## Supplementary data and figures

Compound	Eu-OA-DSTP	Tb-OA-DSTP	Eu-BDC-DSTP	Tb-BDC-DSTP
Formula	$EuC_{22}H_{13}N_3O_8S_2$	$TbC_{22}H_{13}N_{3}O_{8}S_{2} \\$	$EuC_{25}H_{19}N_3O_{10}S_2\\$	EuC25H19N3O10S2
Formula weight	633.43	670.39	737.51	744.47
Crystal color	yellow	yellow	yellow	yellow
Dimensions/mm <sup>3</sup>	0.240×0.050×0.025	0.135×0.027×0.021	0.088×0.037×0.010	0.144×0.092×0.040
Crystal system	monoclinic	monoclinic	triclinic	triclinic
Space group	C2/c	C2/c	Pī	Pī
a/Å	20.080(6)	19.929(7)	10.826(4)	10.803(3)
b/Å	18.657(5)	18.593(6)	12.044(4)	12.027(3)
c/Å	13.864(4)	13.833(5)	12.046(4)	12.040(3)
$\alpha / ^{0}$	90	90	109.677(3)	109.6780(10)
$\beta^{\prime 0}$	117.570(4)	117.925(4)	103.108(2)	103.133(3)
$\gamma^{\prime 0}$	90	90	109.545(2)	109.48
$V/Å^3$	4604(2)	4529(3)	1287.7(7)	1283.1
Z	8	8	2	2
$D_{\text{calcd}}/\text{g cm}^{-3}$	1.914	1.967	1.902	1.927
<i>F</i> (000)	2600	2616	730	734
$\mu/\text{mm}^{-1}$	2.962	3.364	2.663	2.985
$\theta$ for data collection/ <sup>0</sup>	2.19 to 27.51	2.19 to 27.51	2.01 to 27.47	2.01 to 27.48
Reflections collected	5094	5097	5571	5444
Unique reflections/R(int)	4258/0.0524	4491/0.0563	5053/0.0426	4976/0.0421
Parameters	352	325	371	371
GOF	1.027	1.044	1.067	1.036
<i>R</i> 1, <i>wR</i> 2 ( $I > 2\sigma(I)$ )	0.0535, 0.1395	0.0591, 0.1440	0.0359, 0.1069	0.0339, 0.1029
R1, wR2 (all data)	0.0644, 0.1471	0.0698, 0.1585	0.0442, 0.1543	0.0441, 0.1623

Table S1 Crystallographic data for the four compounds <sup>a</sup>

<sup>*a*</sup>  $RI = \sum (|F_{o}| - |F_{c}|) / \sum |F_{o}|, wR2 = [\sum w(F_{o}^{2} - F_{c}^{2})^{2} / \sum w(F_{o}^{2})^{2}]^{0.5}$ 

**Table S2** The molar ratio of  $Tb_xEu_{1-x}$ -OA-DSTP and  $Tb_xEu_{1-x}$ -BDC-DSTP calculated by Inductively coupled plasma (ICP) analysis.

Sample	The molar ratio of the	The Tb/Eu ratio calculated	
	starting Tb/Eu salt	by ICP analysis	
Tb <sub>0.99</sub> Eu <sub>0.01</sub> -OA-DSTP	0.99:0.01	0.9873:0.0125	
Tb <sub>0.99</sub> Eu <sub>0.01</sub> -BDC-DSTP	0.99:0.01	0.9915:0.0096	
Tb <sub>0.98</sub> Eu <sub>0.02</sub> -OA-DSTP	0.98:0.02	0.9792:0.0193	
Tb <sub>0.98</sub> Eu <sub>0.02</sub> -BDC-DSTP	0.98:0.02	0.9814:0.0208	



**Fig. S1** <sup>1</sup>H NMR (DMSO) of 2,4-(2,2':6',2"-terpyridin-4'-yl)-benzenedisulfonic acid.



Fig. S2 Powder X-ray diffraction (PXRD) patterns.



**Fig. S3** The Commission International d'Eclairage (CIE) chromaticity diagram showing the luminescence color of Tb<sub>0.98</sub>Eu<sub>0.02</sub>-OA-DSTP (a) and Tb<sub>0.98</sub>Eu<sub>0.02</sub>-BDC-DSTP (b) at different temperature.



**Fig. S4** The ratiometric temperature-sensing properties of  $Tb_{0.99}Eu_{0.01}$ -OA-DSTP: (a) emission spectra for the solid sample recorded between 77 and 400K upon excitation at 360nm, inset: normalized emission intensities of  ${}^{5}D_{4} \rightarrow {}^{7}F_{5}$  transition of  $Tb^{3+}$  (546nm) and  ${}^{5}D_{0} \rightarrow {}^{7}F_{4}$  transition of  $Eu^{3+}$  (696nm); (b) temperature-dependent intensity ratio ( $\Delta/\Delta_{77k}$ ) and linearly fitted curve; (c) the natural logarithm of the thermometric parameter  $\Delta$  changing with the inverse of temperature and the linearly fitted curve.



**Fig. S5** The ratiometric temperature-sensing properties of  $Tb_{0.99}Eu_{0.01}$ -BDC-DSTP: (a) emission spectra for the solid sample recorded between 77 and 400K upon excitation at 360nm, inset: normalized emission intensities of  ${}^{5}D_{4} \rightarrow {}^{7}F_{5}$  transition of  $Tb^{3+}$  (546nm) and  ${}^{5}D_{0} \rightarrow {}^{7}F_{4}$  transition of  $Eu^{3+}$  (702nm); (b) temperature-dependent intensity ratio ( $\Delta/\Delta_{77k}$ ) and linearly fitted curve; (c) the natural logarithm of the thermometric parameter  $\Delta$  changing with the inverse of temperature and the linearly fitted curve.



**Fig. S6** Relative sensitivity of the thermometers  $Tb_{1-x}Eu_x$ -OA-DSTP and  $Tb_{1-x}Eu_x$ -BDC-DSTP (x = 0.01, 0.02).



Fig. S7 The comparison of lifetimes of  $Tb^{3+}$  (a) and  $Eu^{3+}$  (b) in the pure MOFs and Tb/Eu-codoping MOFs.



Fig. S8 The emission spectra of  $Tb_{0.98}Eu_{0.02}$ -OA-DSTP (a) and  $Tb_{0.98}Eu_{0.02}$ -BDC-DSTP (b) upon excitation of 488nm at room temperature.



Fig. S9 Phosphorescence spectrum of Gd-OA-DSTP in the solid state at 77K.