Two-step synthetic route to GdOF:Ln$^{3+}$ Nanocrystals with multicolor Luminescence Properties

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Figure S1. TG curve (A), SEM image (B), and XRD pattern (C) of the LGdH precursor.

The TG curve shows three distinct mass losses, which is consistent with the results of other layered hydroxide systems. The initial mass loss of 3.4% before 150 °C ascribes to the removal of the cointercalated water from the interlayer galleries. The following mass loss of 10.05 % at 290°C corresponds to the partial decomposition of the layers to Gd$_2$O$_2$(OH)NO$_3$. The complete decomposition to Gd$_2$O$_3$ at 490 °C leads to a further massage loss of 11.73%. The observed whole mass loss of 24% is consistent with the calculated mass loss value (24.12%) of
Gd$_2$(OH)$_5$NO$_3$·0.9H$_2$O.

**Figure S2.** The XRD pattern (A) and AFM image (B) of sample with reaction time of 5min, inset in panel B is the corresponding height profile of the nanosheet.

**Figure S3.** The XRD pattern (A) and SEM image (B) of the sample with reaction time of 10 min at ambient temperature.
**Figure S4** The detailed structure resemblances between the Gd$^{3+}$ atomic arrangements in the Gd(OH)$_2$F (0001) and GdOF (110) planes, respectively.

Structurally, the Gd$^{3+}$ atomic arrangements in the Gd(OH)$_2$F (0001) and GdOF (110) planes are topologically similar. On the one hand, the angles between the Gd$^{3+}$ atomic arrangements along the three basic crystallographic directions in the Gd(OH)$_2$F (0001) plane is approximate to those in the GdOF (110) plane. In the Gd(OH)$_2$F (0001) plane, the angles among the three equivalent directions are 60° ($\angle[\overline{2}\overline{1}0] \& [\overline{1}120]$) and 120° ($\angle[\overline{2}\overline{1}0] \& [\overline{1}2\overline{1}0]$), respectively. Meanwhile, the counterpart angles in the GdOF (110) plane are 55° ($\angle[\overline{2}\overline{2}1] \& [\overline{1}\overline{1}1]$) and 128° ($\angle[\overline{2}\overline{2}1] \& [\overline{1}\overline{1}0]$). On the other hand, the Gd$^{3+}$ atomic arrangements along the three basic directions in the Gd(OH)$_2$F (0001) and GdOF (110) planes are all analogous screw-axis structures. In the Gd(OH)$_2$F (0001) lattice plane, the edge-edge-sharing Gd(O/F)$_9$ polyhedra constitute zigzag chains with a screw-axis structure along the three equivalent directions. While, the the edge-edge-sharing Gd(O/F)$_8$ polyhedra show a similar arrangement along the counterpart directions in the GdOF (110) lattice plane despite that they intuitively exhibit line chains along the $[\overline{1}\overline{1}1]$ and the $[\overline{1}\overline{1}0]$ directions when viewed along [110] direction.

![XRD pattern](image)

**Figure S5** XRD pattern of the final product when the NH$_4$F feeding amount was increased to 2 mmol.
**Figure S5** Schematic energy level diagrams for the UC emissions of GdOF:0.19Yb$^{3+}$,0.01Er$^{3+}$ and GdOF:0.05Yb$^{3+}$,0.01Tm$^{3+}$ samples under 980 nm laser.