1. Chemicals and procedures:

The solvent acetonitrile was dried with calcium hydride and redistilled. Erbium and Neodymium nitrates were obtained from the corresponding oxides Er₂O₃ (99.99%) and Nd₂O₃ (99.99%).

TPY-Si was prepared as follows: A mixture of MPS (1.7mmol), ATP (1.7mmol), 2,2'-azobisisobutyronitrile (AIBN, 0.05mmol) and acetonitrile (15mL) were heated to 80 ℃ for 6 h under nitrogen. Then the solvent was removed in vacuo and the pasty residue was washed with cold hexane, then a clear oil of TPY-Si was obtained.

The hybrid materials were prepared as follows: TPY-Si (0.50g, 1.0mmol) was dissolved in 2mL of DMF, then TEOS and H₂O were added to it with the molar ratio 1:4:16 (TPY-Si:TEOS:H₂O) under stirring. The Er(NO₃)₃ (Nd(NO₃)₃) and DBM solution of DMF were added to the resulting mixture, the mole ratio of TPY-Si/Ln³+/DBM was 1:1:3. The mixture was agitated magnetically to achieve a single phase and then transferred into a cuvette. Thermal curing was performed at 60 ℃ and continued until the sample solidified. After 5 days, the samples were dried for 24 h at 100 ℃ and collected as monolithic bulks and ground as powder materials for the photophysical studies.

2. The spectra of TPY-Si and hybrid materials

List of Figures

**Fig. 1** FT-IR spectra for ATP (A), TPY-Si (B), Er(DBM)₃TPY-Si (C) and Nd(DBM)₃TPY-Si (D).

**Fig. 2** ¹H NMR spectrum of TPY-Si (DMSO-<i>d₆</i>).

**Fig. 3** Mass spectrum of TPY-Si.

**Fig. 4** UV-Vis spectra of ATP and DBM.

**Fig. 5** DR spectra of Er(DBM)₃TPY-Si and Nd(DBM)₃TPY-Si
**Fig. 1** FT-IR spectra for ATP (A), TPY-Si (B), Er(DBM)$_3$TPY-Si (C) and Nd(DBM)$_3$TPY-Si (D).

**Fig. 2** $^1$H NMR spectrum of TPY-Si (DMSO-$d_6$).
Fig. 3 Mass spectrum of TPY-Si.

Fig. 4 UV-Vis spectra of ATP and DBM.
Fig. 5 DR spectra of Er(DBM)$_3$TPY-Si and Nd(DBM)$_3$TPY-Si