

## Supplementary Information

### Terbium-Doped Gadolinium Oxide Nanoparticles Prepared by Laser Ablation in Liquid for Use as Fluorescence and Magnetic Resonance Imaging Dual-Modal Contrast Agents

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#### 1. Preparation of targets.

Gd<sub>2</sub>O<sub>3</sub> and Tb<sub>4</sub>O<sub>7</sub> of analytical grade were first milled in an agate mortar for 30min and 7wt% polyvinyl alcohol (PVA) was added. After dried under 50°C for 10min and sieved with a 60 mesh filter, the powders were pressed into cylinders of diameter 15mm under 15Mpa and finally annealed for 10h at 1500°C with a heating rate of 10°C/min.

#### 2. XRD pattern of targets.

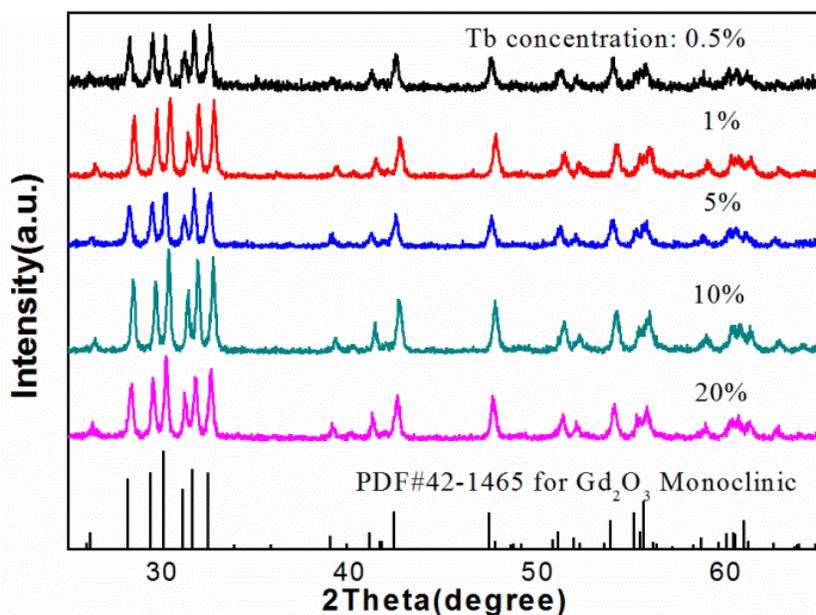
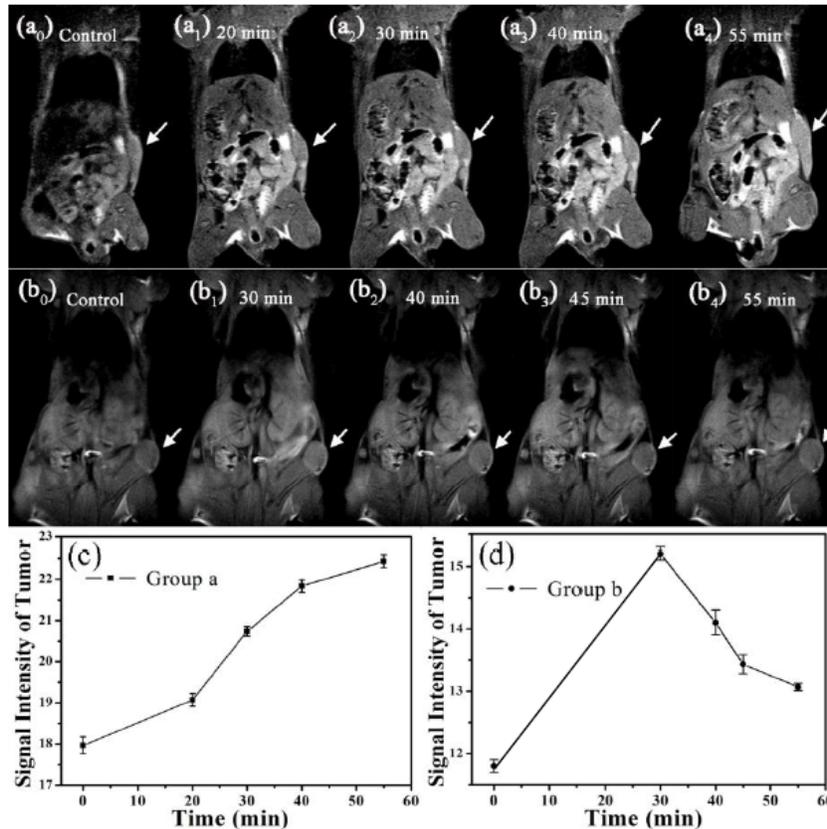


Fig. S1. XRD pattern of 0.5%, 1%, 5%, 10%, 20% Tb doped Gd<sub>2</sub>O<sub>3</sub> bulks compared to PDF#42-1465 of monoclinic Gd<sub>2</sub>O<sub>3</sub>

### 3. Animal modal and dynamic contrast-enhanced T1-weighted MR imaging

Animal experiments were performed according to the National Institutes of Health guidelines on the rules of animal's research. Four to six week old Balb/c nude mice were obtained from the animal experiment center of Medical College, Sun Yat-sen University, China and were subcutaneously injected with NPC CNE1 cells( $5 \times 10^6$  in  $100 \mu\text{L}$  PBS). And mice were all maintained in a specific pathogen-free (SPF) environment (Certificate No. 26-99S031) and randomized by approximately  $6 \text{ mm}^3$  tumor after 8 days.

The signal intensity of the tumors is very complex and related with injected dose and physiological status of experimental animals. Every animal has itself metabolic cycle for same dose of injected nanoparticles, resulting in the difference of the time when the signal intensity reach the highest after the injection. The grey values of tumors are collected as signal intensity. As shown in the dynamic enhancement curves Figure S2 (c, d), the signal intensity increase with measurement time for group a measurement, while it increase to a maximum value at 30 minutes after injected and decrease with measurement time for b group, which means the metabolism of  $\text{Gd}_2\text{O}_3:\text{Tb}$  nanoparticles has occurred for the mice in group b, resulting decreasing of signal intensity at 30 minutes after injected.



**Fig. S2.** Gray scale images of BALB/c nude mice with NPC CNE-1 xenografted tumor ( $a_0$ ), ( $b_0$ ) control group ( $\text{Gd}_2\text{O}_3:\text{Tb}$  NPs injection free ) and ( $a_1$ ) 20, ( $a_2$ ) 30, ( $a_3$ ) 40, ( $a_4$ ) 55, ( $b_1$ ) 30, ( $b_2$ ) 40, ( $b_3$ ) 45, ( $b_4$ ) 55 minutes after injection of  $\text{Gd}_2\text{O}_3:\text{Tb}$  NPs ( $15 \mu\text{mol}/\text{kg}$ ). (c) and (d) dynamic enhancement curve of xenografted tumors.