

## Supporting Information

for

### **Novel solid-phase strategy for the synthesis of ligand- argeted fluorescent-labelled chelating peptide conjugates as a theranostic tool for cancer**

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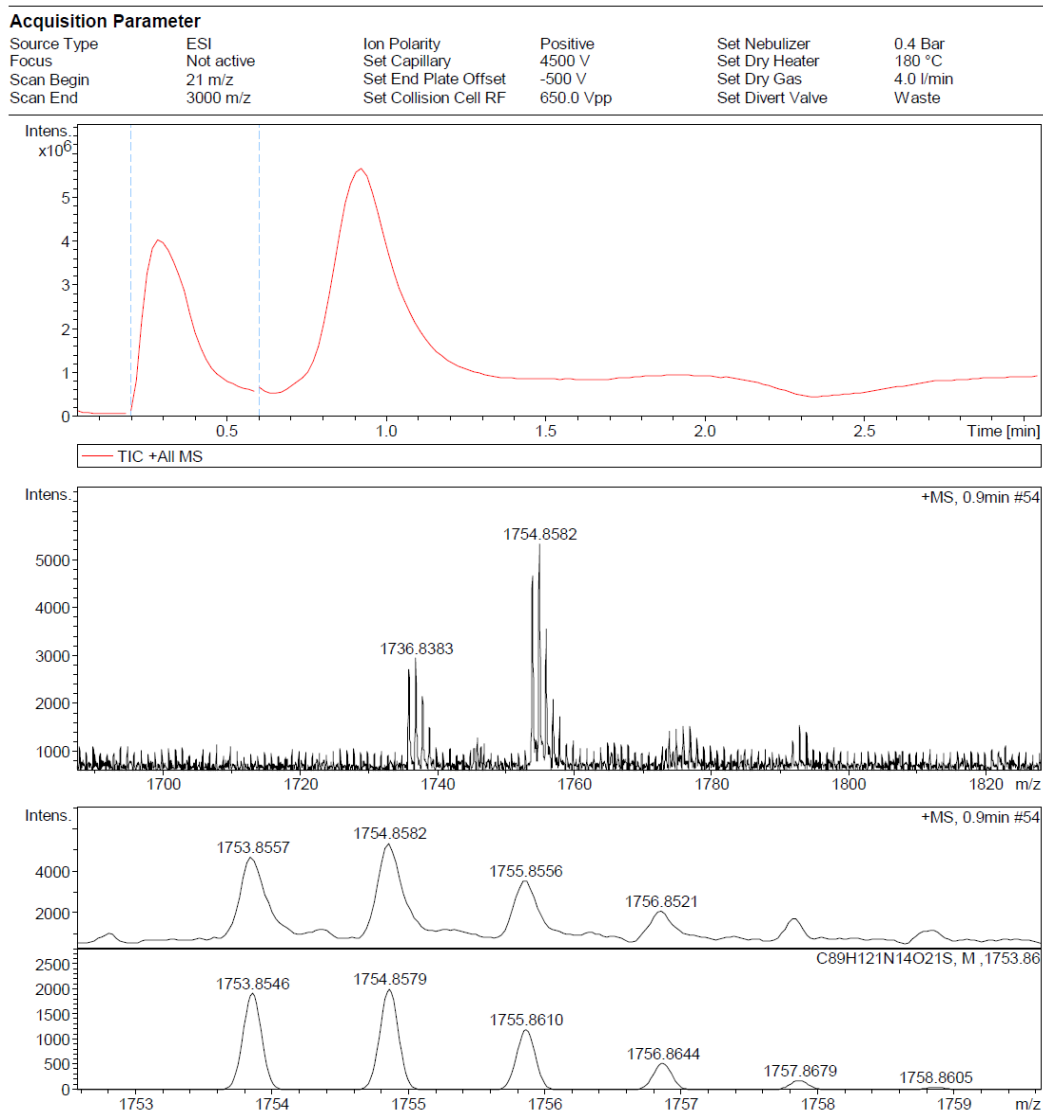
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**NMR and HRMS spectra of compounds 3, 4 and analytical HPLC spectra  
of purified ( $\lambda$  at 254 nm and 225 nm) as well as crude ( $\lambda$  at 225 nm),  
electrospray ionization mass spectra and high-resolution mass spectra for  
bioconjugates 13 and 17.**

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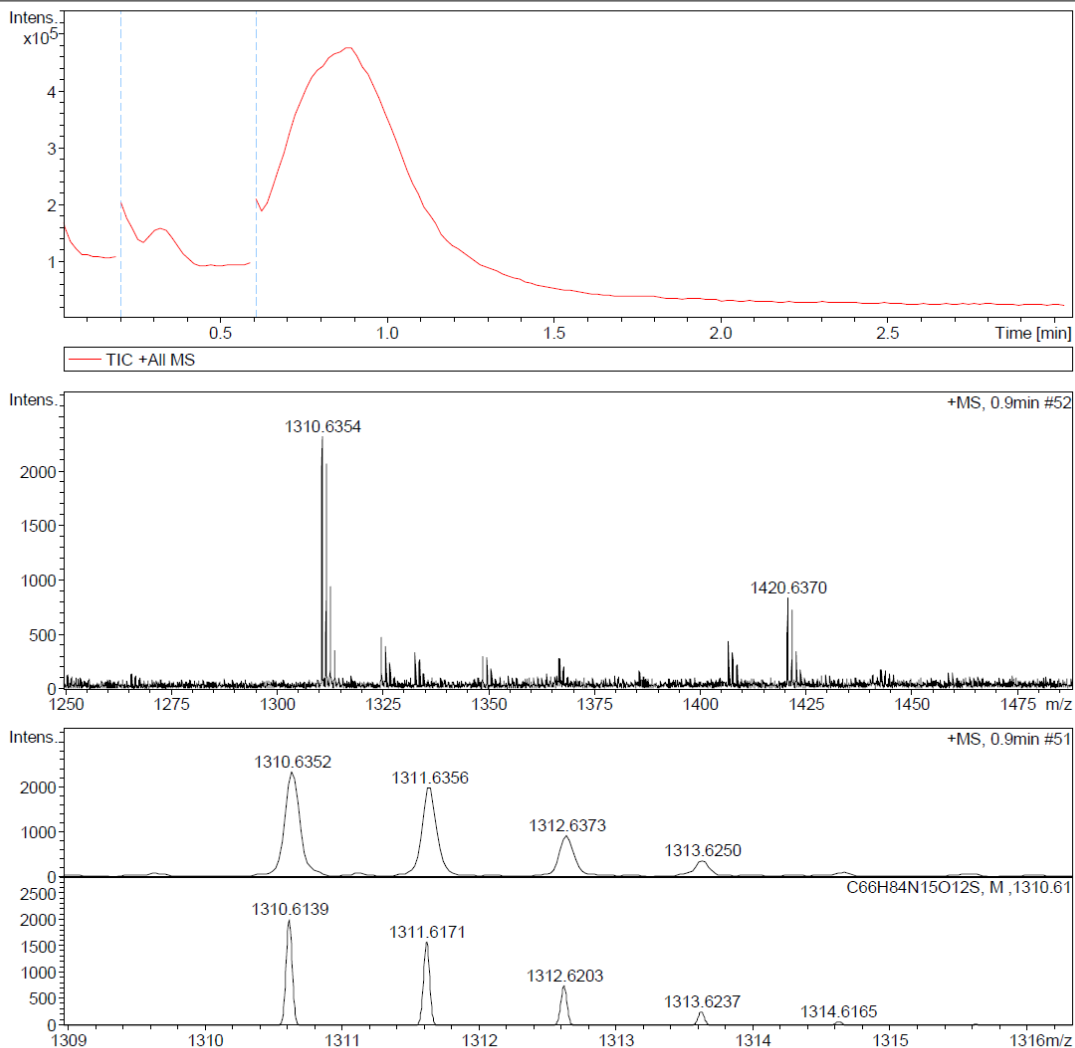
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- Figure S15**  $^{13}\text{C}$  NMR of (*S*)-5-(*tert*-butoxy)-4-(3-((*S*)-1,5-di-*tert*-butoxy-1,5-dioxopentan-2-yl)ureido)-5-oxopentanoic acid (**4**)
- Figure S16** HRMS spectra (*S*)-5-(*tert*-butoxy)-4-(3-((*S*)-1,5-di-*tert*-butoxy-1,5-dioxopentan-2-yl)ureido)-5-oxopentanoic acid (**4**)



**Figure S1:** High-resolution mass spectrometry data for PSMA targeting DUPA rhodamine B chelating conjugate **13**.

**Acquisition Parameter**

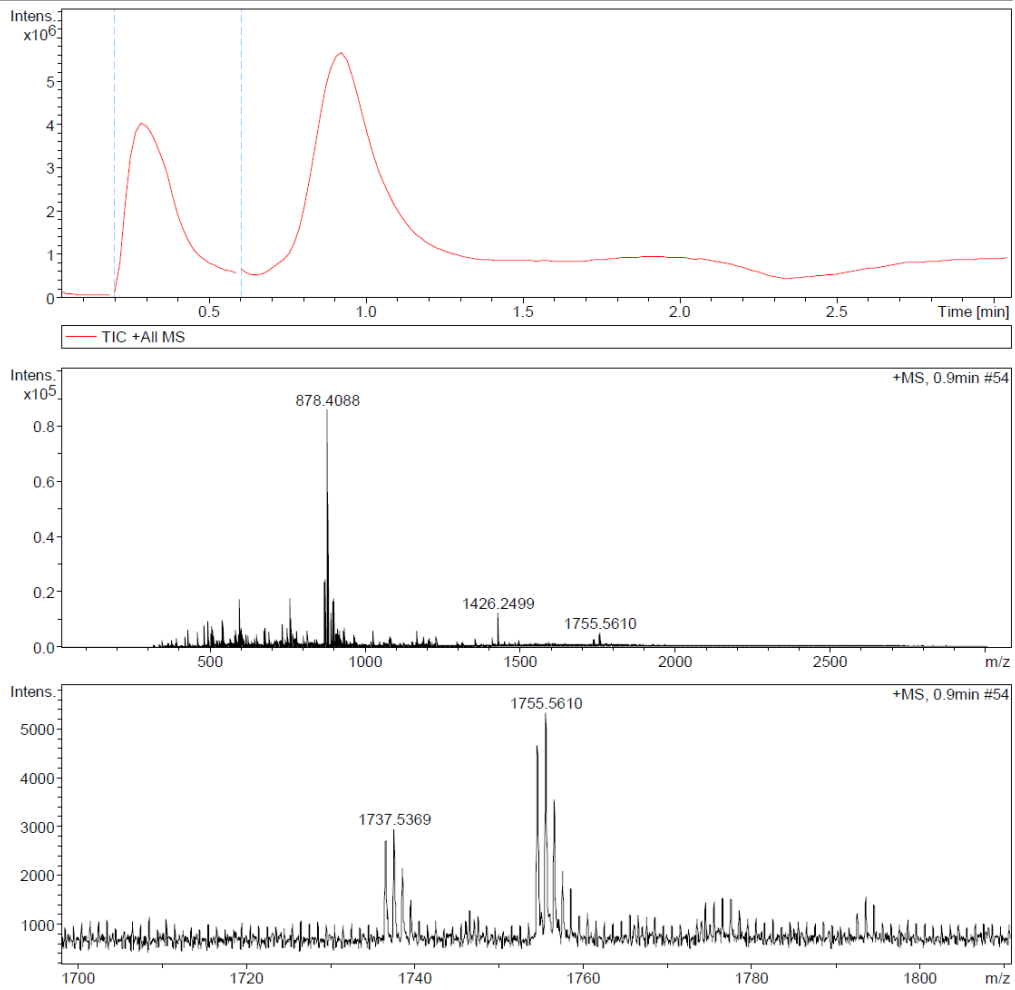
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**Figure S2:** High-resolution mass spectrometry data pterate rhodamine B chelating conjugate **17**.

**Acquisition Parameter**

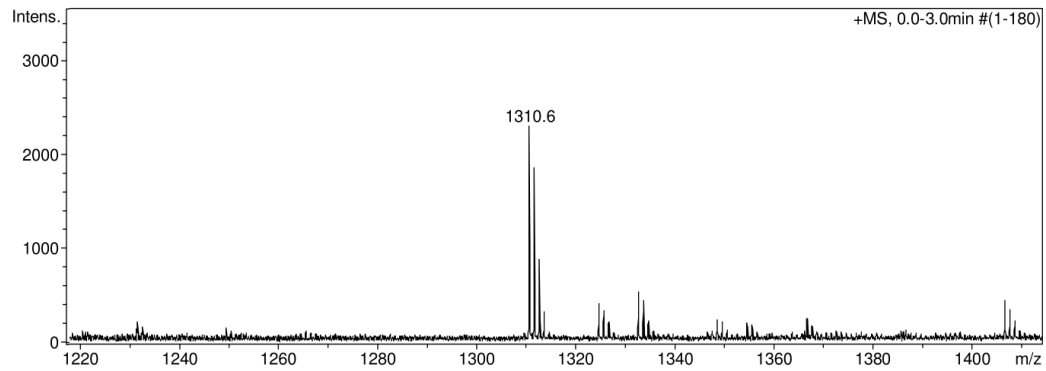
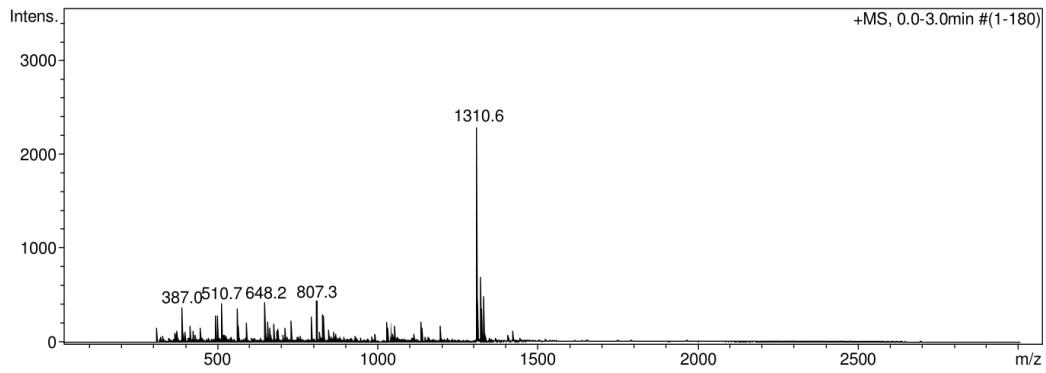
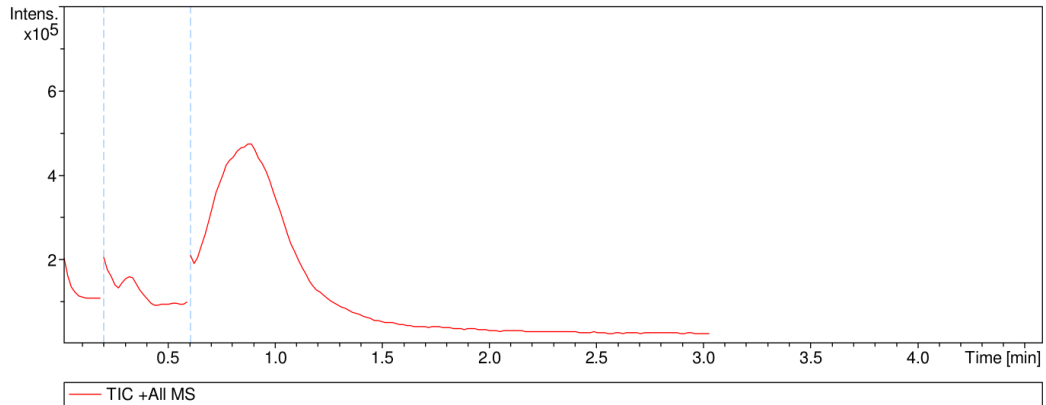
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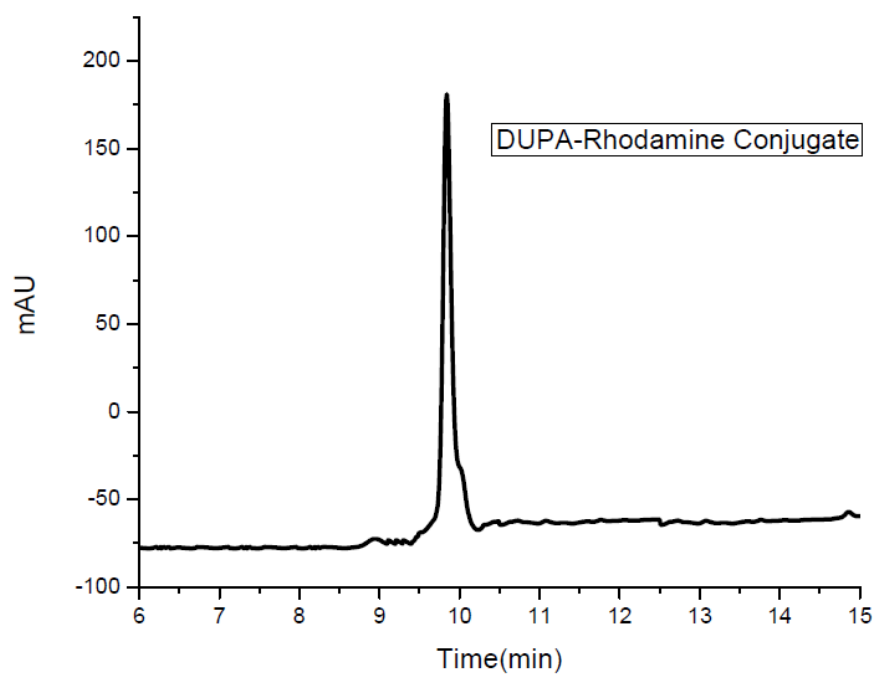
**Figure S3:** ESIMS spectra for PSMA targeting DUPA rhodamine B chelating conjugate **13**.

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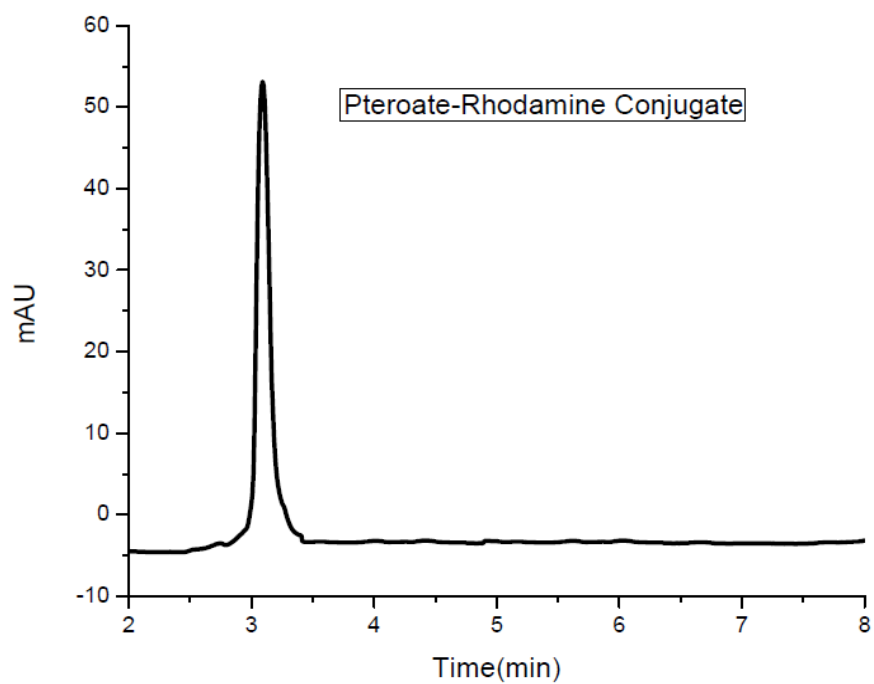
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Scan End	3000 m/z	Set Collision Cell RF	650.0 Vpp	Set Divert Valve	Waste



**Figure S4:** ESIMS spectra for pteroate rhodamine B chelating conjugate **17**.

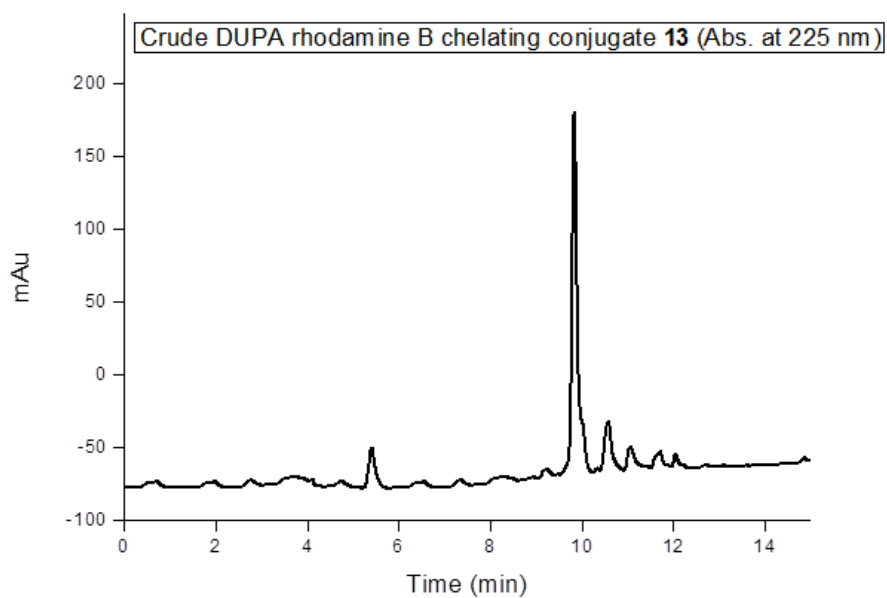


**Figure S5:** Analytical LC data of DUPA rhodamine B chelating conjugate **13** (Abs. at 254 nm).

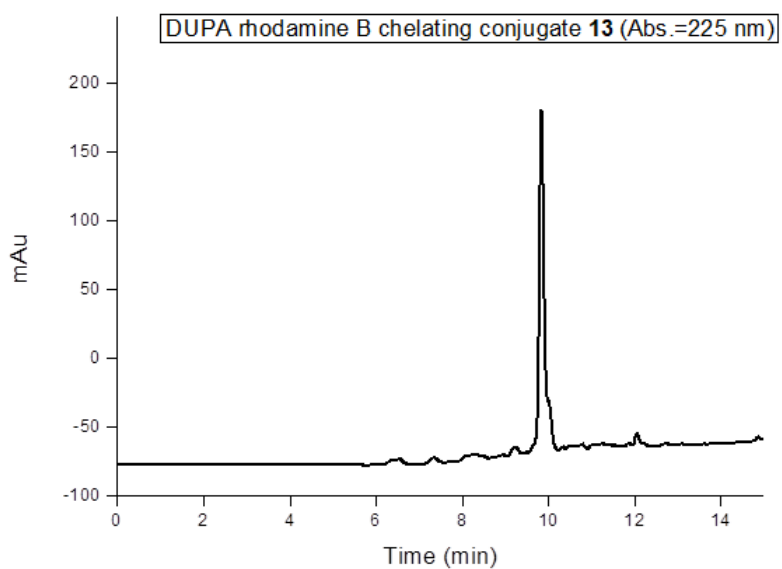


**Figure S6:** Analytical LC data of pteroate rhodamine B chelating conjugate **17** (Abs. at 254 nm).

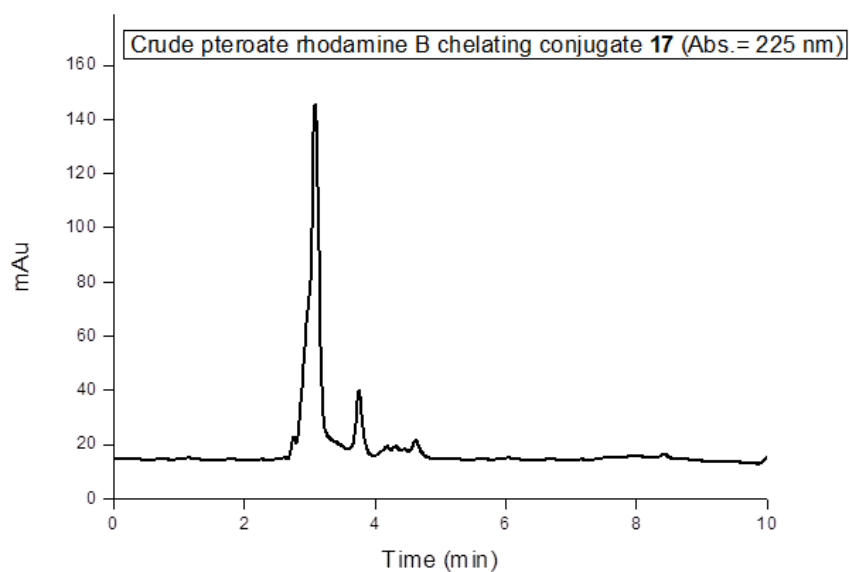




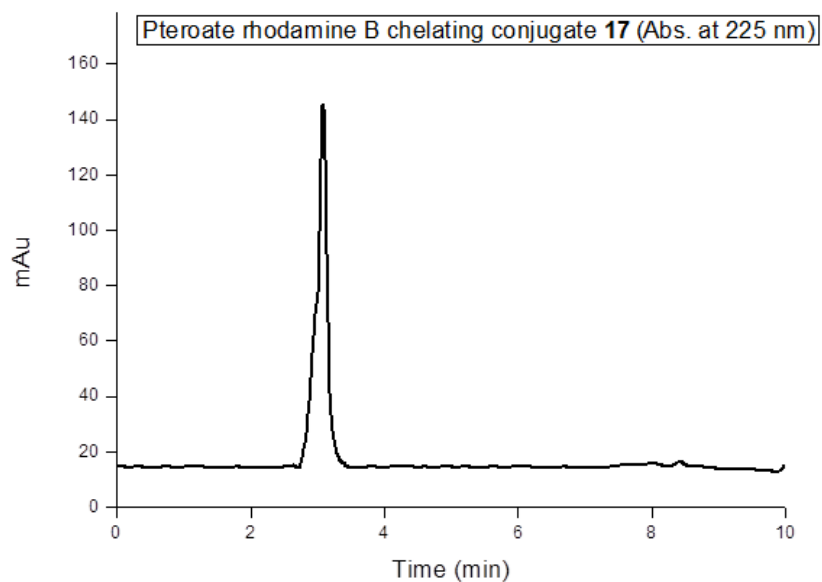
**Figure S7:** Analytical LC data of crude DUPA rhodamine B chelating conjugate **13** (Abs. at 225 nm).



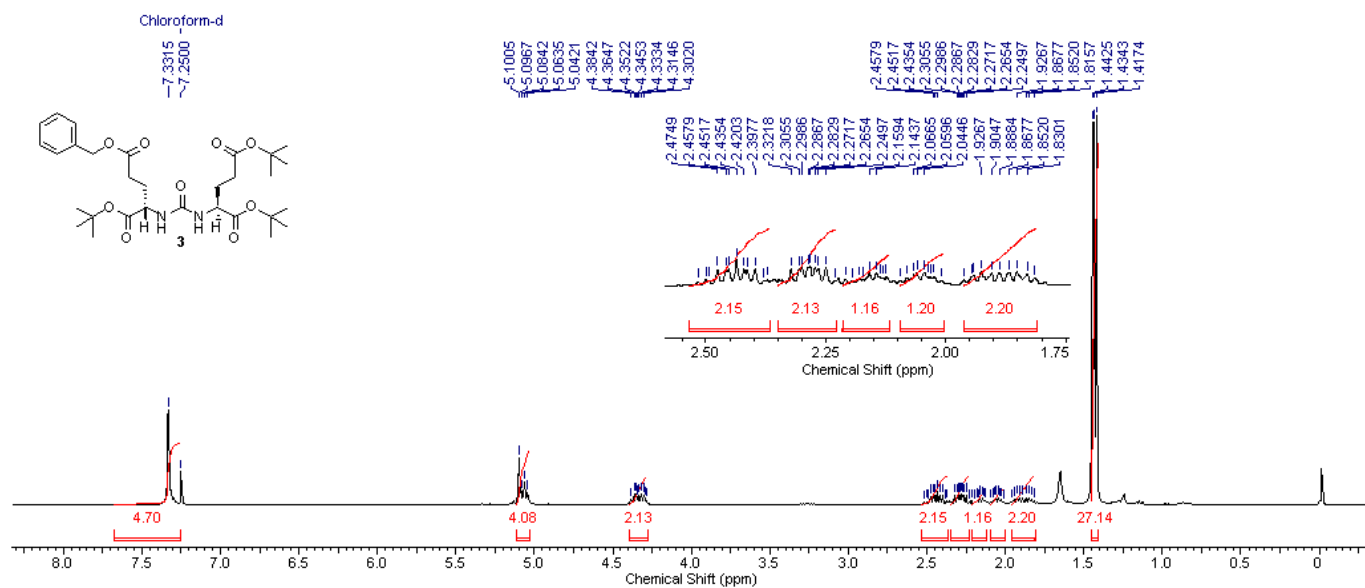
**Figure S8:** Analytical LC data of purified DUPA rhodamine B chelating conjugate **13** (Abs. at 225 nm).



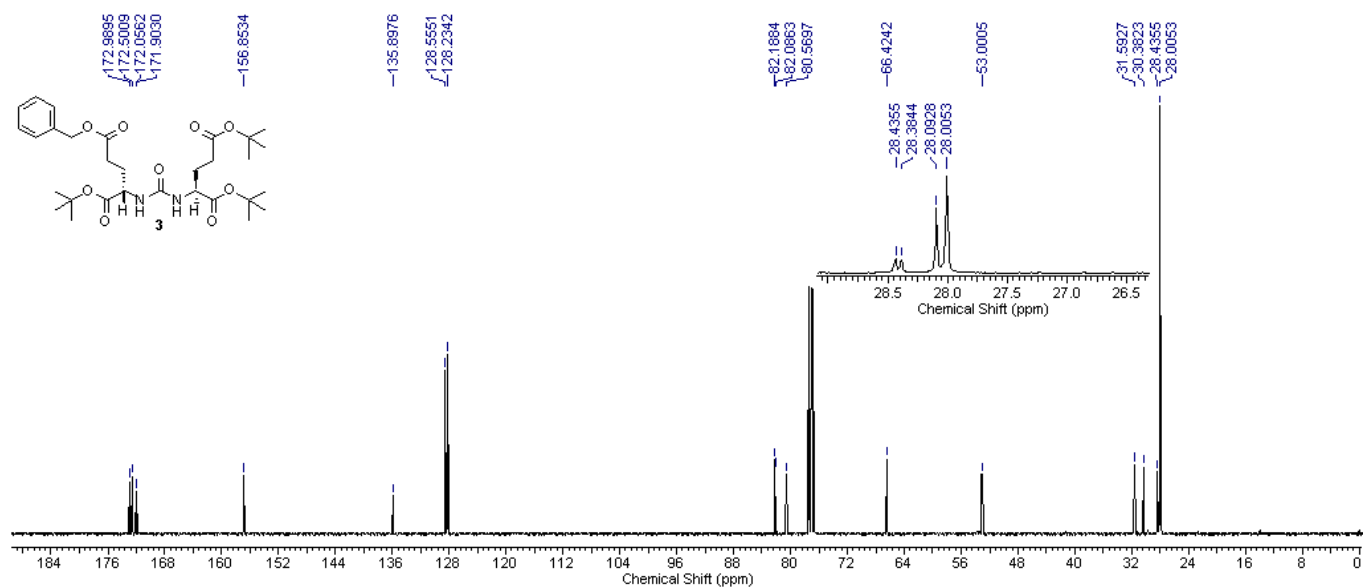
**Figure S9:** Analytical LC data of crude pteroate rhodamine B chelating conjugate **17** (Abs. at 225 nm).



**Figure S10:** Analytical LC data of purified pteroate rhodamine B chelating conjugate **17** (Abs. at 225 nm).



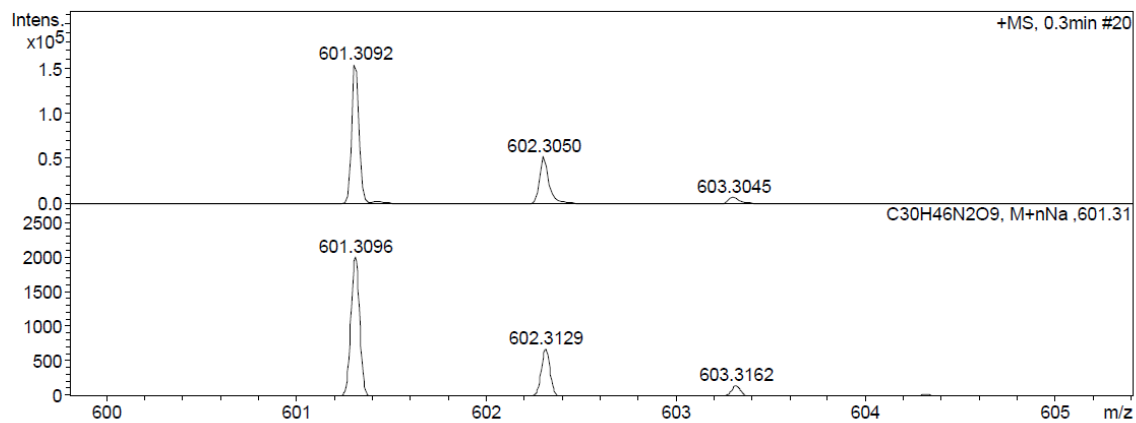
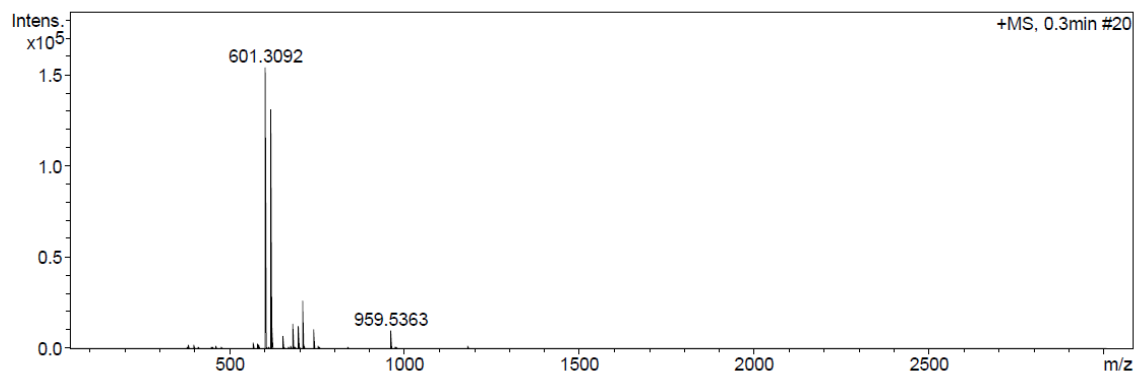
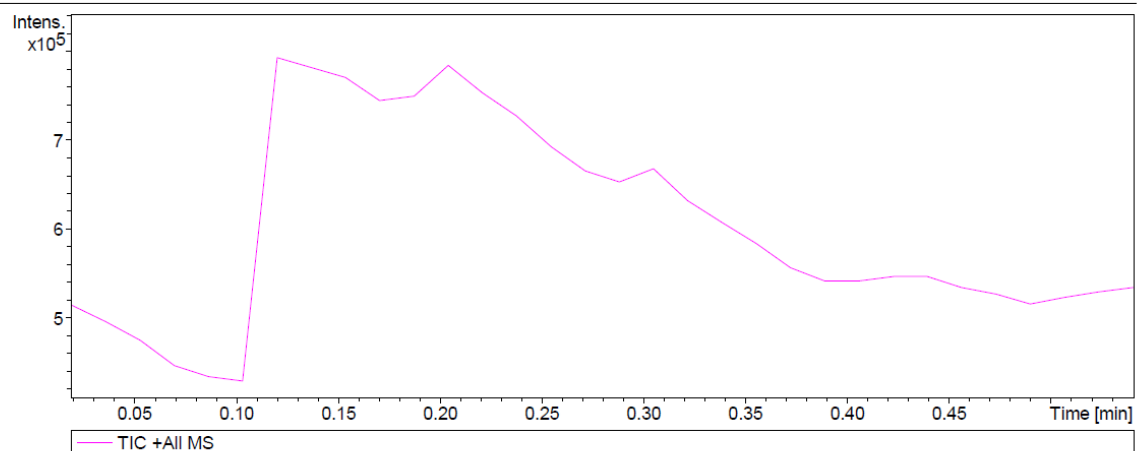
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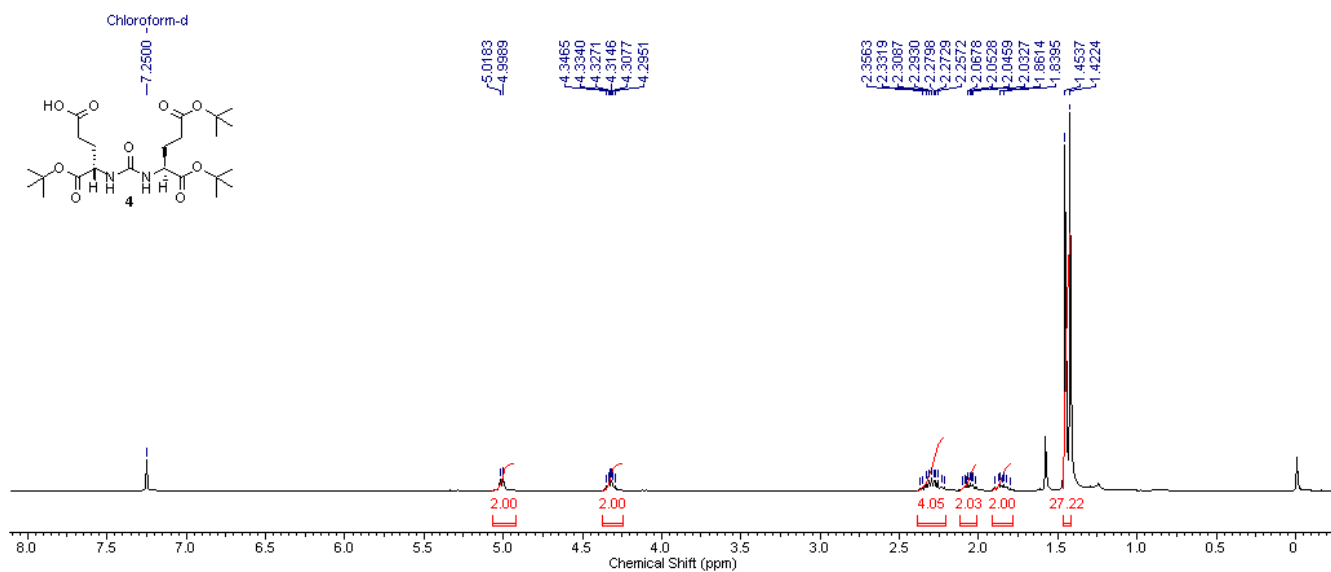
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**Acquisition Parameter**

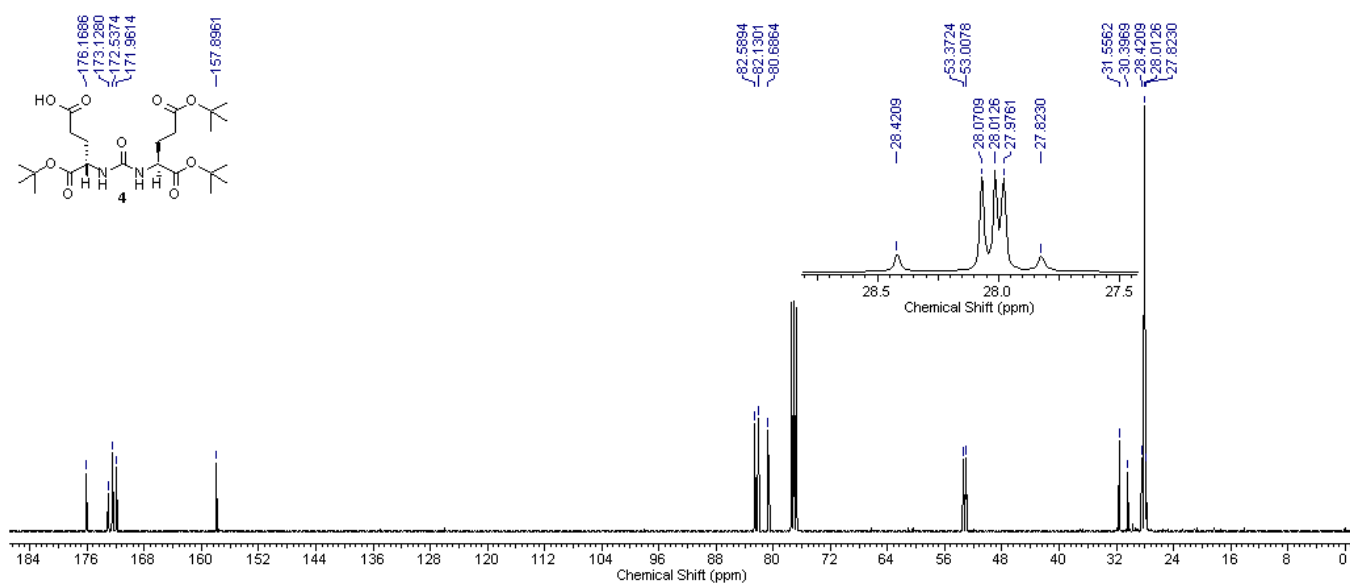
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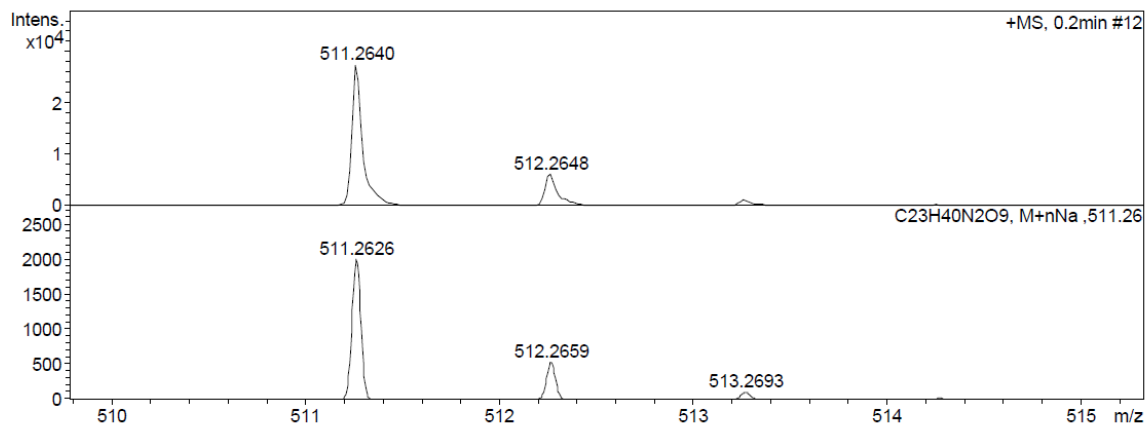
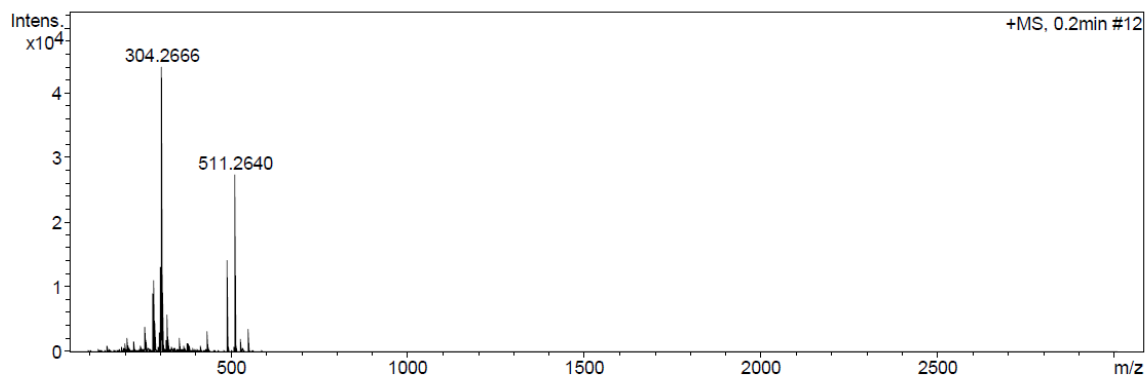
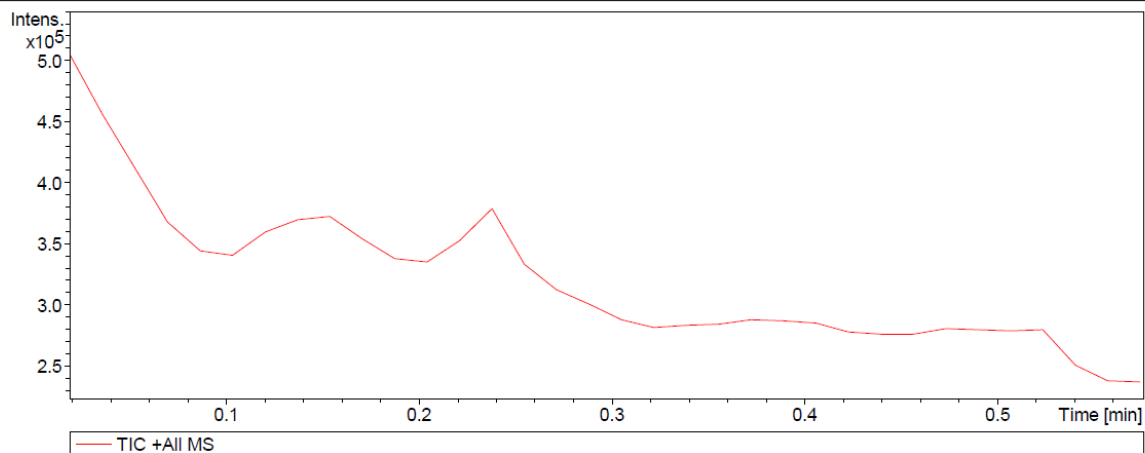
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste



**Figure S16.** HRMS of (*S*)-5-(*tert*-butoxy)-4-(3-((*S*)-1,5-di-*tert*-butoxy-1,5-dioxopentan-2-yl)ureido)-5-oxopentanoic acid (**4**).