

## Supplementary Information

### 1.1 General

HPLC separation was performed on the PET-MF-2 V-IT-I synthesizer module (PET Co. Ltd., Beijing, China) built-in HPLC system with an S1021 pump (SYKAM Chromatography Vertriebs GmbH, Bayern, Germany), Well-ChromK-2001 UV detector (KNAUER Wissenschaftliche Geräte GmbH, Berlin, Germany), radioisotope detectors, and an Agilent ZORBAX SB-C18 9.4×250mm 5 $\mu$  semi-preparative column. The mobile phase was 60% solvent A (0.1% TFA in water): 40% solvent B (0.1% TFA in MeCN) with a flow rate of 4 mL/min. Analytical HPLC was performed using a LC-20AD HPLC system (Shimadzu, Japan) equipped with a ZORBAX Eclipse XDB-C18 analytic column (4.6  $\times$  150 mm, 5  $\mu$ m; Agilent Technologies, USA) using a flow rate of 1 mL/min. The gradient program started from 95% solvent A (0.1% TFA in water) and 5% solvent B (0.1% TFA in MeCN) (0–2 min) and ramped to 20% solvent A and 80% solvent B at 25 min. The elution profile was detected with an ultraviolet detector (Agilent interface 35900E, Agilent Technologies, USA) at 210 nm and a B-FC-3200 high energy PMT Detector (Bioscan Inc, Washington DC, USA). NMR spectra were recorded on a Bruker 400 Nanobay spectrometer operating at 400 MHz and 100 MHz for  $^1\text{H}$  and  $^{13}\text{C}$  acquisitions, respectively. Chemical shifts were referenced to the residual proton solvent peaks ( $^1\text{H}$ :  $\text{CDCl}_3$ ,  $\delta$  7.26), solvent  $^{13}\text{C}$  signals ( $\text{CDCl}_3$ ,  $\delta$  77.16). High-resolution mass spectra were recorded with a ThermoFisher Scientific Orbitrap Fusion mass spectrometer.

### 1.2 Spectroscopic Data

Figure S1.  $^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of 4-nitrophenyl-2-fluoropropionate (NFP)

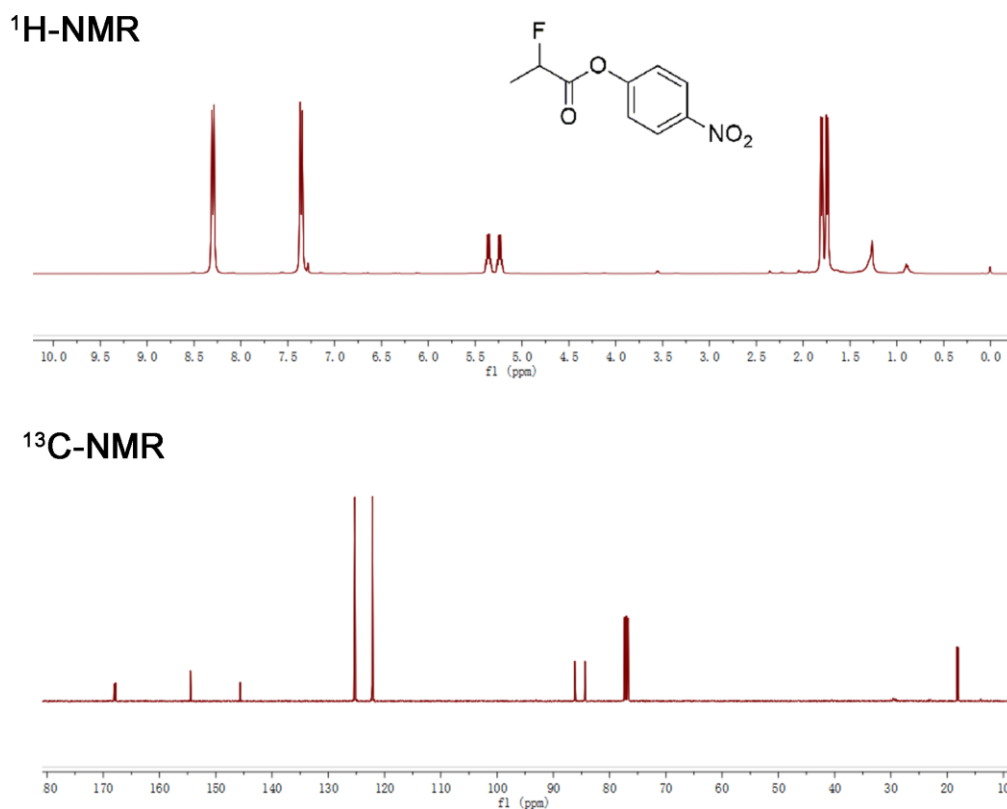
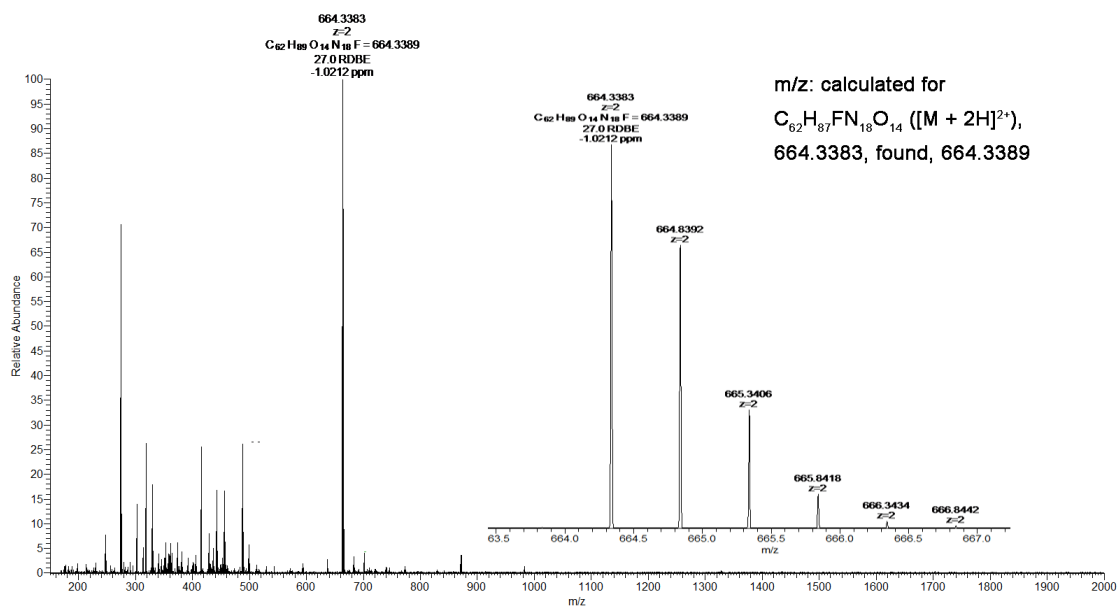


Figure S2. Mass spectra of FP-D-Lys<sup>6</sup>-GnRH



### 1.3 Chromatography Data

Figure S3. Semi-preparative radio-high-performance liquid chromatography

(Radio-HPLC) of [ $^{18}\text{F}$ ]NFP

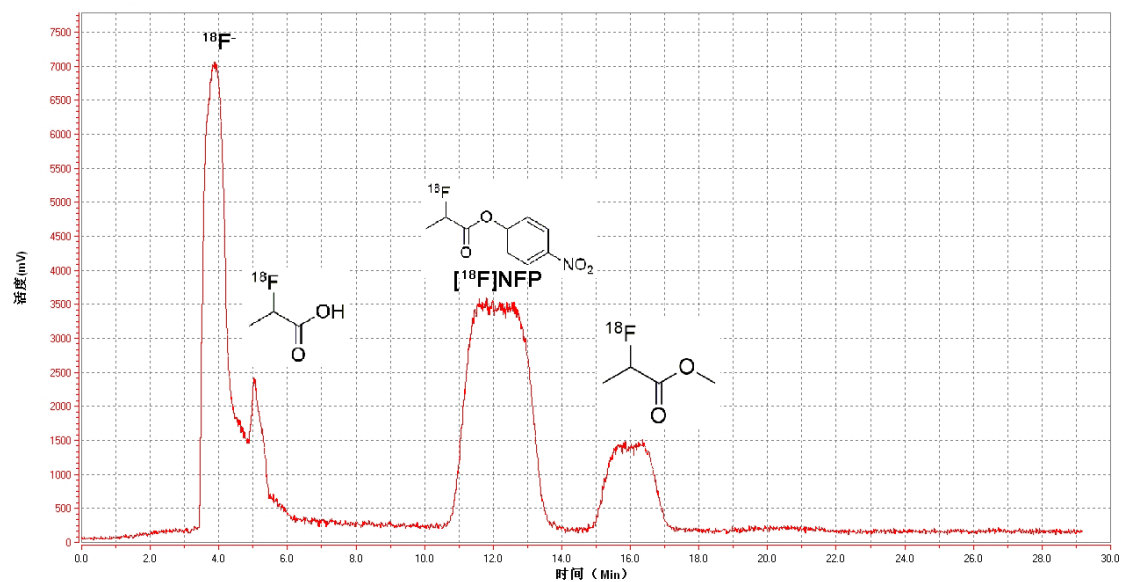


Figure S4. Radio-HPLC of [ $^{18}\text{F}$ ]FP-D-Lys $^6$ -GnRH for vitro stability

