes **1**, **6**, **7**, **8** and **3** (theoretical diagram).

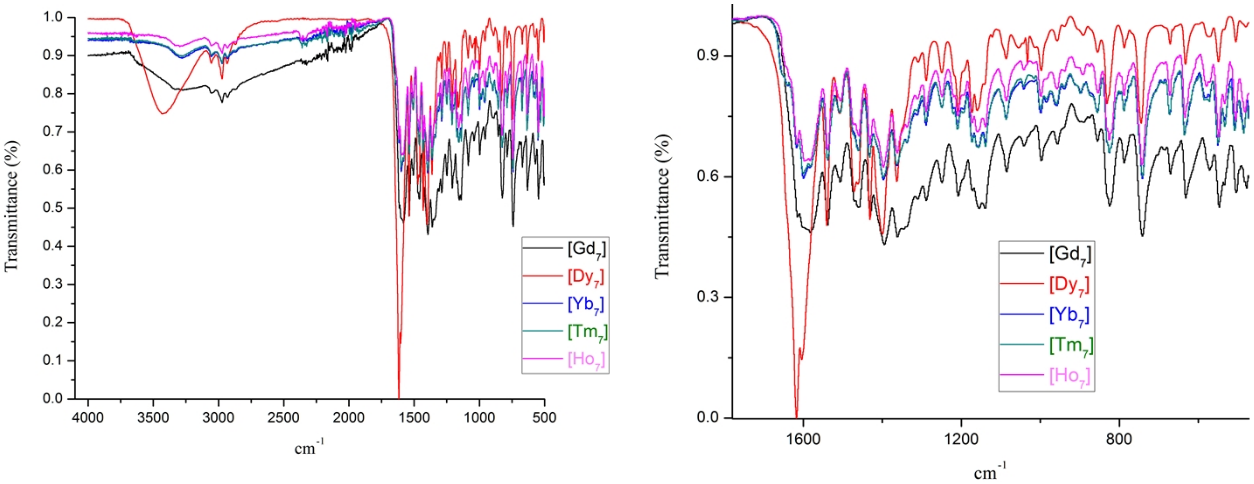


Figure S12. IR data comparison for representative complexes **1**, **3**, **4**, **6** and **7**.