

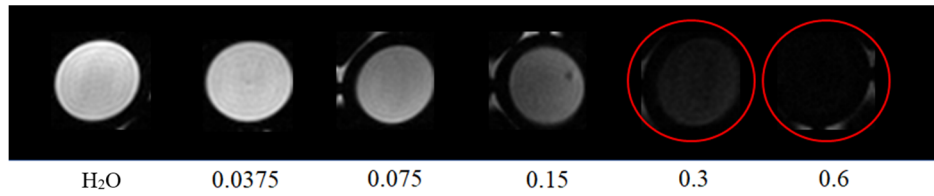
# **Supplementary Information**

## **Glu-Urea-Lys Scaffold Functionalized Superparamagnetic Iron Oxide Nanoparticles targeting PSMA for In Vivo Molecular MRI of Prostate Cancer**

### **Content**

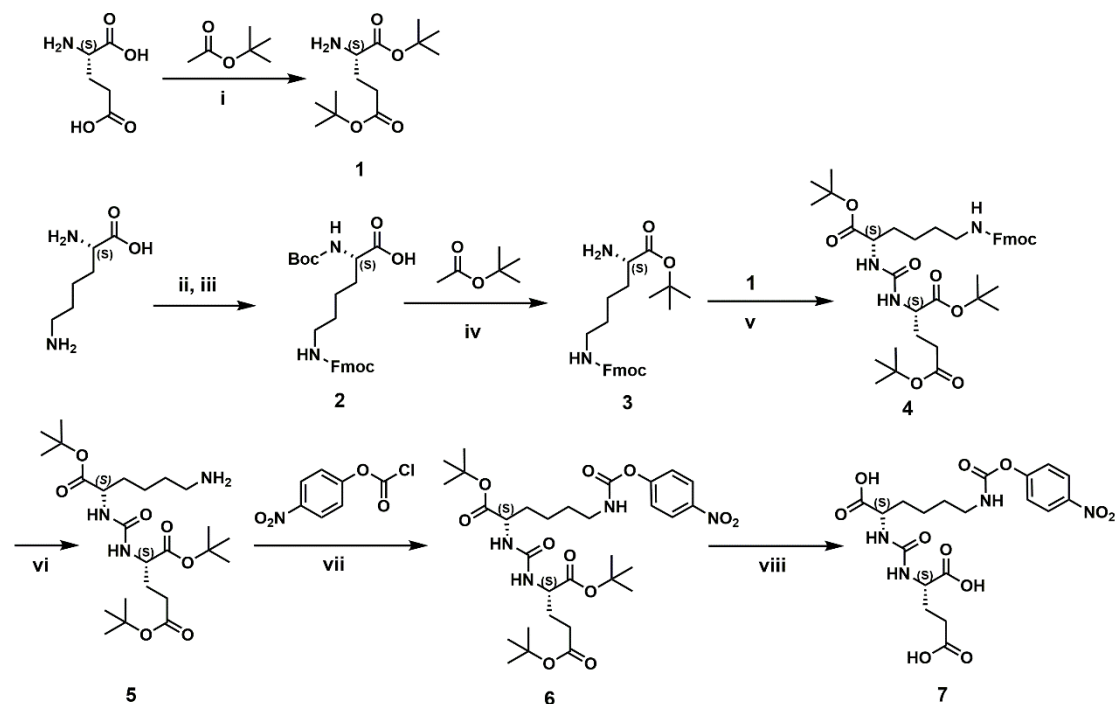
1. The  $T_2$  weighted imaging of  $\text{Fe}_3\text{O}_4@\text{DPA-PEG-PSMA-1}$  in PBS
2.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of PSMA-1 and intermediates
3. HRMS spectra of PSMA-1 and intermediates

1. The T<sub>2</sub> weighted imaging of Fe<sub>3</sub>O<sub>4</sub>@DPA-PEG-PSMA-1 in PBS



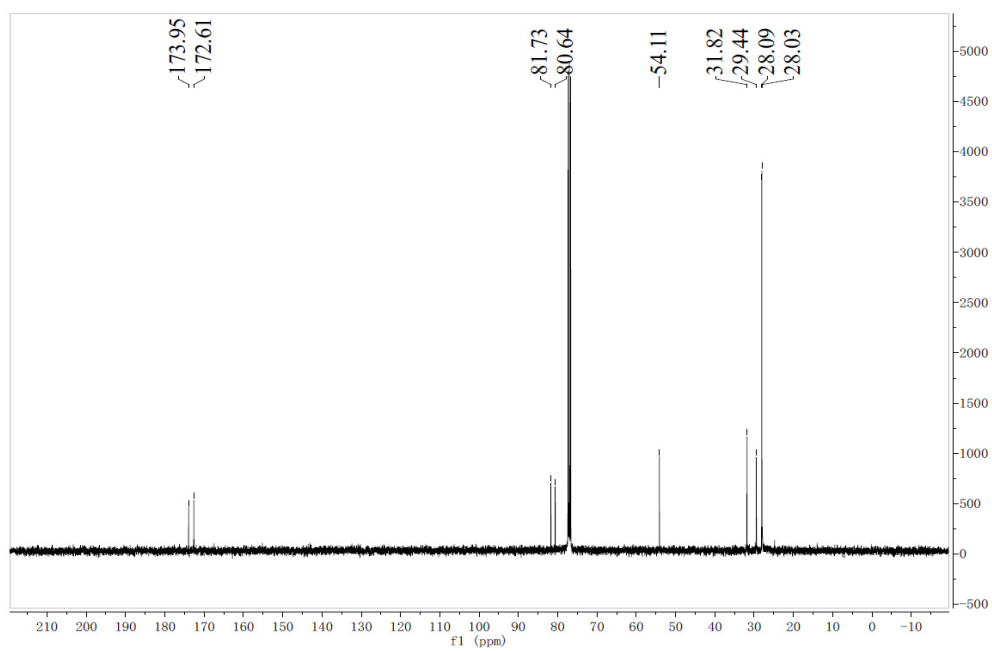
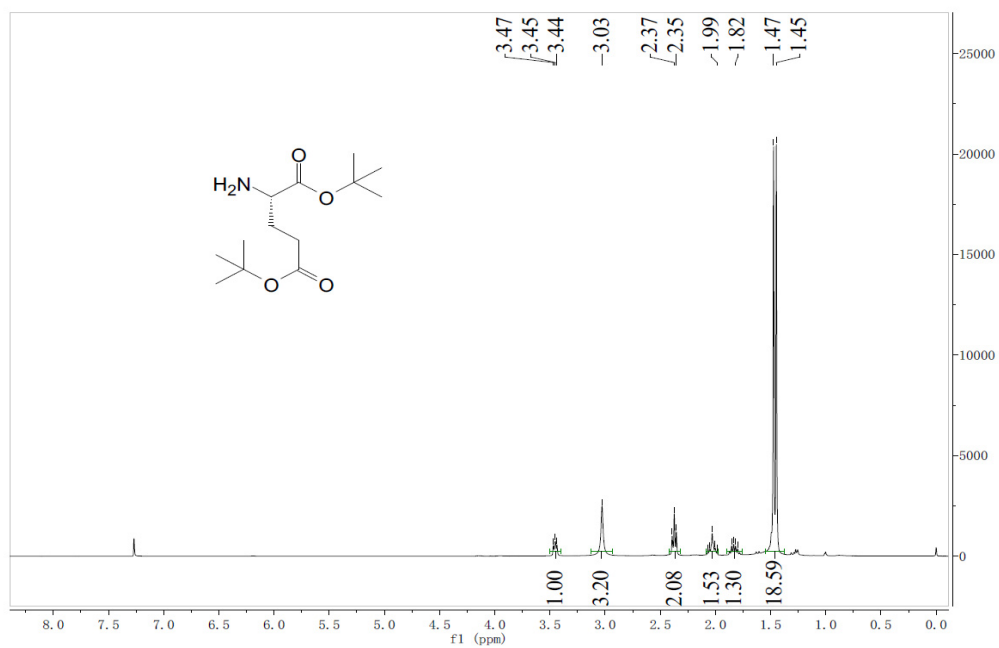
**Supplementary Figure S1.** The T<sub>2</sub> weighted imaging of Fe<sub>3</sub>O<sub>4</sub>@DPA-PEG-PSMA-1 of different concentrations (0.0375-0.6  $\mu\text{g}(\text{Fe})/\text{ml}$ ) in PBS.

## 2. $^1\text{H}$ and $^{13}\text{C}$ NMR spectra of PSMA-1 and intermediates

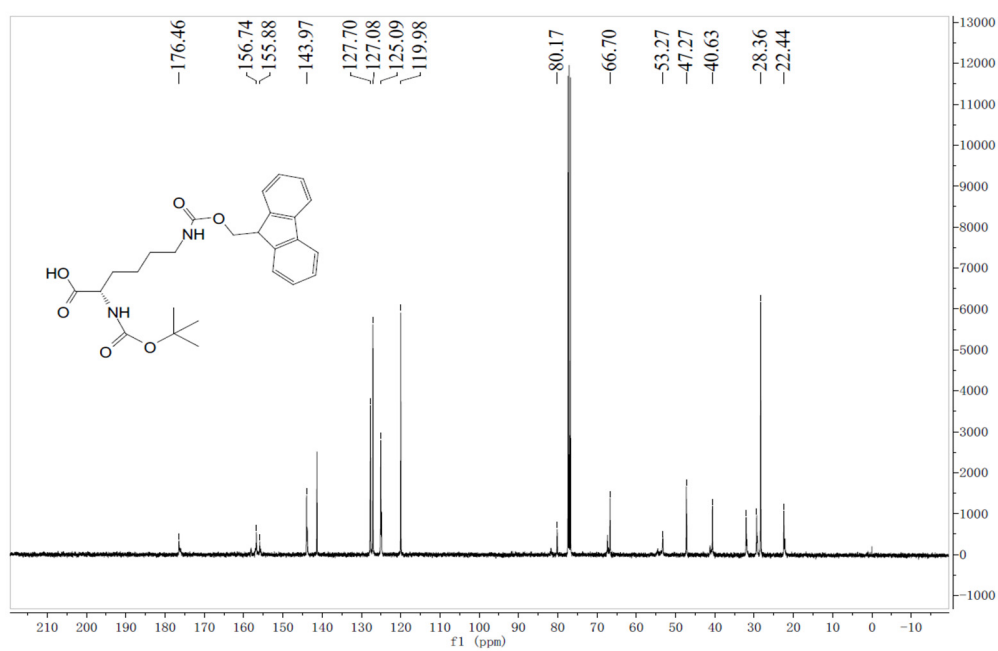
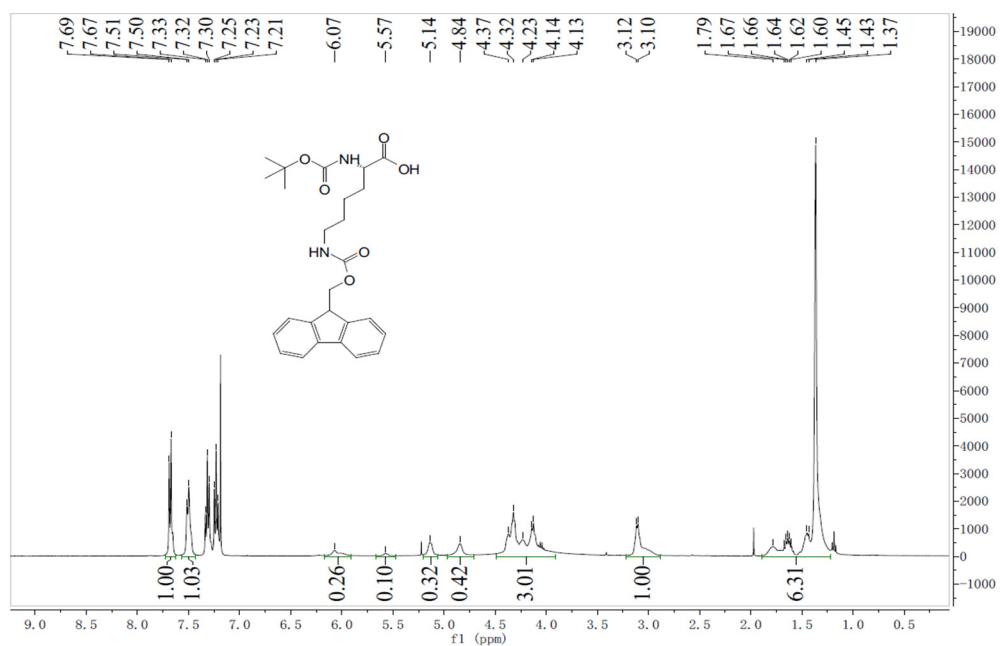


**Supplementary Scheme S1.** The synthesis of PSMA-1. Reagents and conditions: (i) 70%  $\text{HClO}_4$ , 47.6%; (ii) Fmoc-OSU,  $\text{CuSO}_4$ ,  $\text{NaHCO}_3$ ; (iii)  $\text{Na}_2\text{CO}_3$ , 8-hydroxyquinoline,  $\text{Boc}_2\text{O}$ , 64% in 2 steps; (iv) 70%  $\text{HClO}_4$ , 70%; (v) triphosgene, DIEA, 75.3%; (vi) DEA, 70%; (vii) DEA,  $\text{CH}_2\text{Cl}_2$ , 73%; (viii) 25% TFA/ $\text{CH}_2\text{Cl}_2$ , 85%.

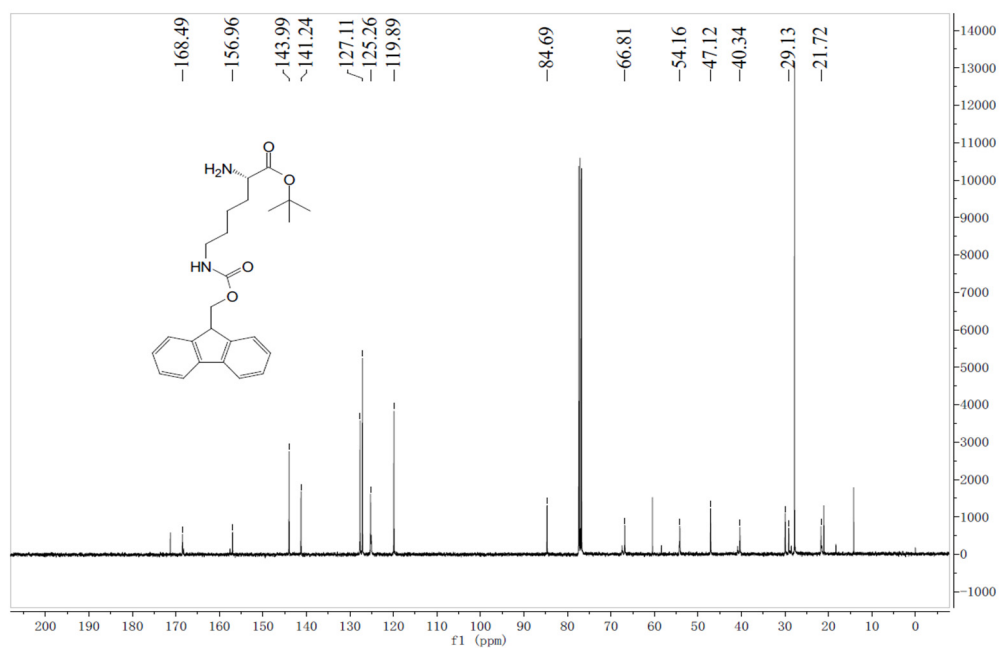
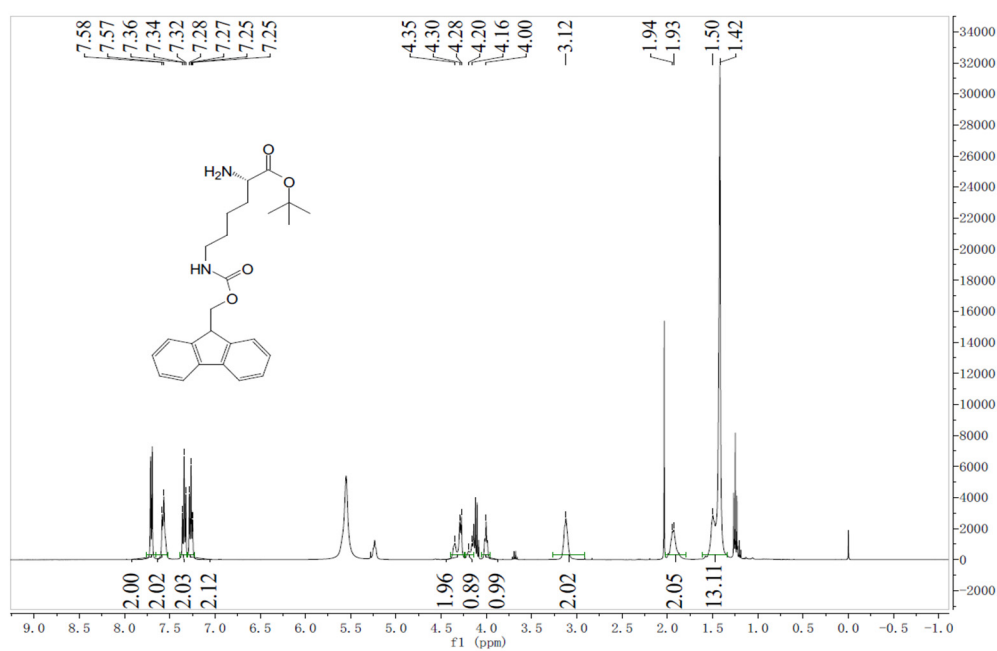
di-tert-butyl L-glutamate (**1**)



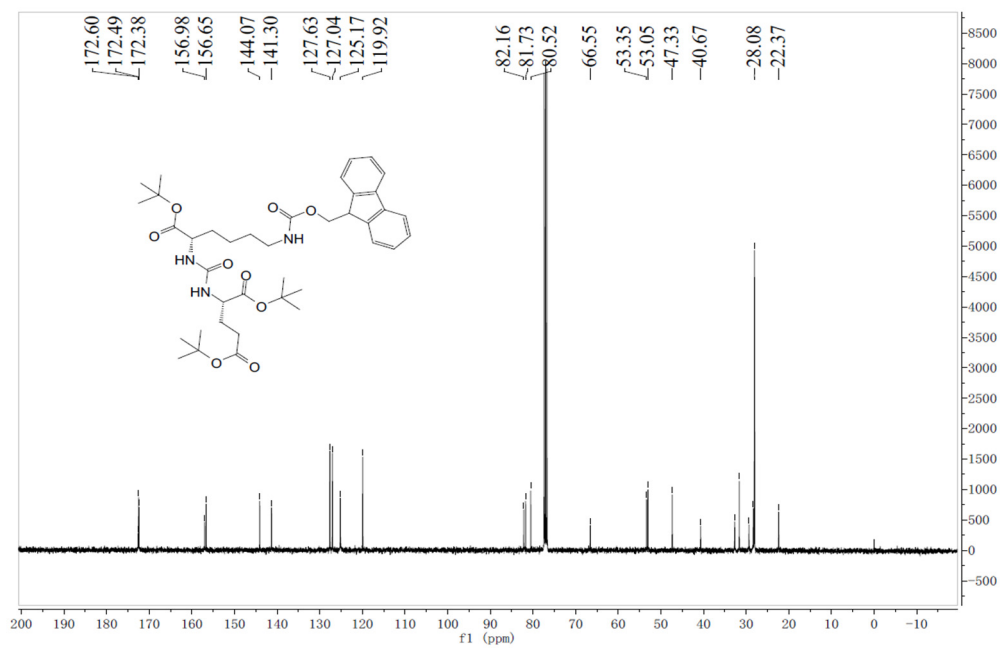
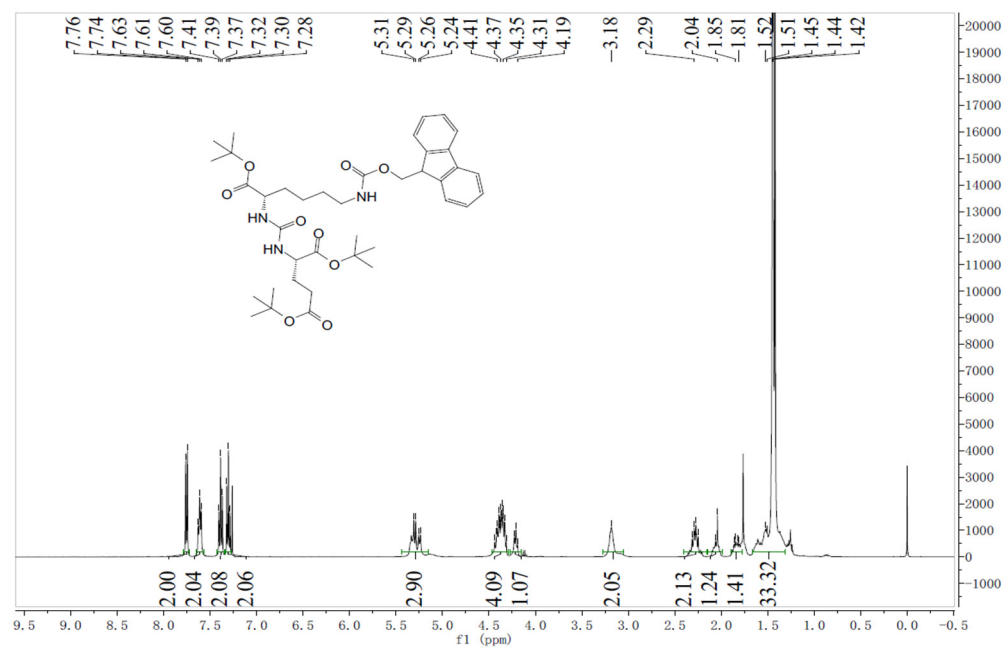
*N*<sup>6</sup>-Fmoc-*N*<sup>2</sup>-Boc-L-lysine (**2**)



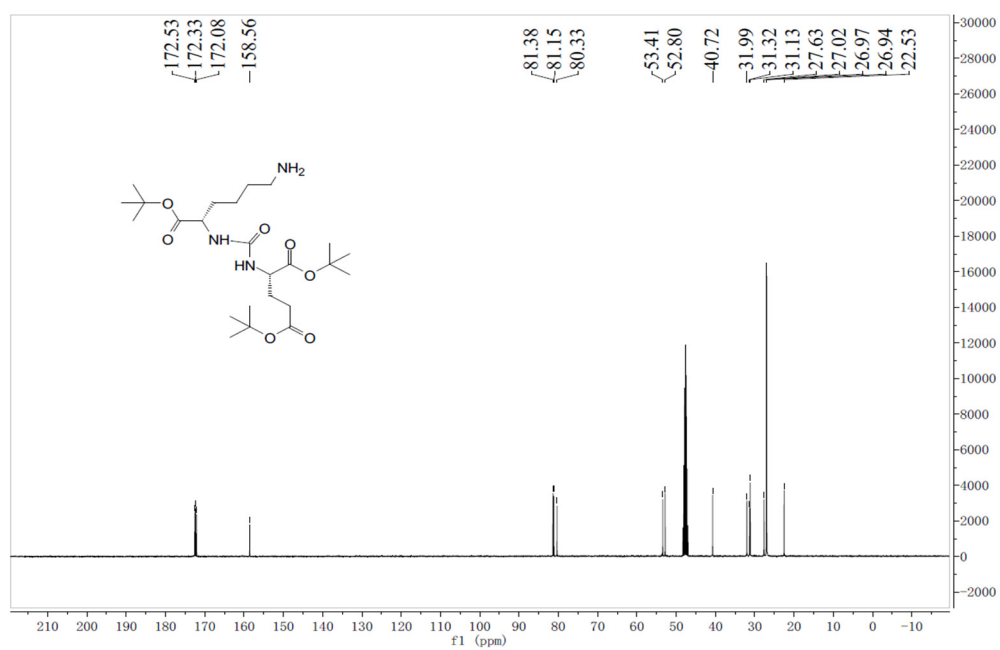
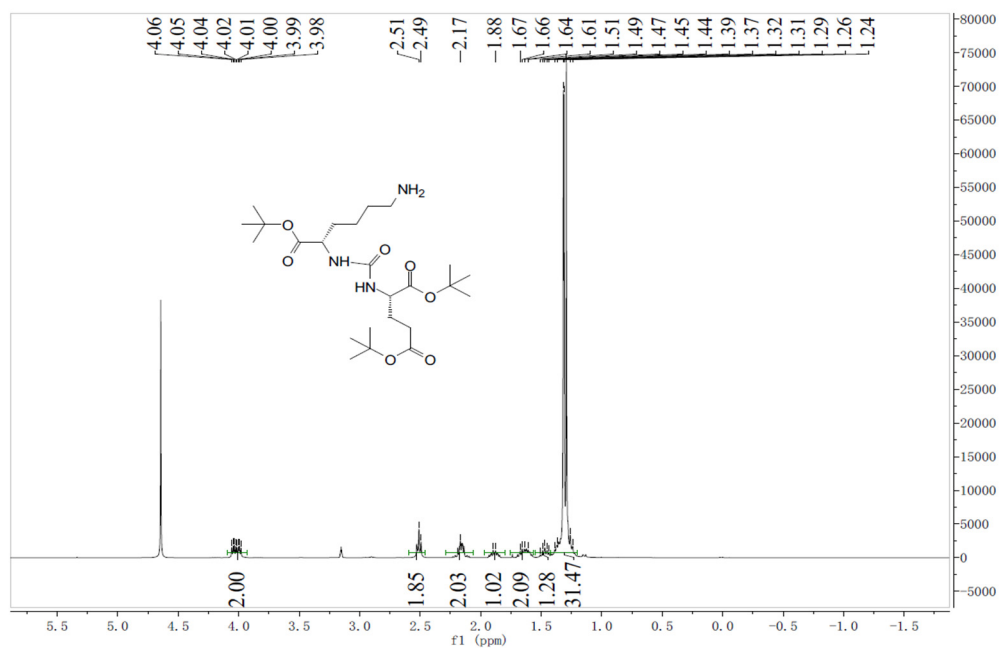
*tert*-butyl *N*<sup>6</sup>-Fmoc-L-lysinate (**3**)



(*Tert*-butyl-*N*<sup>6</sup>-Fmoc)-*L*-Lys-Urea-(*di-tert*-butyl)-*L*-Glu (**4**)

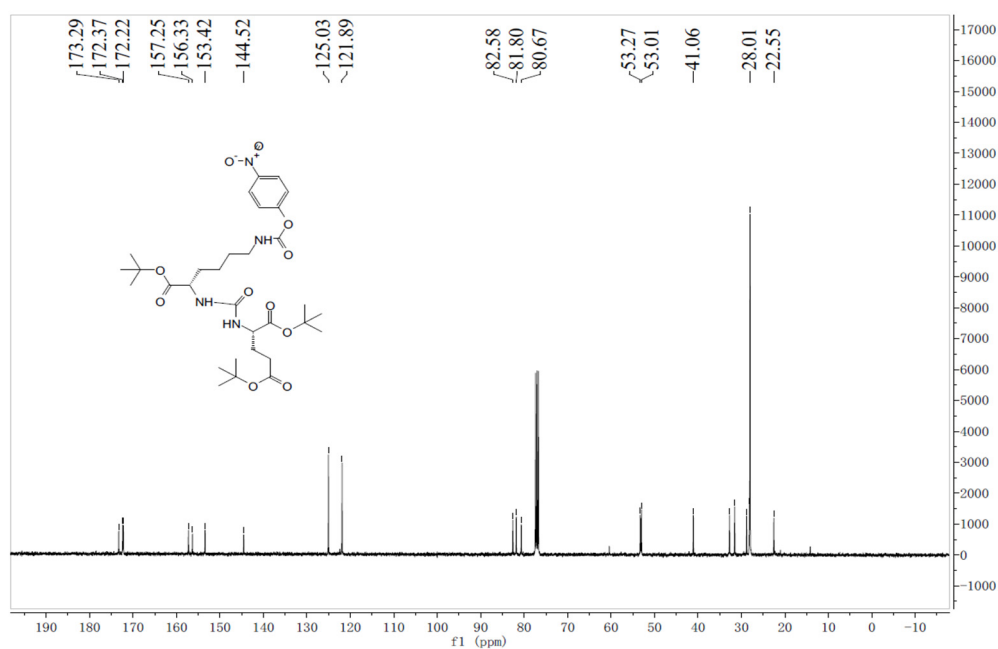
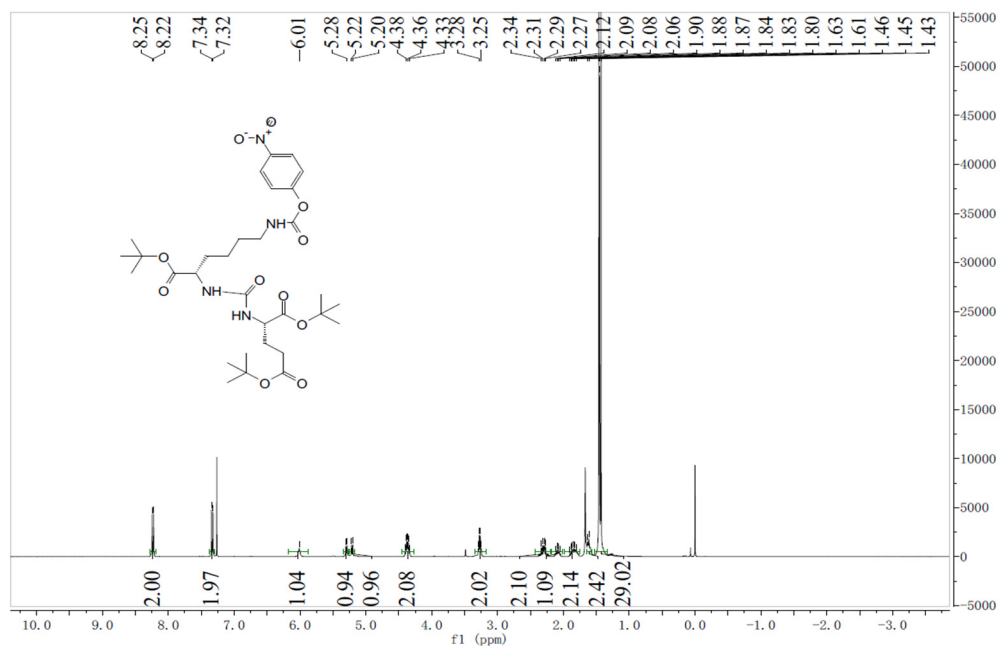


#### 4.2.5. (Tert-butyl)-L-Lys-Urea-(di-tert-butyl)-L-Glu (5)

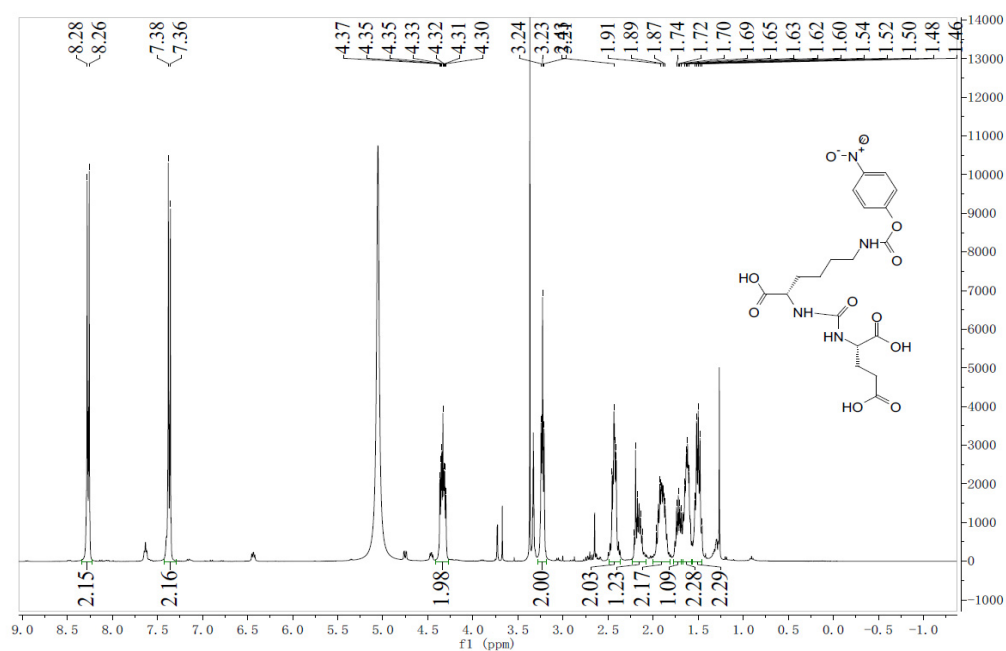




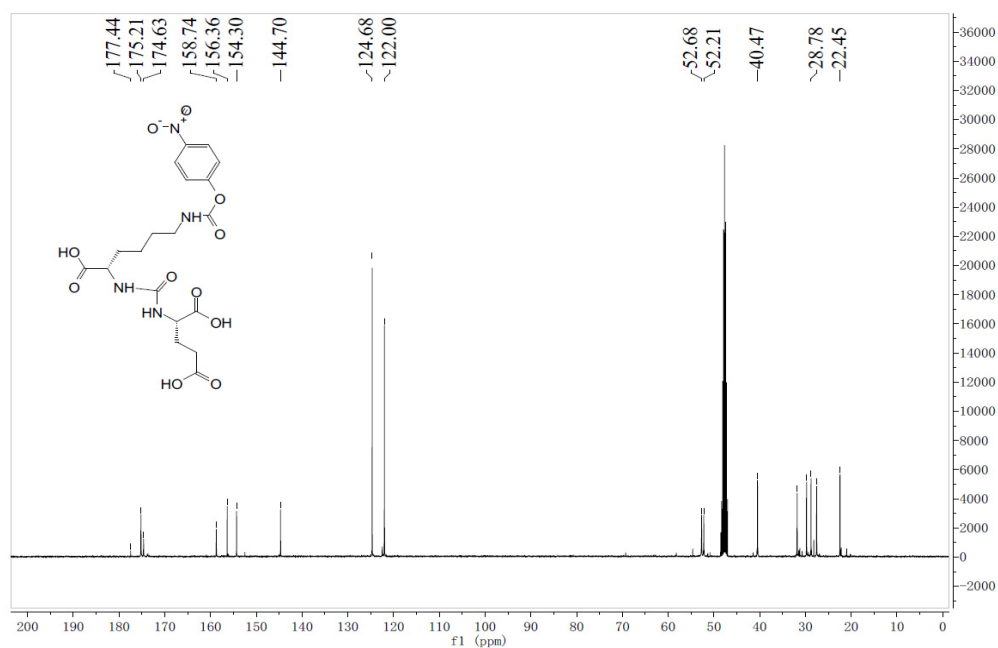
*di-tert-butyl (((S)-1-(tert-butoxy)-6-(((4-nitrophenoxy)carbonyl)amino)-1-oxohexan-2-yl) carbamoyl)-L-glutamate (6)*



*(((S)-1-carboxy-5-(((4-nitrophenoxy)carbonyl)amino)pentyl)carbamoyl)-L-glutamic acid (PSMA-1)*



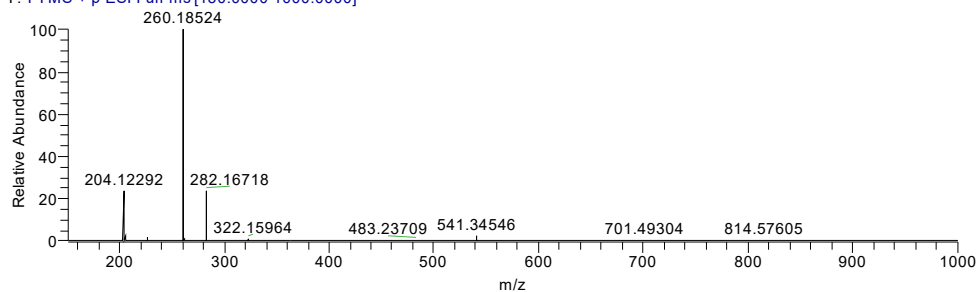
<sup>1</sup>H NMR of Compound 3-007



### 3. HRMS spectra of PSMA-1 and intermediates

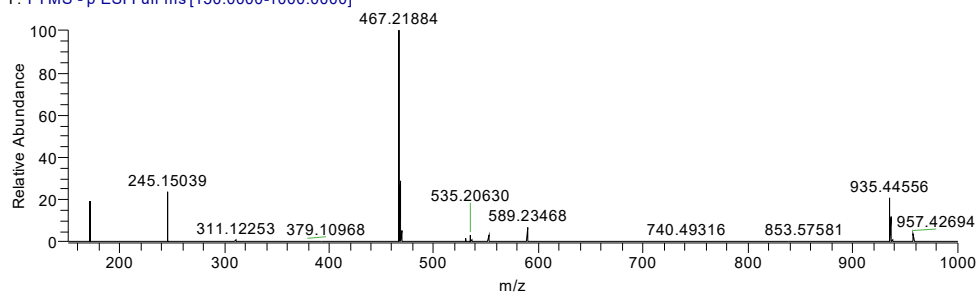
#### *di-tert-butyl L-glutamate (1)*

3-001 #9 RT: 0.09 AV: 1 NL: 7.50E8  
T: FTMS + p ESI Full ms[150.0000-1000.0000]



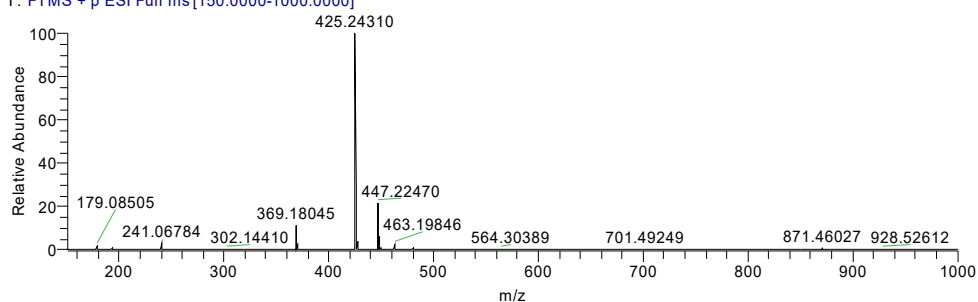
#### *N<sup>6</sup>-Fmoc-N<sup>2</sup>-Boc-L-lysine (2)*

3-002 #8 RT: 0.08 AV: 1 NL: 1.37E8  
T: FTMS - p ESI Full ms[150.0000-1000.0000]



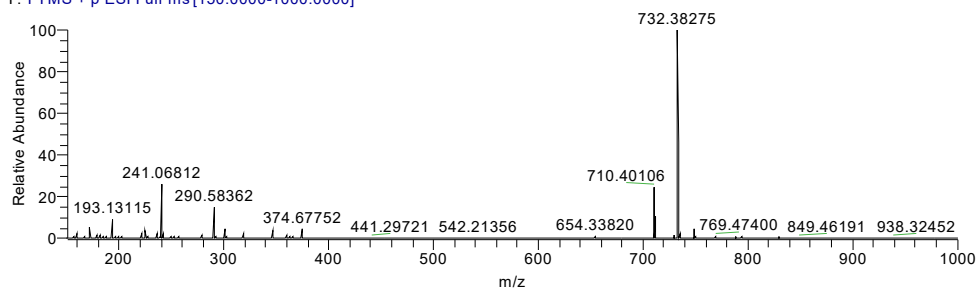
#### *tert-butyl N<sup>6</sup>-Fmoc-L-lysinate (3)*

3-004 #17 RT: 0.17 AV: 1 NL: 3.94E8  
T: FTMS + p ESI Full ms[150.0000-1000.0000]



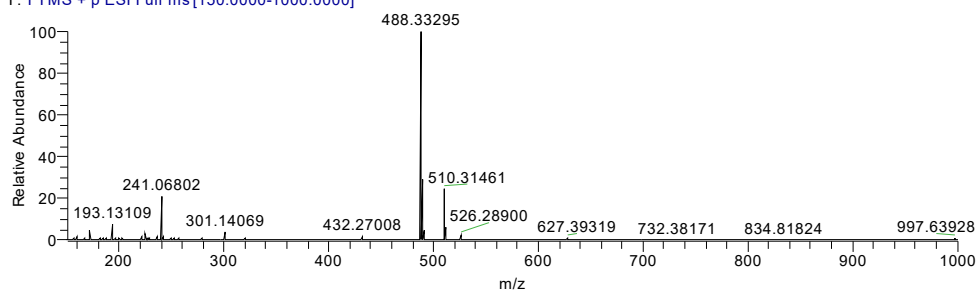
**(Tert-butyl-*N*<sup>6</sup>-Fmoc)-L-Lys-Urea-(di-tert-butyl)-L-Glu (4)**

3-005 #31 RT: 0.30 AV: 1 NL: 4.61E7  
T: FTMS + p ESI Full ms[150.0000-1000.0000]



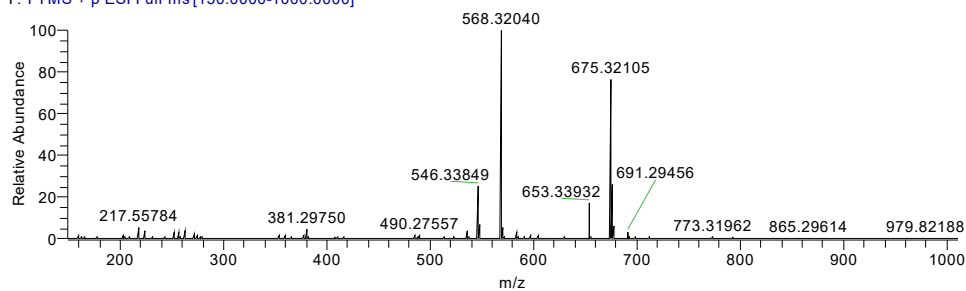
**(Tert-butyl)-L-Lys-Urea-(di-tert-butyl)-L-Glu (5)**

3-006 #27 RT: 0.26 AV: 1 NL: 6.00E7  
T: FTMS + p ESI Full ms[150.0000-1000.0000]



**di-tert-butyl (((S)-1-(tert-butoxy)-6-(((4-nitrophenoxy)carbonyl)amino)-1-oxohexan-2-yl) carbamoyl)-L-glutamate (6)**

3-007 #21 RT: 0.21 AV: 1 SB: 1 0.03 NL: 1.37E7  
T: FTMS + p ESI Full ms[150.0000-1000.0000]



*(((S)-1-carboxy-5-(((4-nitrophenoxy)carbonyl)amino)pentyl)carbamoyl)-L-glutamic acid (PSMA-1)*

3-008 #8 RT: 0.08 AV: 1 SB: 1 0.03 NL: 8.92E7  
T: FTMS - p ESI Full ms [150.0000-1000.0000]

