

Supplementary information

Structure, Spectral, Thermal, Magnetic Properties, and Hydrolytic Stability of Three Novel Fluorinated MOFs Based on Ho(III), Dy(III), and Yb(III) Ions

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Table S1 Crystal data for compound **UPJS-21(Ho)** and **UPJS-22(Yb)**.

Sample designation	UPJS-21(Ho)	UPJS-22(Yb)
Empirical formula	C _{60.6} H _{46.7} N _{3.5} O _{15.5} F ₁₈ Ho ₂	C ₄₃ H ₃₄ N ₂ O ₁₄ F ₁₂ Yb ₂
Formula weight	1743.77	1376.80
Temperature	120(2) K	150(2) K
Wavelength	1.54178 Å	0.71073
Crystal system	Monoclinic	Triclinic
Space group	<i>P2/c</i>	<i>P-1</i>
Unit cell dimensions	<i>a</i> = 17.9849(17) Å	<i>a</i> = 8.0141(8) Å
	<i>b</i> = 14.7321(13) Å	<i>b</i> = 14.6271(15) Å
	<i>c</i> = 31.121(3) Å	<i>c</i> = 21.274(2) Å
		<i>α</i> = 104.827(2) °
	<i>β</i> = 91.603(4) °	<i>β</i> = 97.524(2) °
	<i>γ</i> = 101.728(2) °	
Volume	8242.5(13) Å ³	2315.9(4) Å ³
<i>Z</i>	4	2
Density (calculated)	1.405 mg/m ³	1.974 mg/m ³
Absorption coefficient	4.327 mm ⁻¹	4.130 mm ⁻¹
<i>F</i> (000)	3419	1332
Crystal size	0.311 x 0.245 x 0.086 mm	0.673 x 0.202 x 0.120 mm
Theta range for data collection	3.458 to 79.670 °	2.130 to 27.133 °
Index ranges	-22 ≤ <i>h</i> ≤ 22 -15 ≤ <i>k</i> ≤ 18 -38 ≤ <i>l</i> ≤ 33	-10 ≤ <i>h</i> ≤ 10 -18 ≤ <i>k</i> ≤ 18 0 ≤ <i>l</i> ≤ 27
Reflections collected	121248	10395

Independent reflections	17662 [$R(\text{int}) = 0.0399$]	10395 [$R(\text{int}) = 0.0401$]
Data completeness	99.8%	99.9%
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	17662 / 1873 / 1143	10395 / 292 / 804
Goodness-of-fit on F^2	1.023	1.093
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0518$, $wR2 = 0.1468$	$R1 = 0.0196$, $wR2 = 0.0427$
R indices (all data)	$R1 = 0.0596$, $wR2 = 0.1553$	$R1 = 0.0238$, $wR2 = 0.0450$
Largest diff. peak and hole	1.610 and -1.306 e.Å ⁻³	0.883 and -0.762 e.Å ⁻³
Registration Number CCDC	2386674	2386675

Table S2 Selected geometric parameters [Å, °] for compound **UPJS-21(Ho)**.

Bond length	[Å]	Bond angle	[°]	Bond angle	[°]
Ho(1)-O(1)	2.262(3)	O(1)-Ho(1)-O(9)	79.51(10)	O(10)-Ho(2)-O(6)	122.87(11)
Ho(1)-O(9)	2.285(2)	O(1)-Ho(1)-O(3) ⁱ	146.22(10)	O(15)-Ho(2)-O(6)	76.7(3)
Ho(1)-O(3) ⁱ	2.298(2)	O(9)-Ho(1)-O(3) ⁱ	89.38(10)	O(2)-Ho(2)-O(6)	77.03(10)
Ho(1)-O(4) ⁱⁱ	2.327(2)	O(1)-Ho(1)-O(4) ⁱⁱ	76.63(9)	O(8A) ^{vi} -Ho(2)-O(6)	150.5(4)
Ho(1)-O(11) ⁱⁱⁱ	2.337(3)	O(9)-Ho(1)-O(4) ⁱⁱ	106.80(10)	O(13)-Ho(2)-O(6)	80.3(5)
Ho(1)-O(5)	2.418(3)	O(3) ⁱ -Ho(1)-O(4) ⁱⁱ	137.09(9)	O(14)-Ho(2)-O(7A) ^{vi}	81.8(6)
Ho(1)-O(12) ^{iv}	2.422(2)	O(1)-Ho(1)-O(11) ⁱⁱⁱ	131.86(9)	O(10)-Ho(2)-O(7A) ^{vi}	81.3(5)
Ho(1)-O(11) ^{iv}	2.500(2)	O(9)-Ho(1)-O(11) ⁱⁱⁱ	74.41(10)	O(2)-Ho(2)-O(7A) ^{vi}	121.1(4)
Ho(1)-Ho(1) ^v	3.9359(6)	O(3) ⁱ -Ho(1)-O(11) ⁱⁱⁱ	73.42(9)	O(8A) ^{vi} -Ho(2)-O(7A) ^{vi}	55.3(3)
Ho(2)-O(7)	2.120(8)	O(4) ⁱⁱ -Ho(1)-O(11) ⁱⁱⁱ	73.29(9)	O(13)-Ho(2)-O(7A) ^{vi}	72.2(7)
Ho(2)-O(14)	2.258(14)	O(1)-Ho(1)-O(5)	72.82(10)	O(6)-Ho(2)-O(7A) ^{vi}	143.4(5)
Ho(2)-O(8)	2.284(15)	O(9)-Ho(1)-O(5)	89.34(10)	C(2A)-C(1A)-C(6A)	120.0
Ho(2)-O(10)	2.285(3)	O(3) ⁱ -Ho(1)-O(5)	75.28(9)	C(2A)-C(1A)-C(7)	124.7(3)
Ho(2)-O(15)	2.311(8)	O(4) ⁱⁱ -Ho(1)-O(5)	142.04(9)	C(6A)-C(1A)-C(7)	115.1(3)
Ho(2)-O(2)	2.328(3)	O(11) ⁱⁱⁱ -Ho(1)-O(5)	144.66(8)	C(3A)-C(2A)-C(1A)	120.0
Ho(2)-O(8A) ^{vi}	2.34(2)	O(1)-Ho(1)-O(12) ^{iv}	88.20(9)	C(3A)-C(2A)-C(8A)	118.1(4)
Ho(2)-O(13)	2.377(17)	O(9)-Ho(1)-O(12) ^{iv}	161.89(10)	C(1A)-C(2A)-C(8A)	121.8(4)
Ho(2)-O(6)	2.389(3)	O(3) ⁱ -Ho(1)-O(12) ^{iv}	93.66(9)	C(4A)-C(3A)-C(2A)	120.0
Ho(2)-O(7A) ^{vi}	2.44(3)	O(4) ⁱⁱ -Ho(1)-O(12) ^{iv}	82.89(9)	C(3A)-C(4A)-C(5A)	120.0
C(1A)-C(2A)	1.3900	O(11) ⁱⁱⁱ -Ho(1)-O(12) ^{iv}	123.54(8)	C(3A)-C(4A)-C(12A)	120.9(5)
C(1A)-C(6A)	1.3900	O(5)-Ho(1)-O(12) ^{iv}	74.24(8)	C(5A)-C(4A)-C(12A)	119.1(5)
C(1A)-C(7)	1.536(5)	O(1)-Ho(1)-O(11) ^{iv}	132.14(8)	C(6A)-C(5A)-C(4A)	120.0
C(2A)-C(3A)	1.3900	O(9)-Ho(1)-O(11) ^{iv}	144.18(9)	C(5A)-C(6A)-C(1A)	120.0
C(2A)-C(8A)	1.492(7)	O(3) ⁱ -Ho(1)-O(11) ^{iv}	71.76(8)	F(1A)-C(8A)-F(3A)	107.3(7)
C(3A)-C(4A)	1.3900	O(4) ⁱⁱ -Ho(1)-O(11) ^{iv}	72.38(8)	F(1A)-C(8A)-F(2A)	105.1(6)
C(4A)-C(5A)	1.3900	O(11) ⁱⁱⁱ -Ho(1)-O(11) ^{iv}	71.15(9)	F(3A)-C(8A)-F(2A)	109.4(6)
C(4A)-C(12A)	1.512(4)	O(5)-Ho(1)-O(11) ^{iv}	113.59(8)	O(2)-C(7)-O(1)	125.3(3)
C(5A)-C(6A)	1.3900	O(12) ^{iv} -Ho(1)-O(11) ^{iv}	52.85(8)	O(2)-C(7)-C(1A)	114.2(4)
C(8A)-F(1A)	1.336(6)	O(7)-Ho(2)-O(10)	156.5(5)	O(1)-C(7)-C(1A)	120.3(3)
C(8A)-F(3A)	1.339(6)	O(14)-Ho(2)-O(10)	83.4(4)	C(11A)-C(12A)-C(13A)	120.0
C(8A)-F(2A)	1.342(6)	O(8)-Ho(2)-O(10)	154.5(5)	C(11A)-C(12A)-C(4A)	120.8(8)
C(7)-O(2)	1.242(5)	O(7)-Ho(2)-O(15)	90.8(4)	C(13A)-C(12A)-C(4A)	118.9(8)
C(7)-O(1)	1.263(5)	O(10)-Ho(2)-O(15)	77.0(2)	C(12A)-C(11A)-C(10A)	120.0

C(12A)-C(11A)	1.3900	O(7)-Ho(2)-O(2)	86.1(5)	C(11A)-C(10A)-C(9A)	120.0
C(12A)-C(13A)	1.3900	O(14)-Ho(2)-O(2)	152.1(5)	C(11A)-C(10A)-C(16A)	118.5(9)
C(11A)-C(10A)	1.3900	O(8)-Ho(2)-O(2)	83.7(5)	C(9A)-C(10A)-C(16A)	121.4(9)
C(10A)-C(9A)	1.3900	O(10)-Ho(2)-O(2)	113.72(11)	C(10A)-C(9A)-C(14A)	120.0
C(10A)-C(16A)	1.508(7)	O(15)-Ho(2)-O(2)	153.2(3)	C(10A)-C(9A)-C(15A)	126.0(8)
C(9A)-C(14A)	1.3900	O(14)-Ho(2)-O(8A) ^{vi}	133.6(6)	C(14A)-C(9A)-C(15A)	114.0(8)
C(9A)-C(15A)	1.528(5)	O(10)-Ho(2)-O(8A) ^{vi}	74.3(4)	C(13A)-C(14A)-C(9A)	120.0
C(14A)-C(13A)	1.3900	O(2)-Ho(2)-O(8A) ^{vi}	73.8(4)	C(14A)-C(13A)-C(12A)	120.0
C(15A)-O(3)	1.245(5)	O(14)-Ho(2)-O(13)	91.4(6)	O(3)-C(15A)-O(4)	127.7(3)
C(15A)-O(4)	1.257(4)	O(10)-Ho(2)-O(13)	153.4(5)	F(6A)-C(16A)-F(5A)	106.1(9)
C(16A)-F(6A)	1.339(6)	O(2)-Ho(2)-O(13)	82.1(5)	F(6A)-C(16A)-F(4A)	107.0(9)
C(16A)-F(5A)	1.346(6)	O(8A) ^{vi} -Ho(2)-O(13)	91.2(6)	F(5A)-C(16A)-F(4A)	107.5(9)
C(16A)-F(4A)	1.347(6)	O(7)-Ho(2)-O(6)	72.2(4)	F(6A)-C(16A)-C(10A)	111.7(11)
		O(14)-Ho(2)-O(6)	75.2(5)	F(5A)-C(16A)-C(10A)	111.0(10)
		O(8)-Ho(2)-O(6)	77.9(4)	F(4A)-C(16A)-C(10A)	113.2(10)

Symmetry transformations used to generate equivalent atoms:

ⁱ $x, y-1, z$ ⁱⁱ $-x, y+1, -z+1$ ⁱⁱⁱ $-x, y, -z+1/2$ ^{iv} $x, -y, z+1/2$ ^v $-x, -y, -z+1$ ^{vi} $x, -y, z-1/2$ ^{vii} $x, y+1, z$

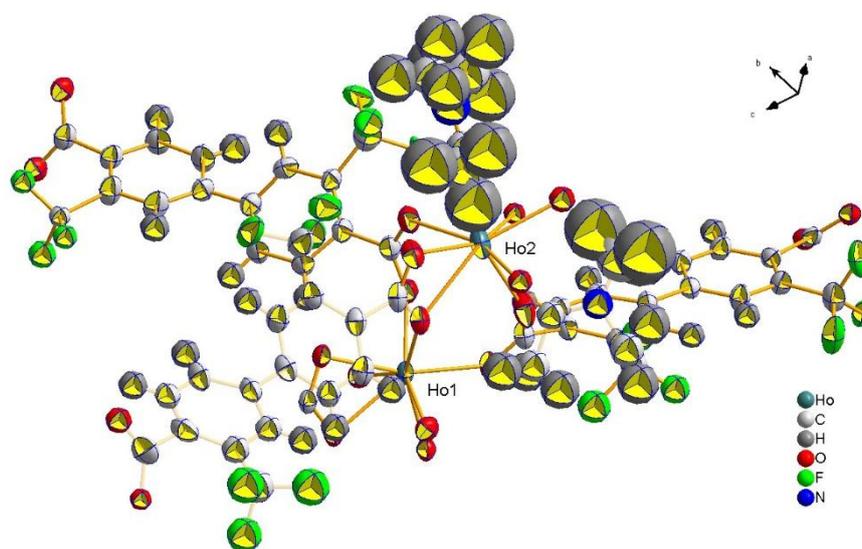


Fig. S1 The ellipsoid view of the structure model of **UPJS-21(Ho)**.

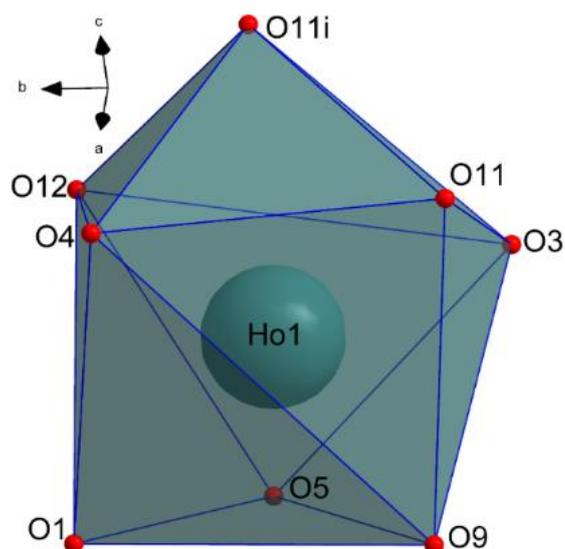


Fig. S2 The coordination geometry of Ho1(III) ion forming deformed triangular dodecahedron shape in the **UPJS-21(Ho)** compound. See also Table S3.

Table S3 The geometrical deviation of a coordination environment from an ideal reference polyhedron in the **UPJS-21(Ho)** calculated by SHAPE software. A value of 0 indicates the coordination environment perfectly matches the ideal reference shape. The values < 1.0 represent minor distortions from the ideal geometry. The values 1.0 – 3.0 indicate significant distortions, though the reference shape still provides a reasonable description of the stereochemistry. The values > 3.0 show that the structure is poorly described by that specific reference shape.

Shape [ML ₈]	OP	HPY	HBPY	CU	SAPR	TDD	JGBF	JETBPY	JBTPR	BTPR	JSD	TT	ETBPY
Ho1	32.533	22.133	14.402	10.724	2.063	1.820	12.405	28.221	2.709	2.305	3.699	11.359	23.664
Ho2	27.823	23.308	9.896	7.002	3.646	2.826	10.486	25.312	3.768	2.970	5.282	7.507	22.838

Abbreviations: OP – Octagon, HPY - Heptagonal pyramid, HBPY - Hexagonal bipyramid, CU – Cube, SAPR - Square antiprism, TDD - Triangular dodecahedron, JGBF - Johnson gyrobifastigium, JETBPY - Johnson elongated triangular bipyramid, JBTPR - Biaugmented trigonal prism, BTPR - Biaugmented trigonal prism, JSD - Snub diphenoïd, TT - Triakistetrahedron, ETBPY - Elongated trigonal bipyramid

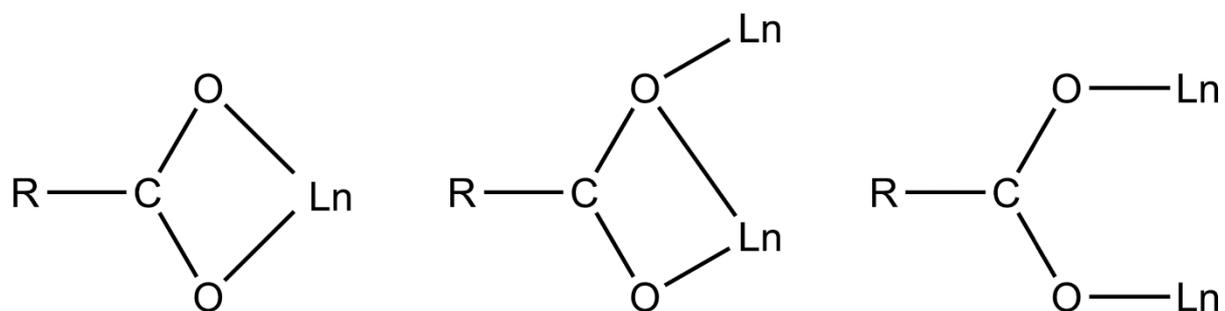


Fig. S3 Schematic view of different coordination modes observed in **UPJS-21(Ho)** compound.

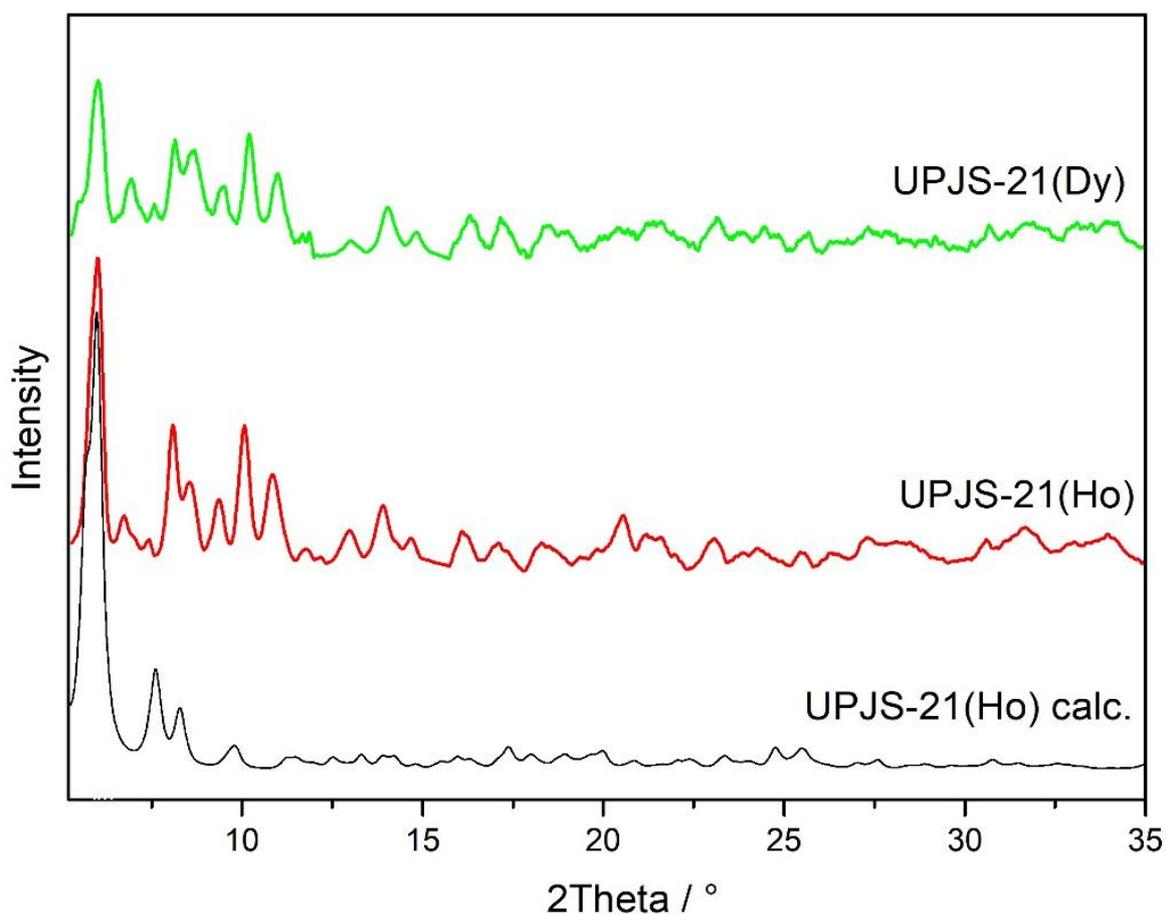


Fig. S4 The PXR D patterns of the **UPJS-21(Ho)** and **UPJS-21(Dy)** compounds. The small differences in intensities of experimental and simulated patterns may arise from low crystallisation, different solvation and preferential orientation of the crystals.

Table S4 Selected geometric parameters [Å, °] for compound **UPJS-22(Yb)**.

<i>Bond length</i>	[Å]	<i>Bond angle</i>	[°]	<i>Bond angle</i>	[°]
Yb(1)-O(21)	2.207(2)	O(21)-Yb(1)-O(23) ⁱ	87.29(8)	O(22)-Yb(2)-Yb(1) ^{vi}	115.64(6)
Yb(1)-O(23) ⁱ	2.228(2)	O(21)-Yb(1)-O(3) ⁱⁱ	176.51(8)	O(2)-Yb(2)-Yb(1) ^{vi}	138.86(5)
Yb(1)-O(3) ⁱⁱ	2.229(2)	O(23) ⁱ -Yb(1)-O(3) ⁱⁱ	95.10(8)	O(4) ^{iv} -Yb(2)-Yb(1) ^{vi}	63.68(6)
Yb(1)-O(1)	2.244(2)	O(21)-Yb(1)-O(1)	95.50(8)	O(24) ^v -Yb(2)-Yb(1) ^{vi}	63.14(5)
Yb(1)-O(42) ⁱⁱⁱ	2.290(2)	O(23) ⁱ -Yb(1)-O(1)	154.02(8)	O(52)-Yb(2)-Yb(1) ^{vi}	127.58(5)
Yb(1)-O(51)	2.344(2)	O(3) ⁱⁱ -Yb(1)-O(1)	81.24(8)	O(41)-Yb(2)-Yb(1) ^{vi}	79.28(6)
Yb(1)-O(52)	2.403(2)	O(21)-Yb(1)-O(42) ⁱⁱⁱ	88.74(8)	O(42)-Yb(2)-Yb(1) ^{vi}	27.39(5)
Yb(1)-C(51)	2.754(3)	O(23) ⁱ -Yb(1)-O(42) ⁱⁱⁱ	76.31(7)	C(41)-Yb(2)-Yb(1) ^{vi}	54.12(6)
Yb(2)-O(22)	2.2107(19)	O(3) ⁱⁱ -Yb(1)-O(42) ⁱⁱⁱ	89.36(8)	O(22)-Yb(2)-Yb(1)	64.12(6)
Yb(2)-O(2)	2.220(2)	O(1)-Yb(1)-O(42) ⁱⁱⁱ	77.94(7)	O(2)-Yb(2)-Yb(1)	65.79(5)
Yb(2)-O(4) ^{iv}	2.224(2)	O(21)-Yb(1)-O(51)	101.67(8)	O(4) ^{iv} -Yb(2)-Yb(1)	113.39(6)
Yb(2)-O(24) ^v	2.251(2)	O(23) ⁱ -Yb(1)-O(51)	79.59(8)	O(24) ^v -Yb(2)-Yb(1)	89.12(5)
Yb(2)-O(52)	2.296(2)	O(3) ⁱⁱ -Yb(1)-O(51)	81.29(8)	O(52)-Yb(2)-Yb(1)	28.75(5)
Yb(2)-O(41)	2.333(2)	O(1)-Yb(1)-O(51)	124.67(8)	O(41)-Yb(2)-Yb(1)	127.30(6)
Yb(2)-O(42)	2.392(2)	O(42) ⁱⁱⁱ -Yb(1)-O(51)	153.22(8)	O(42)-Yb(2)-Yb(1)	158.00(5)
Yb(2)-C(41)	2.745(3)	O(21)-Yb(1)-O(52)	84.25(8)	C(41)-Yb(2)-Yb(1)	147.86(6)
C(1)-C(6)	1.391(4)	O(23) ⁱ -Yb(1)-O(52)	130.44(7)	Yb(1) ^{vi} -Yb(2)-Yb(1)	150.962(5)
C(1)-C(2)	1.399(4)	O(3) ⁱⁱ -Yb(1)-O(52)	96.08(8)	C(6)-C(1)-C(2)	118.3(3)
C(1)-C(7)	1.503(4)	O(1)-Yb(1)-O(52)	75.51(7)	C(6)-C(1)-C(7)	115.6(3)
C(2)-C(3)	1.393(4)	O(42) ⁱⁱⁱ -Yb(1)-O(52)	151.70(7)	C(2)-C(1)-C(7)	126.0(3)
C(2)-C(8)	1.510(4)	O(51)-Yb(1)-O(52)	54.89(7)	C(3)-C(2)-C(1)	120.6(3)
C(3)-C(4)	1.396(4)	O(21)-Yb(1)-C(51)	95.52(9)	C(3)-C(2)-C(8)	118.2(3)
C(4)-C(5)	1.392(4)	O(23) ⁱ -Yb(1)-C(51)	105.28(8)	C(1)-C(2)-C(8)	121.2(2)
C(4)-C(9)	1.494(4)	O(3) ⁱⁱ -Yb(1)-C(51)	86.31(8)	C(2)-C(3)-C(4)	120.8(3)
C(5)-C(6)	1.391(4)	O(1)-Yb(1)-C(51)	100.16(8)	C(5)-C(4)-C(3)	118.4(3)
C(7)-O(1)	1.258(4)	O(42) ⁱⁱⁱ -Yb(1)-C(51)	175.50(8)	C(5)-C(4)-C(9)	119.4(3)
C(7)-O(2)	1.260(4)	O(51)-Yb(1)-C(51)	26.73(8)	C(3)-C(4)-C(9)	122.1(3)
C(8)-F(1)	1.337(4)	O(52)-Yb(1)-C(51)	28.31(8)	C(6)-C(5)-C(4)	120.8(3)
C(8)-F(3)	1.338(4)	O(21)-Yb(1)-Yb(2) ⁱⁱⁱ	113.90(6)	C(1)-C(6)-C(5)	121.1(3)
C(8)-F(2)	1.340(4)	O(23) ⁱ -Yb(1)-Yb(2) ⁱⁱⁱ	65.91(5)	O(1)-C(7)-O(2)	124.3(2)
C(9)-C(14)	1.389(4)	O(3) ⁱⁱ -Yb(1)-Yb(2) ⁱⁱⁱ	65.02(6)	O(1)-C(7)-C(1)	118.8(3)
C(9)-C(10)	1.398(4)	O(1)-Yb(1)-Yb(2) ⁱⁱⁱ	89.60(6)	O(2)-C(7)-C(1)	116.8(2)
C(10)-C(11)	1.397(4)	O(42) ⁱⁱⁱ -Yb(1)-Yb(2) ⁱⁱⁱ	28.72(5)	C(7)-O(1)-Yb(1)	131.47(19)
C(11)-C(12)	1.406(4)	O(51)-Yb(1)-Yb(2) ⁱⁱⁱ	127.78(6)	C(7)-O(2)-Yb(2)	132.89(18)
C(11)-C(15)	1.509(4)	O(52)-Yb(1)-Yb(2) ⁱⁱⁱ	157.85(5)	F(1)-C(8)-F(3)	106.9(3)
C(12)-C(13)	1.389(4)	C(51)-Yb(1)-Yb(2) ⁱⁱⁱ	148.04(6)	F(1)-C(8)-F(2)	106.5(3)
C(12)-C(16)	1.512(4)	O(21)-Yb(1)-Yb(2)	63.62(6)	F(3)-C(8)-F(2)	106.3(3)
C(13)-C(14)	1.392(4)	O(23) ⁱ -Yb(1)-Yb(2)	138.90(5)	F(1)-C(8)-C(2)	111.7(2)
C(15)-F(4)	1.338(5)	O(3) ⁱⁱ -Yb(1)-Yb(2)	115.52(6)	F(3)-C(8)-C(2)	112.4(3)
C(15)-F(5)	1.339(5)	O(1)-Yb(1)-Yb(2)	62.82(6)	F(2)-C(8)-C(2)	112.6(3)
C(15)-F(6)	1.346(4)	O(42) ⁱⁱⁱ -Yb(1)-Yb(2)	127.52(5)	C(14)-C(9)-C(10)	118.3(3)
C(16)-O(3)	1.249(4)	O(51)-Yb(1)-Yb(2)	78.85(6)	C(14)-C(9)-C(4)	120.6(3)
C(16)-O(4)	1.263(4)	O(52)-Yb(1)-Yb(2)	27.36(5)	C(10)-C(9)-C(4)	121.1(3)
O(61A)-C(61A)	1.204(16)	C(51)-Yb(1)-Yb(2)	53.88(6)	C(11)-C(10)-C(9)	121.2(3)

C(61A)-N(61A)	1.298(17)	Yb(2) ⁱⁱⁱ -Yb(1)-Yb(2)	150.962(5)	C(10)-C(11)-C(12)	120.0(3)
C(61A)-C(62A)	1.527(18)	O(22)-Yb(2)-O(2)	96.02(8)	C(10)-C(11)-C(15)	117.7(3)
N(61A)-C(64A)	1.361(16)	O(22)-Yb(2)-O(4) ^{iv}	174.20(8)	C(12)-C(11)-C(15)	122.2(3)
N(61A)-C(63A)	1.430(17)	O(2)-Yb(2)-O(4) ^{iv}	87.34(8)	C(13)-C(12)-C(11)	118.3(3)
N(71A)-C(71A)	1.301(14)	O(22)-Yb(2)-O(24) ^v	80.78(8)	C(13)-C(12)-C(16)	116.8(3)
N(71A)-C(73A)	1.408(16)	O(2)-Yb(2)-O(24) ^v	152.96(8)	C(11)-C(12)-C(16)	124.9(3)
N(71A)-C(74A)	1.491(16)	O(4) ^{iv} -Yb(2)-O(24) ^v	94.07(8)	C(12)-C(13)-C(14)	121.3(3)
C(71A)-O(71A)	1.182(12)	O(22)-Yb(2)-O(52)	88.27(8)	C(9)-C(14)-C(13)	120.7(3)
		O(2)-Yb(2)-O(52)	75.85(7)	F(4)-C(15)-F(5)	107.2(3)
		O(4) ^{iv} -Yb(2)-O(52)	87.97(8)	F(4)-C(15)-F(6)	106.5(3)
		O(24) ^v -Yb(2)-O(52)	77.22(7)	F(5)-C(15)-F(6)	105.9(3)
		O(22)-Yb(2)-O(41)	82.33(8)	F(4)-C(15)-C(11)	112.5(3)
		O(2)-Yb(2)-O(41)	79.76(8)	F(5)-C(15)-C(11)	112.9(3)
		O(4) ^{iv} -Yb(2)-O(41)	102.96(8)	F(6)-C(15)-C(11)	111.3(3)
		O(24) ^v -Yb(2)-O(41)	125.81(7)	O(3)-C(16)-O(4)	125.0(3)
		O(52)-Yb(2)-O(41)	152.75(8)	O(3)-C(16)-C(12)	119.0(3)
		O(22)-Yb(2)-O(42)	96.80(8)	O(4)-C(16)-C(12)	116.0(2)
		O(2)-Yb(2)-O(42)	130.52(7)	C(16)-O(3)-Yb(1) ⁱⁱ	139.11(19)
		O(4) ^{iv} -Yb(2)-O(42)	84.50(7)	C(16)-O(4)-Yb(2) ^{iv}	143.36(19)
		O(24) ^v -Yb(2)-O(42)	76.42(7)	O(61A)-C(61A)-N(61A)	127.6(13)
		O(52)-Yb(2)-O(42)	151.94(7)	O(61A)-C(61A)-C(62A)	128.8(14)
		O(41)-Yb(2)-O(42)	55.11(7)	N(61A)-C(61A)-C(62A)	103.3(12)
		O(22)-Yb(2)-C(41)	87.43(8)	C(61A)-N(61A)-C(64A)	142.1(12)
		O(2)-Yb(2)-C(41)	105.37(8)	C(61A)-N(61A)-C(63A)	121.7(12)
		O(4) ^{iv} -Yb(2)-C(41)	96.26(8)	C(64A)-N(61A)-C(63A)	95.8(12)
		O(24) ^v -Yb(2)-C(41)	101.32(8)	C(71A)-N(71A)-C(73A)	125.5(12)
		O(52)-Yb(2)-C(41)	175.64(8)	C(71A)-N(71A)-C(74A)	120.4(11)
		O(41)-Yb(2)-C(41)	26.78(8)	C(73A)-N(71A)-C(74A)	114.1(10)
		O(42)-Yb(2)-C(41)	28.46(8)	O(71A)-C(71A)-N(71A)	124.8(13)

Symmetry transformations used to generate equivalent atoms:

ⁱ -x,-y,-z+1

ⁱⁱ -x,-y+1,-z

ⁱⁱⁱ x-1,y,z

^{iv} -x+1,-y+1,-z

^v -x+1,-y,-z+1

^{vi} x+1,y,z

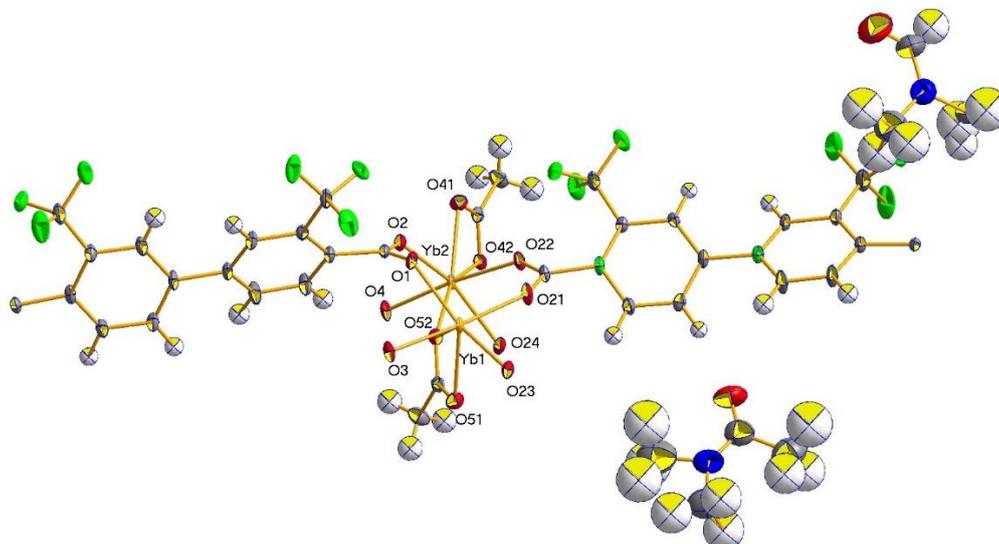


Fig. S5 The ellipsoid view of the structure model of **UPJS-22(Yb)**.

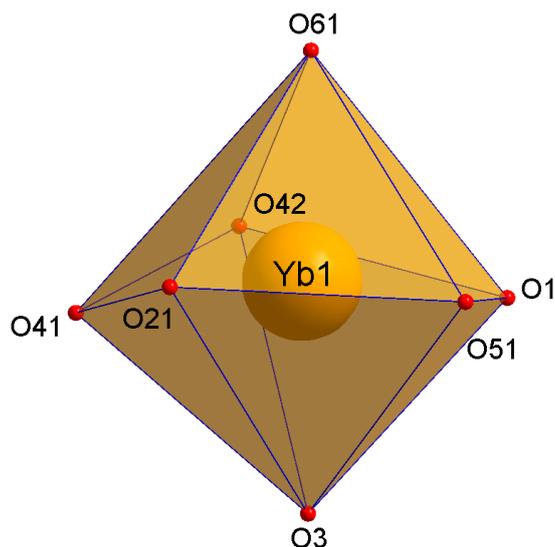


Fig. S6 The coordination geometry of Yb1(III) ion forming deformed pentagonal bipyramid shape in the **UPJS-22(Yb)** compound. See also Table S5.

Table S5. The geometrical deviation of a coordination environment from an ideal reference polyhedron in the **UPJS-22(Yb)** calculated by SHAPE software. A value of 0 indicates the coordination environment perfectly matches the ideal reference shape. The values < 1.0 represent minor distortions from the ideal geometry. The values 1.0 – 3.0 indicate significant distortions, though the reference shape still provides a reasonable description of the stereochemistry. The values > 3.0 show that the structure is poorly described by that specific reference shape.

Shape [ML7]	HP	HPY	PBPY	COC	CTPR	JPBPY	JETPY
Yb1	33.352	24.019	1.770	4.503	2.984	4.925	21.610
Yb2	33.430	24.057	1.830	4.355	2.876	4.981	21.293

Abbreviations: HP – Heptagon, HPY - Hexagonal pyramid, PBPY - Pentagonal bipyramid, COC - Capped octahedron, CTPR - Capped trigonal prism, JPBPY - Johnson pentagonal bipyramid, JETPY - Johnson elongated triangular pyramid

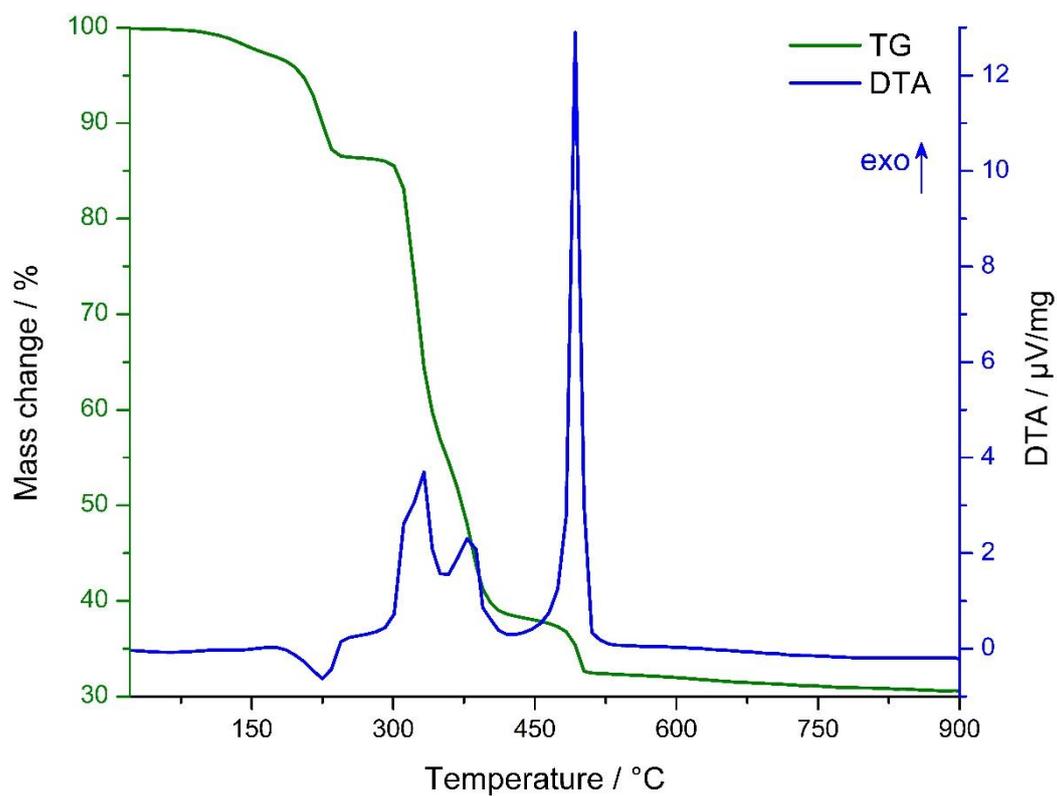


Fig. S7 Thermoanalytical curves (TG/DTA) of the **UPJS-21(Dy)** compound.

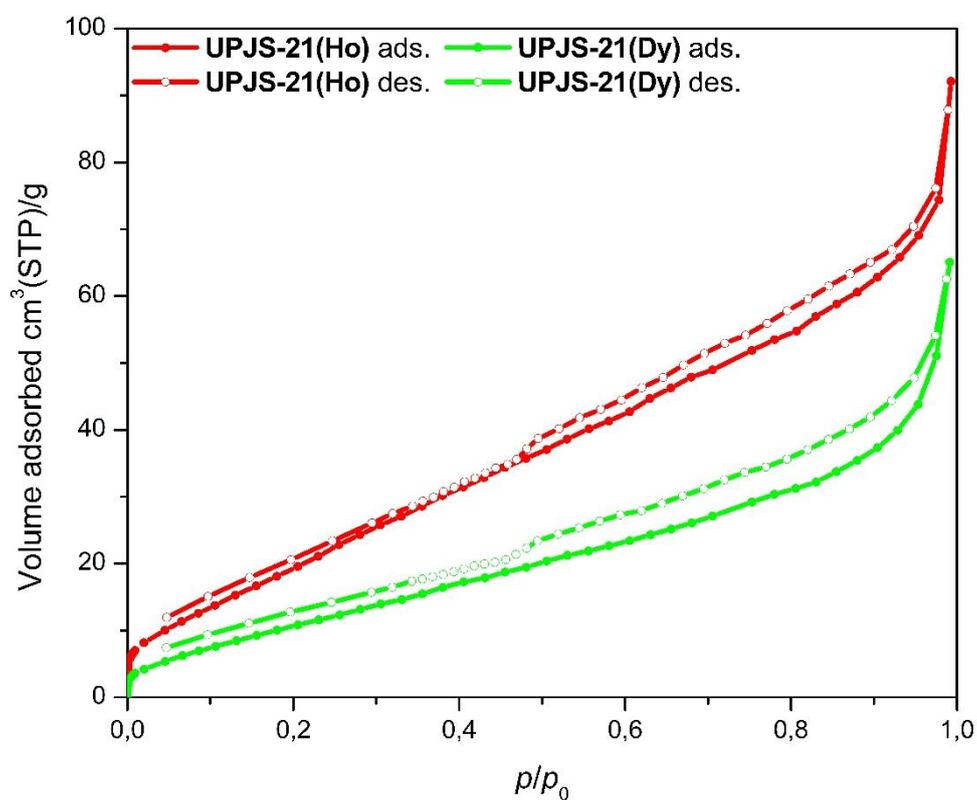


Fig. S8 N₂ adsorption/desorption isotherms of **UPJS-21(Ho)** and **UPJS-21(Dy)**.

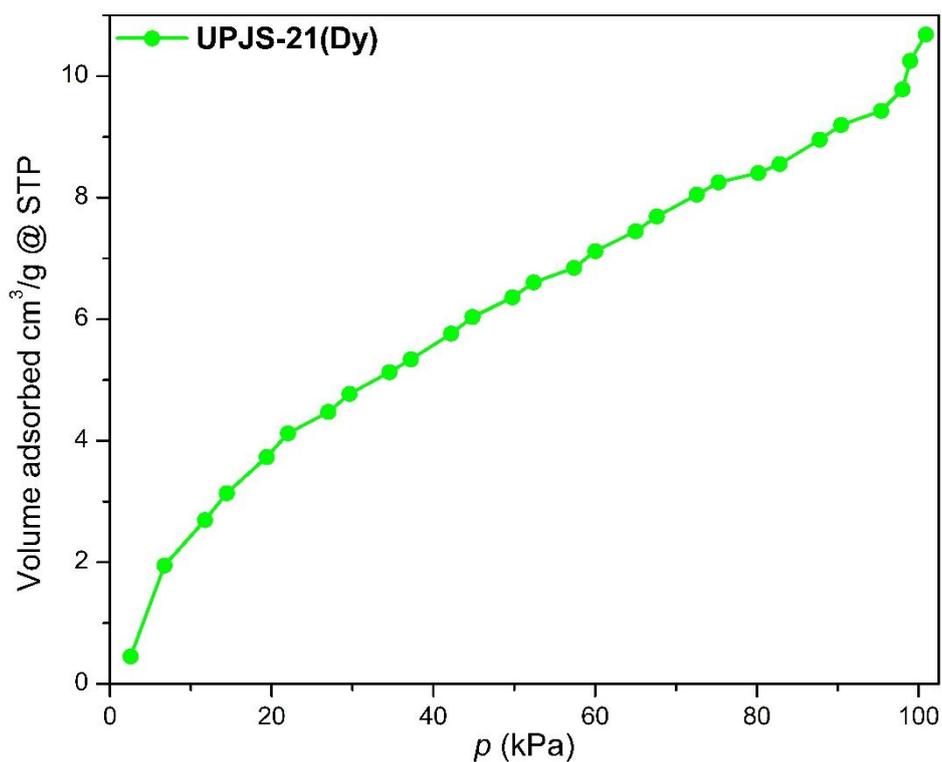


Fig. S9 CO₂ adsorption isotherms of UPJS-21(Dy).

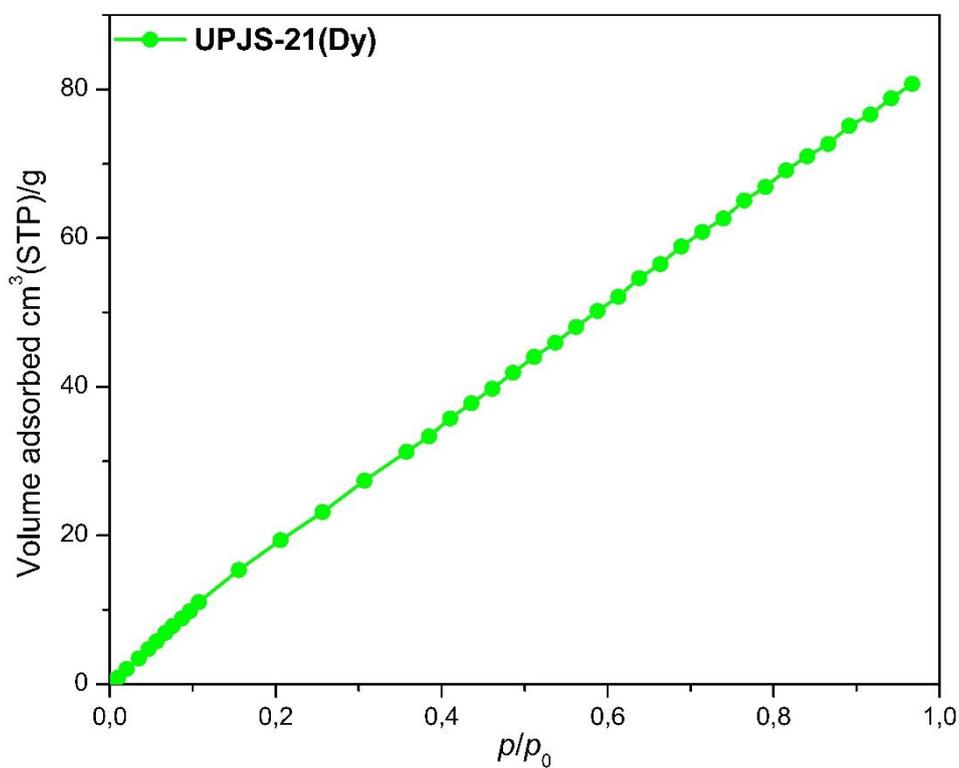


Fig. S10 H₂ adsorption isotherm of UPJS-21(Dy).

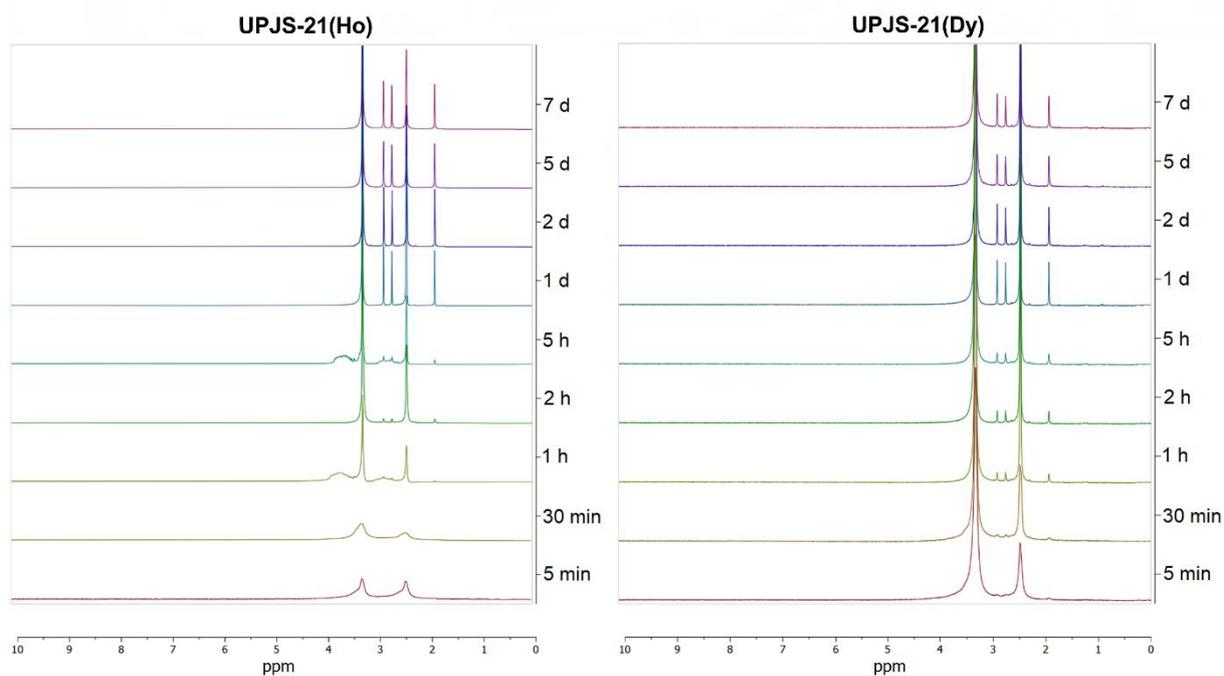


Fig. S11 Monitoring of hydrolytic stability by ^1H NMR spectra of the **UPJS-21(Ho)** (left) and **UPJS-21(Dy)** (right) complexes.

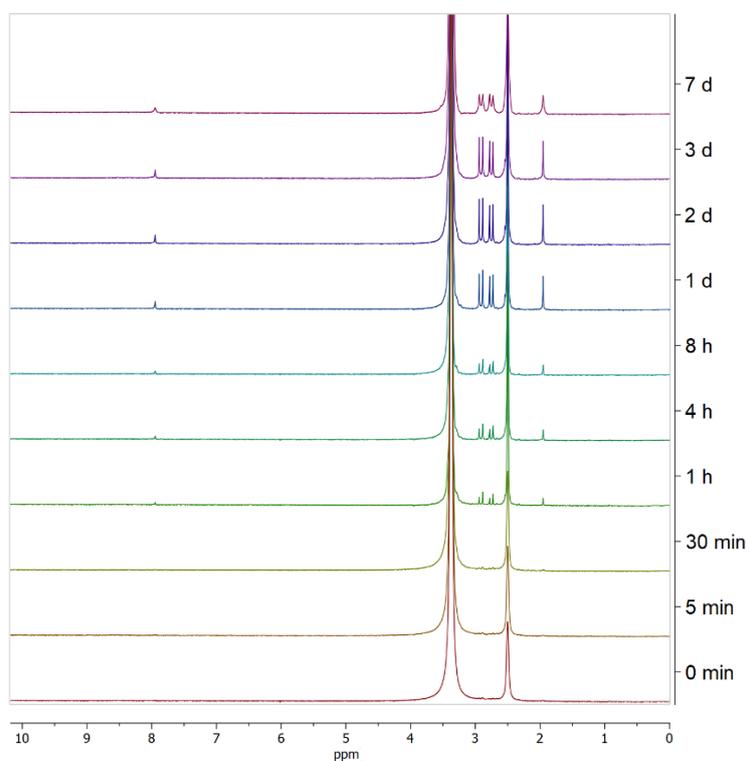


Fig. S12 Monitoring of hydrolytic stability by ^1H NMR spectra of the **UPJS-22(Yb)** complex.

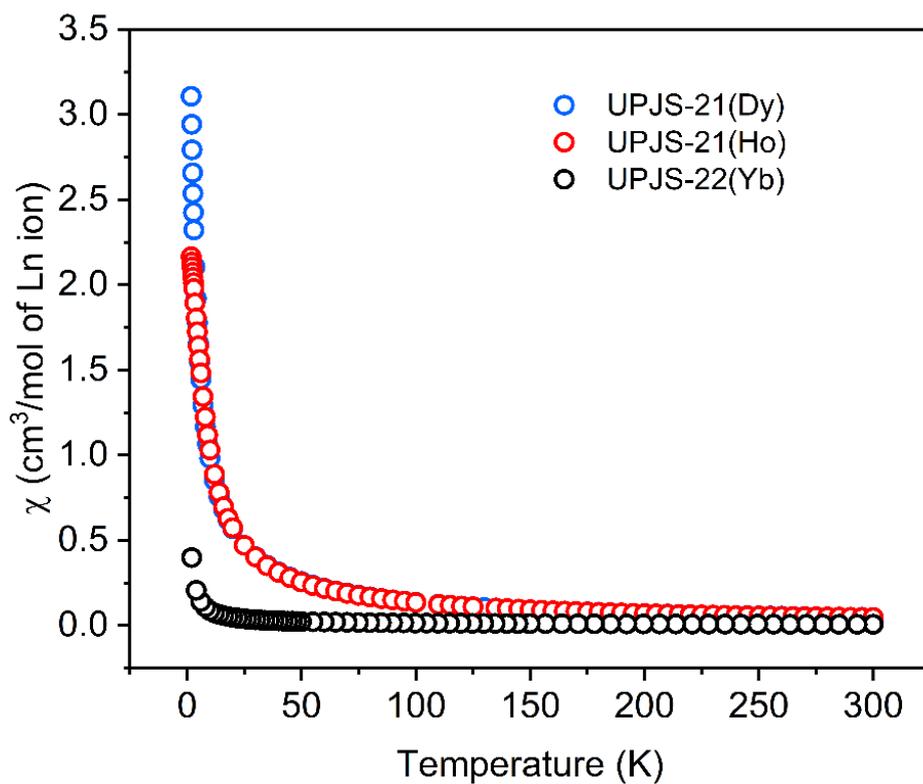


Fig. S13 Temperature dependence of the susceptibility of **UPJS-21(Dy)** (blue circles), **UPJS-21(Ho)** (red circles), and **UPJS-22(Yb)** (black circles) estimated as the ratio of the measured magnetic moment and applied magnetic field of 1 kOe.

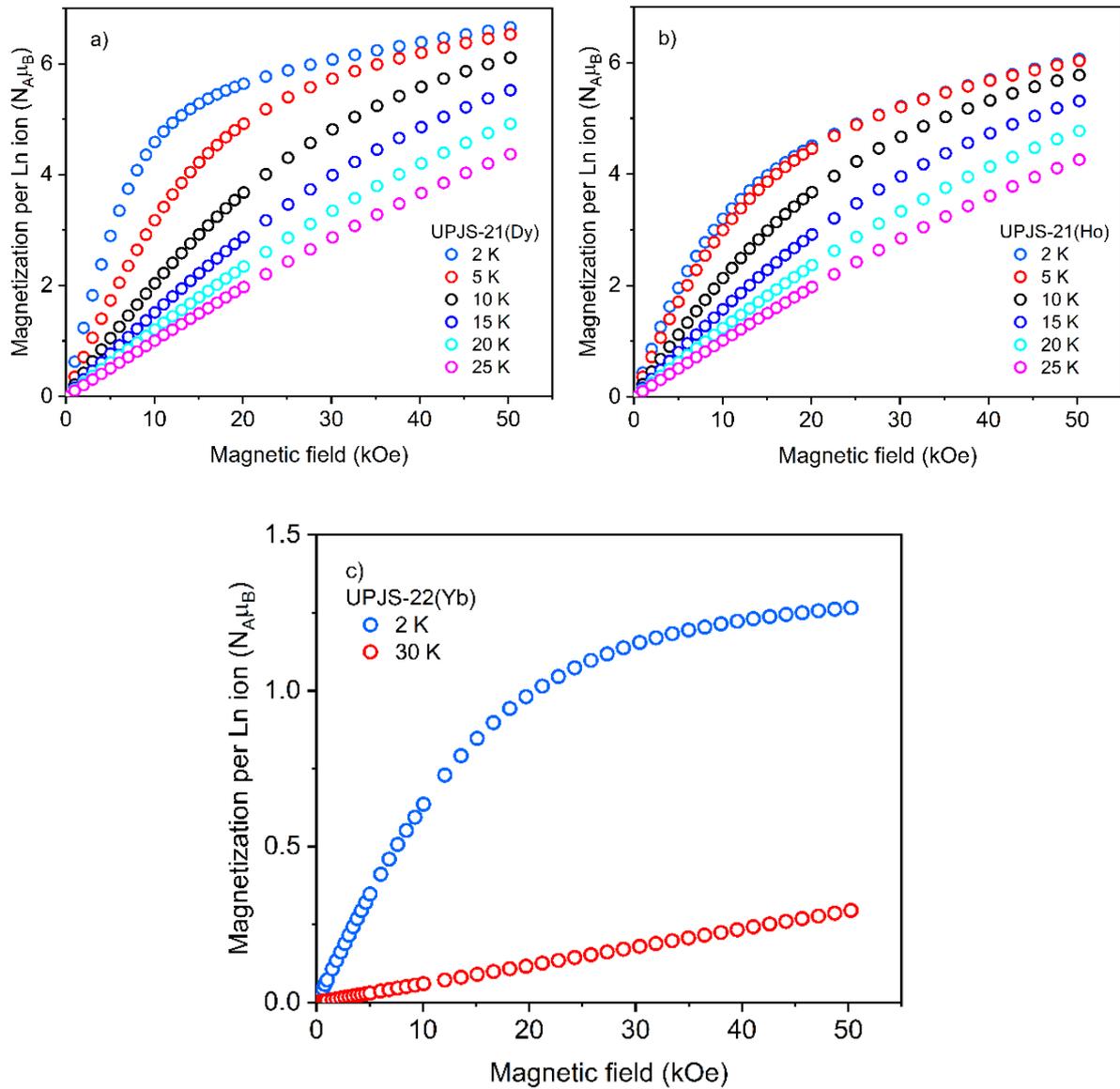


Fig. S14 Field dependence of the magnetization of **a)** UPJS-21(Dy), **b)** UPJS-21(Ho), and **c)** UPJS-22(Yb) measured at several temperatures.

Table S6 The energies of the Stark levels of the ground multiplet in cm^{-1} and their eigenstate m_J composition predicted from the CF parameter values of lanthanide ions in studied MOFs as obtained from the analysis of magnetic response.

UPJS-21(Dy)		UPJS-21(Ho)		UPJS-22(Yb)	
Energy*	$ m_J $	Energy / multiplicity	$ m_J $	Energy*	$ m_J $
0	0.11 15/2 +0.875 7/2 +1.5 1/2	0 / 1	0.05 4 +0.95 0	0	0.733 7/2 +0.267 3/2
62.98	0.915 5/2 +0.085 3/2	7.3 / 2	0.14 7 +0.16 3 +0.70 1	191.5	0.267 7/2 +0.733 3/2
175.3	0.89 15/2 +0.11 7/2	221.5 / 1	0.08 6 +0.92 2	553.6	5/2
631.3	0.087 5/2 +0.913 3/2	484.4 / 2	0.77 7 + 0.02 3 + 0.21 1	1450.6	1/2
682.6	0.955 9/2 +0.045 1/2	945.0 / 1	0.88 8 +0.12 4		
968.1	0.015 7/2 +0.045 9/2 +0.94 1/2	963.5 / 1	0.90 8 +0.08 4 +0.02 0		
1968	11/2	1083.4 / 1	0.67 6 +0.33 2		
2719	13/2	1392.9 / 1	0.92 6 +8 2		
		1747.3 / 1	+0.33 6 +0.67 2		
		1940.8 / 2	0.09 7 +0.06 5 +0.77 3 +0.08 1		
		1972.3 / 2	0.94 5 +0.05 3 +0.01 1		
		2208.8 / 1	0.12 8 +0.88 4		
		2275.4 / 1	0.10 8 +0.87 4 +0.03 0		

*multiplicity 2 for all energies

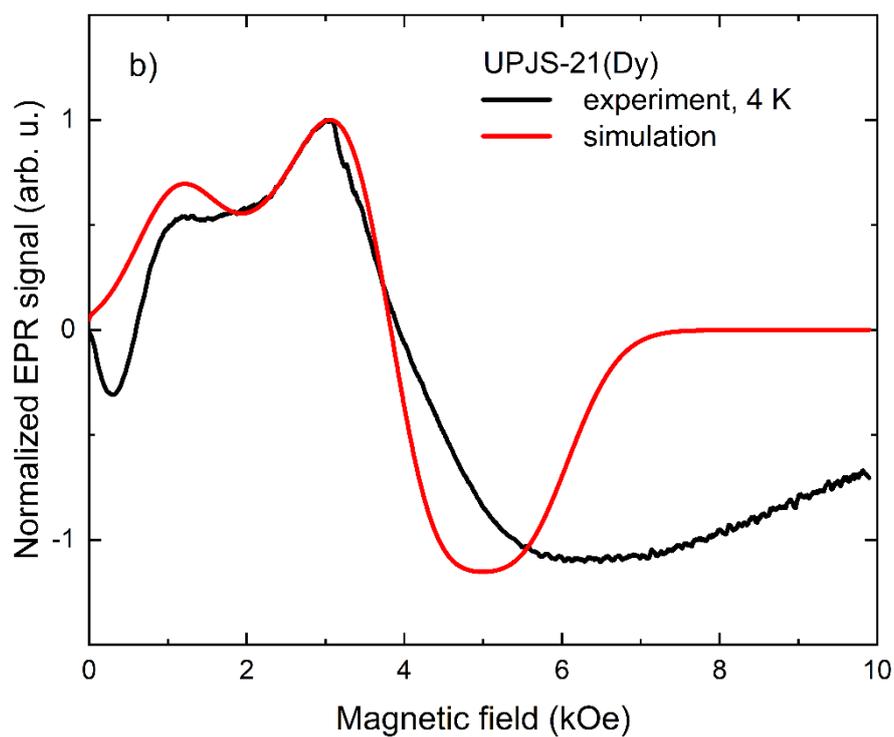
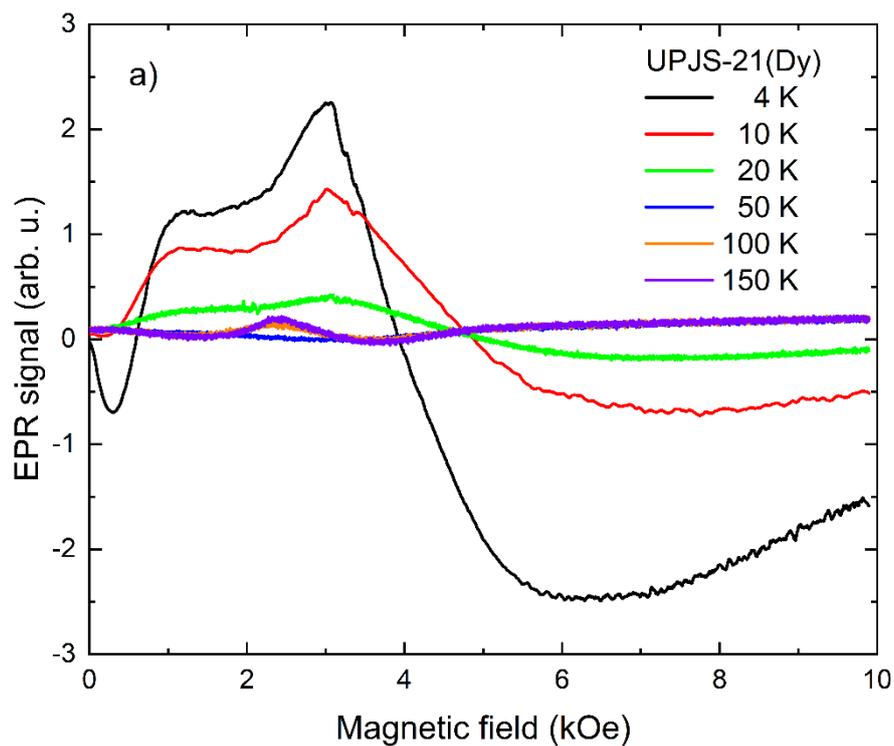


Fig. S15 **a)** Evolution of the X-band EPR spectra of **UPJS-21(Dy)** in the temperature range 4-150 K. **b)** Spectra measured at 4 K (black solid line) with the simulation (red solid line) using an effective spin $S_{\text{eff}} = 1/2$ model of the ground doublet.

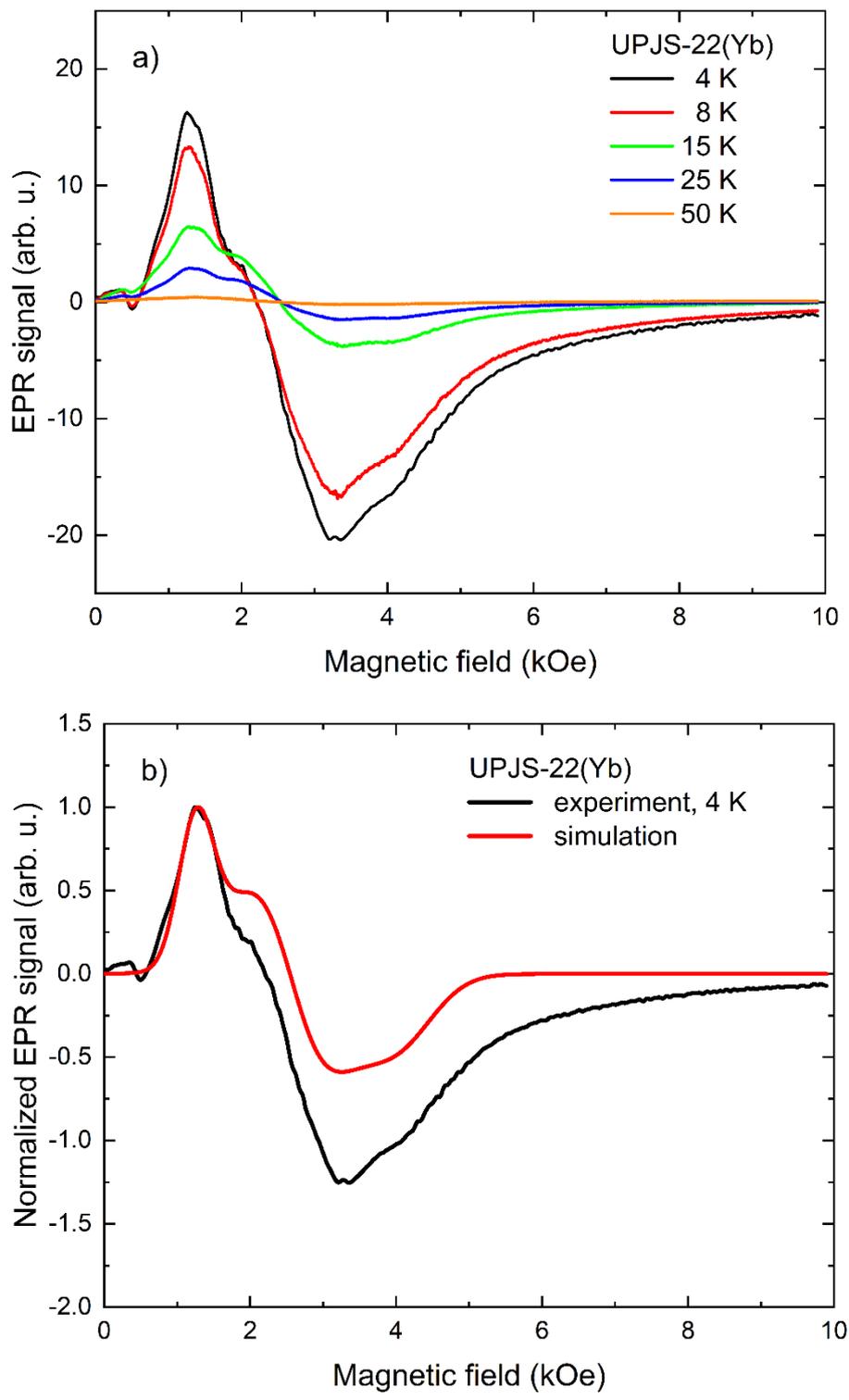


Fig. S16 **a)** Evolution of the X-band EPR spectra of **UPJS-22(Yb)** in the temperature range 4-50 K. **b)** Spectra measured at 4 K (black solid line) with the simulation (red solid line) using an effective spin $S_{\text{eff}} = 1/2$ model of the ground doublet.

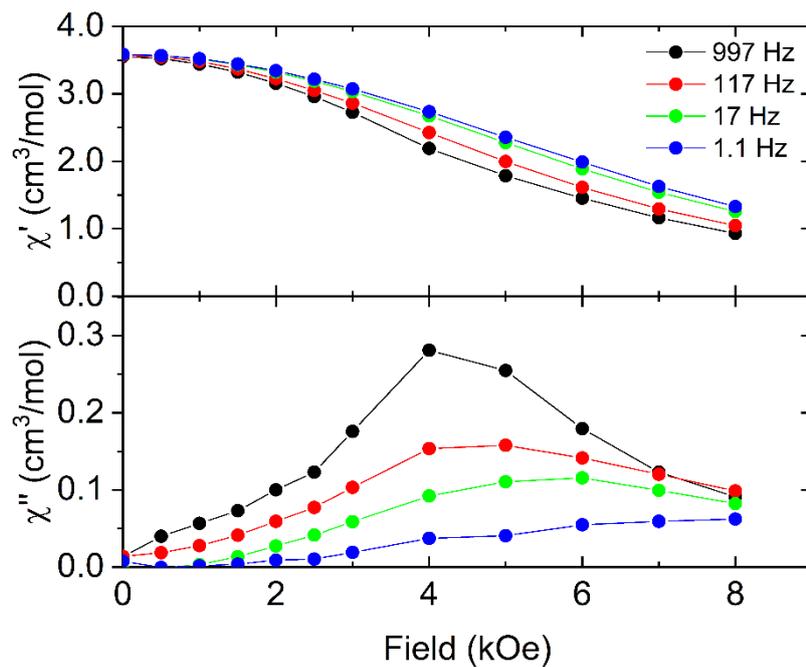


Fig. S17 Temperature dependence of the in-phase and out-of-phase component of *ac* susceptibility of **UPJS-21(Dy)** measured at 2 K in the field range from 0 to 0.8 kOe with four different frequencies of the excitation *ac* field.