

Electronic Supplementary Information (ESI):

**Hydrothermal Approach and Luminescent Properties for
Synthesis of Orthoniobates $\text{GdNbO}_4:\text{Ln}^{3+}$ ($\text{Ln} = \text{Dy, Eu}$)
Single Crystals under High-Temperature High-Pressure
Conditions**

Min Yang^a, Xudong Zhao^{a*}, Ying Ji^a, Fuyang Liu^a, Wei Liu^a, Jiayin Sun^b, Xiaoyang Liu^{a*}

a State Key Laboratory of Inorganic Synthesis and Preparative Chemistry
Jilin University. 2699 Qianjin Street, Changchun.

b School of Chemistry & Chemical Engineering, Anqing Normal University, Anqing,
Anhui 246011, China.

* E-mail: liuxy@jlu.edu.cn;



Fig. S1 Model HR-1B-2, LECO hydrothermal synthesis reactor

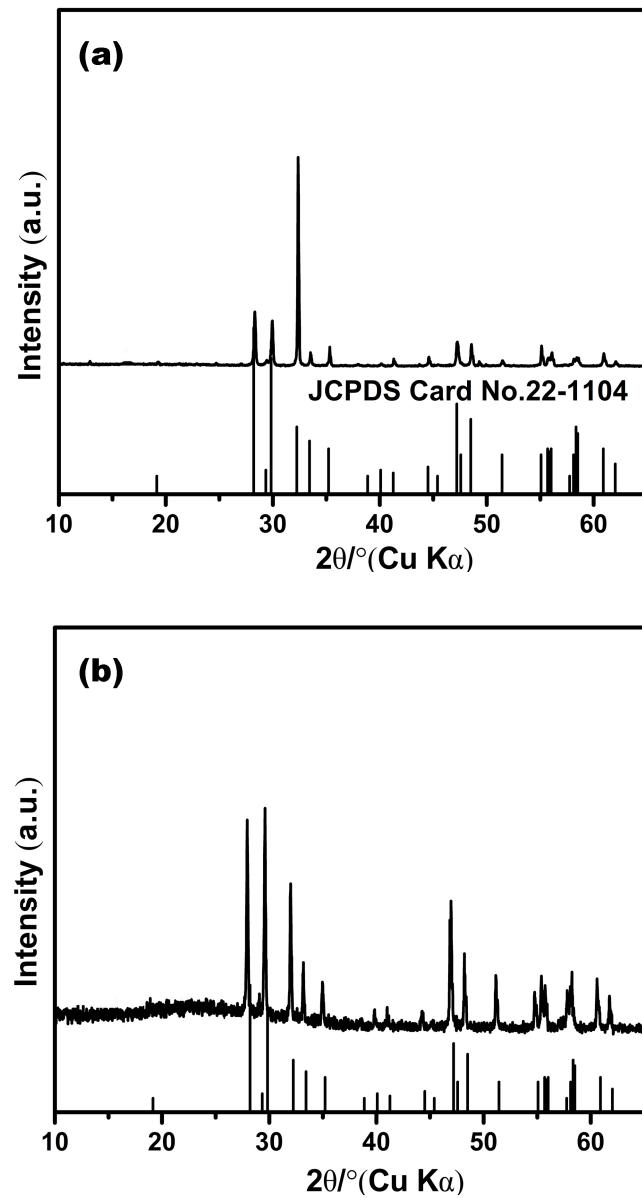


Fig. S2. (a) Power XRD pattern of the as-synthesized Dy³⁺ doped GdNbO₄; (b) Power XRD pattern of the as-synthesized Eu³⁺ doped GdNbO₄.

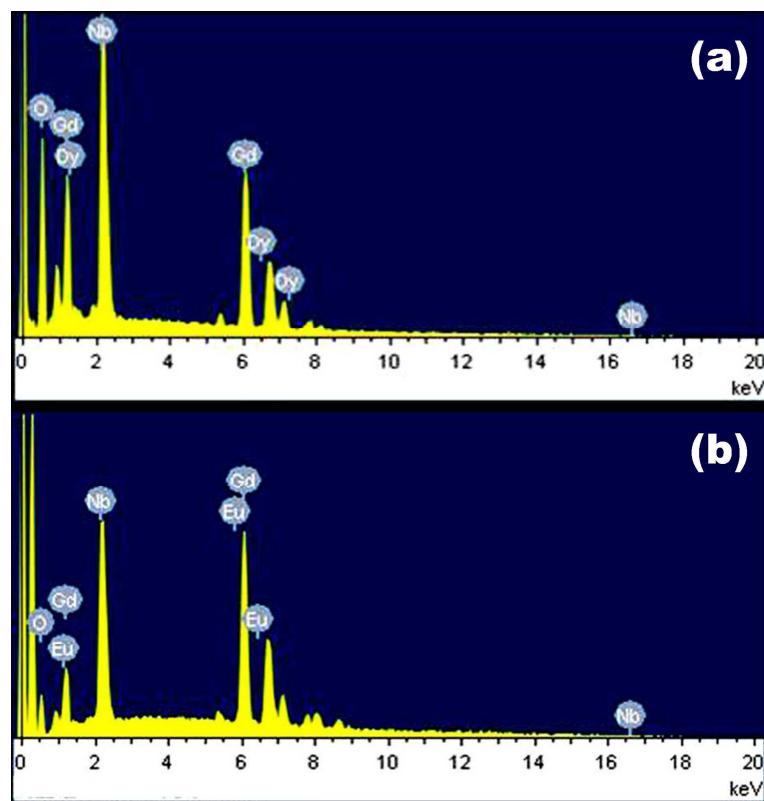


Fig. S3 (a) EDS spectrum of the as-synthesized Dy³⁺ doped GdNbO₄. (b) EDS spectrum of the as-synthesized Eu³⁺ doped GdNbO₄.

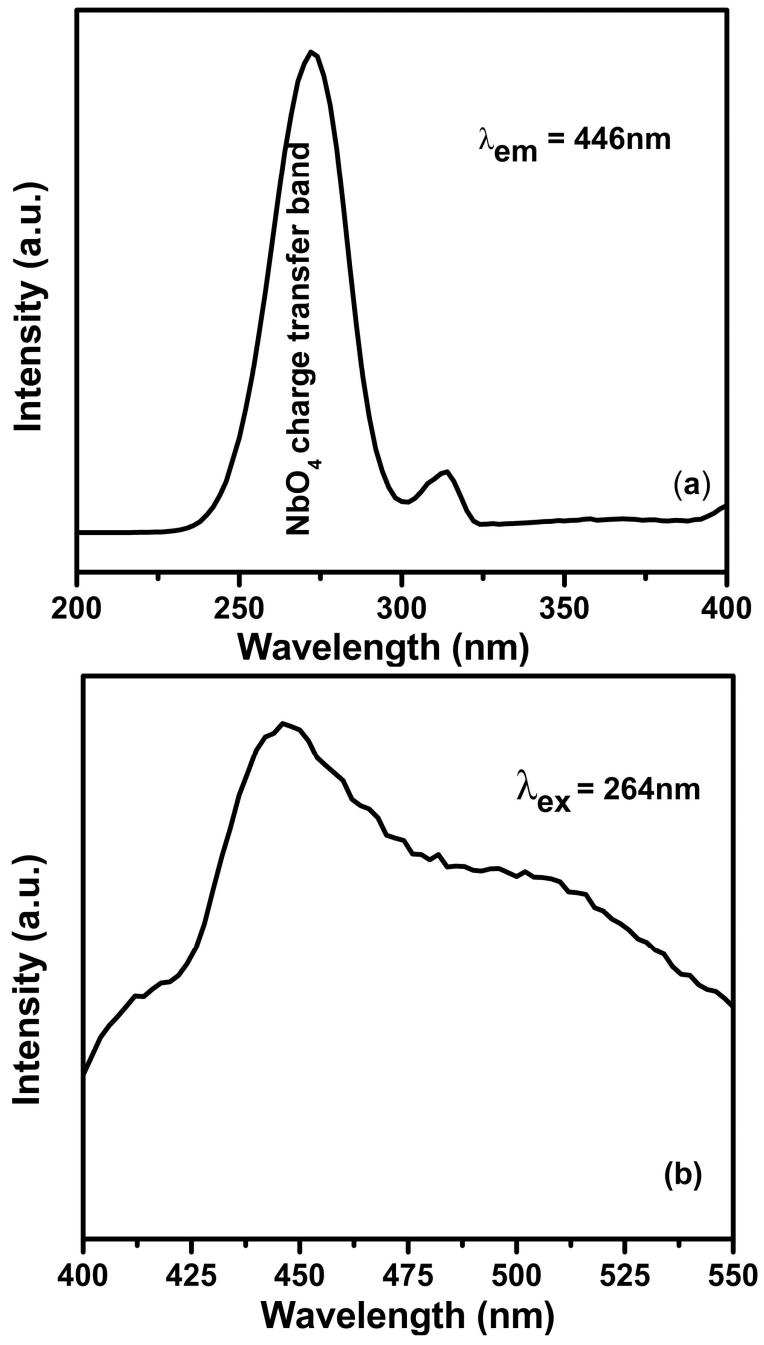


Fig. S4 (a) Excitation (a) and emission (b) spectra of GdNbO₄.

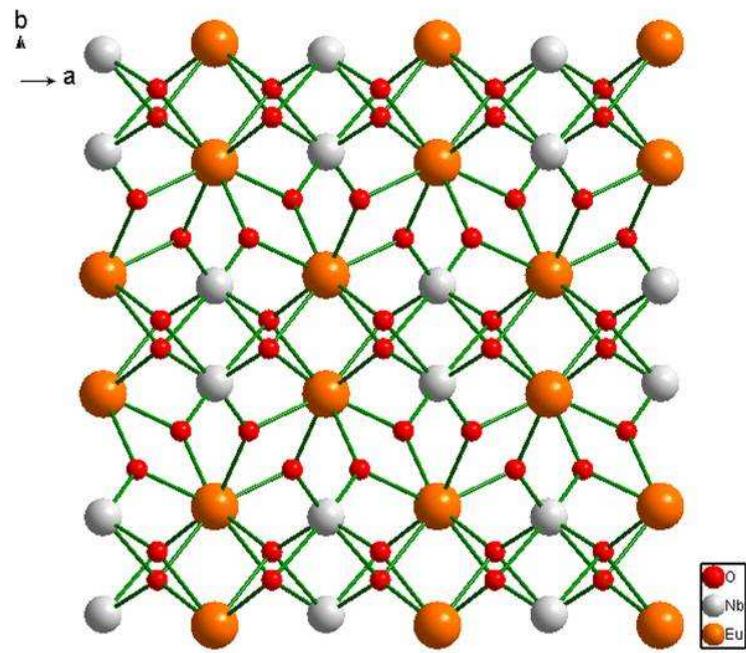


Fig. S5. Crystal structure of Eu³⁺ doped GdNbO₄ showing coordination geometry of the GdO₈ polyhedron and NbO₆ polyhedron therein and 3D packing viewed along the c axis. Orange, white and red spheres represent Eu, Nb and O atoms, respectively.

Table S1. Crystallographic Data for Gd(OH)₃

| | |
|--|-----------------------------------|
| Chemical formula | Gd(OH) ₃ |
| Formula weight (g/mol) | 205.25 |
| Temperature (K) | 296(2) |
| Wavelength (nm) | 0.071073 (Mo) |
| Crystal system | hexagonal |
| | $a = 0.6350$ (12) |
| Unit cell dimensions/nm | $b = 0.6350$ (12) |
| | $c = 0.3642$ (15) |
| Cell volume (nm ³), Z | 0.1272 (6), 2 |
| Density (g/cm ³), calculated | 5.360 |
| Crystal size (mm) | 0.1×0.030×0.030 |
| Θ range for data collection | 3.71-24.61 ° |
| Goodness-of-fit on F^2 | 1.263 |
| Final R indices [$I > 2\sigma(I)$] | $R_1 = 0.0199$, $wR_2 = 0.0467$ |
| R indices (all data) | $R_1 = 0.0201$, $wR_2 = 0.04681$ |
| Extinction coefficient | 0.33(3) |
| Largest diff. peak and hole/(e·nm ⁻³) | 1.346 and -0.996 |

Table S2. Crystallographic Data for Dy³⁺, Eu³⁺ doped GdNbO₄

| Chemical formula | GdNbO ₄ :Dy ³⁺ | GdNbO ₄ :Eu ³⁺ |
|--|--|---|
| Formula weight (g/mol) | 314.16 | 308.87 |
| Temperature (K) | 296(2) | 296(2) |
| Wavelength (nm) | 0.071073 (Mo) | 0.071073 (Mo) |
| Crystal system space group | monoclinic, <i>C2/c</i> | Monoclinic <i>C2/c</i> |
| Unit cell dimensions/nm | $a = 0.7160(5)$ $b = 1.1170(10)$ $c = 0.5139(4)$ | $a = 0.7062(2)$ $b = 1.1019(3)$ $c = 0.5063(4)$ |
| Cell volume (nm ³), <i>Z</i> | 0.3095(4), <i>Z</i> = 4 | 0.2956 (8), 4 |
| Density (g/cm ³) (calculated) | 6.743 | 6.940 |
| Crystal size (mm) | 0.05×0.03×0.03 | 0.08×0.050×0.050 |
| Θ range for data collection | 3.70-25.00 ° | 3.70-25.00 ° |
| Goodness-of-fit on F^2 | 1.204 | 1.115 |
| Final <i>R</i> indices [$I > 2\sigma(I)$] | $R_1 = 0.0548$, $wR_2 = 0.1490$ | $R_1 = 0.0515$, $wR_2 = 0.1604$ |
| <i>R</i> indices (all data) | $R_1 = 0.0549$, $wR_2 = 0.1492$ | $R_1 = 0.0515$, $wR_2 = 0.1604$ |
| Extinction coefficient | 0.024(2) | 0.034(6) |
| Largest diff. peak and hole/(e·nm ⁻³) | 3.201 and -3576 | 3.201 and -3576 |