

**Supplementary Information for**  
**Turning Non-Specific Interference into Signal Amplification:**  
**Covalent Biosensing Nanoassembly Enabled by Metal-Catalyzed**  
**Cross-Coupling**

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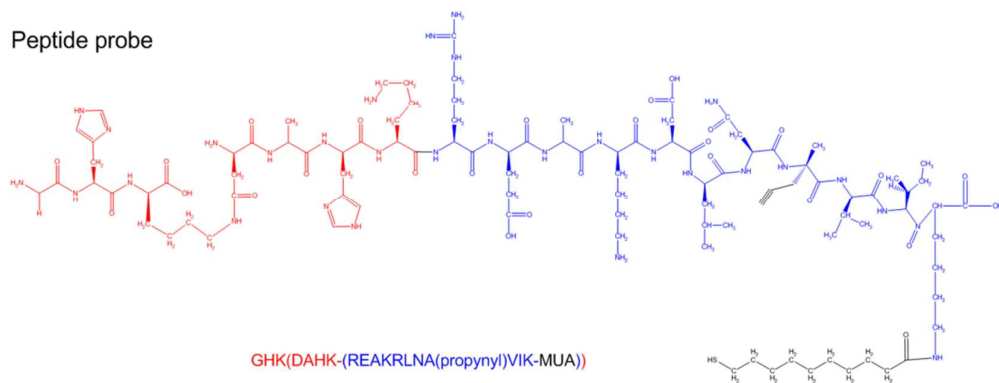
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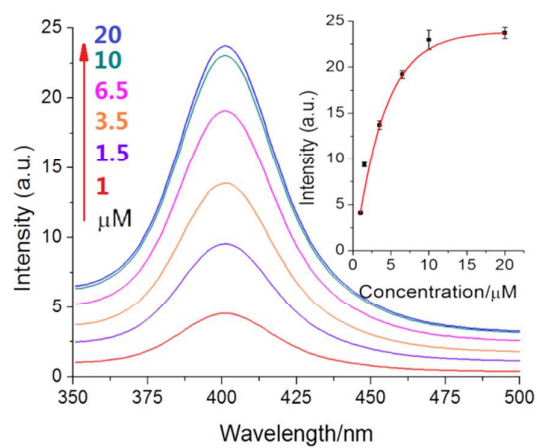
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**Supplementary Figures**

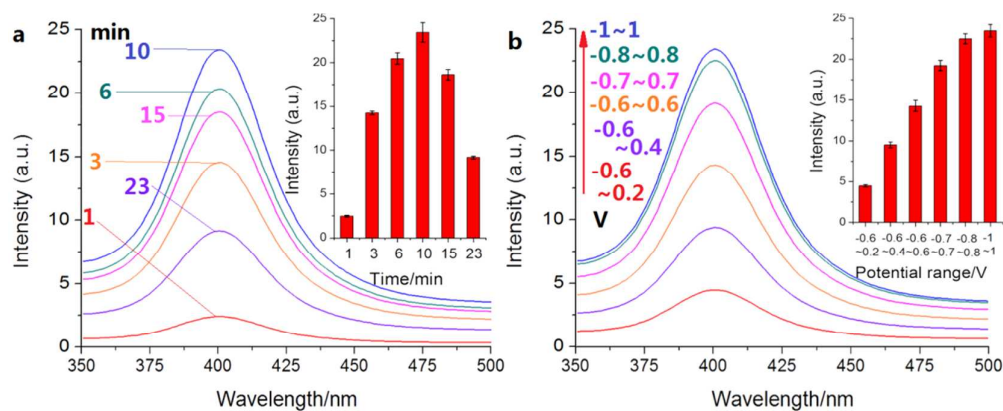
<b>1</b>	<b>Probe sequence</b>	<b>pS-2</b>
<b>2</b>	<b>Condition optimization of major steps of the detection procedure</b>	<b>pS-3~7</b>



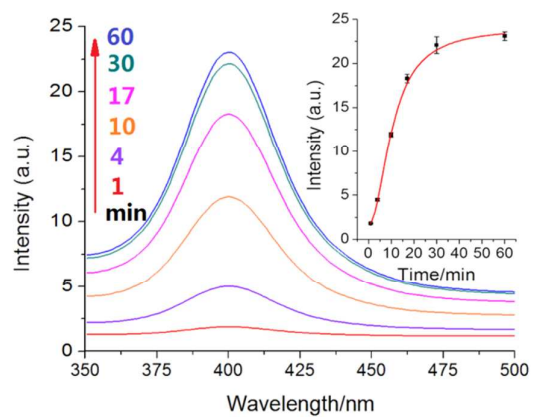
**Figure S1.** The chemical scheme of the peptide sequence as proposed in Scheme 1a.



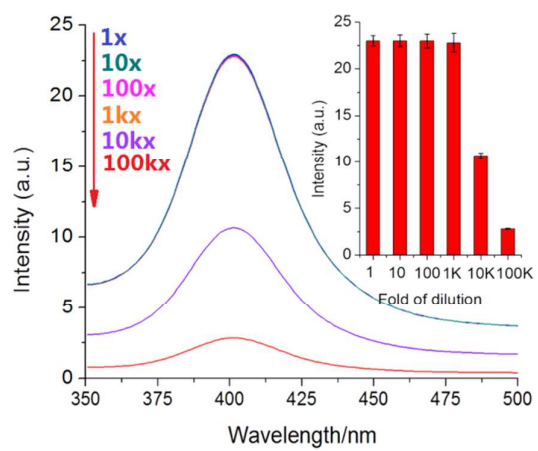
**Figure S2.** Fluorescent response of dityrosine obtained for optimizing the concentration of the peptide probe used for surface immobilization, inset shows the peak response as a function of probe concentration, error bars indicate standard deviation (n=3).



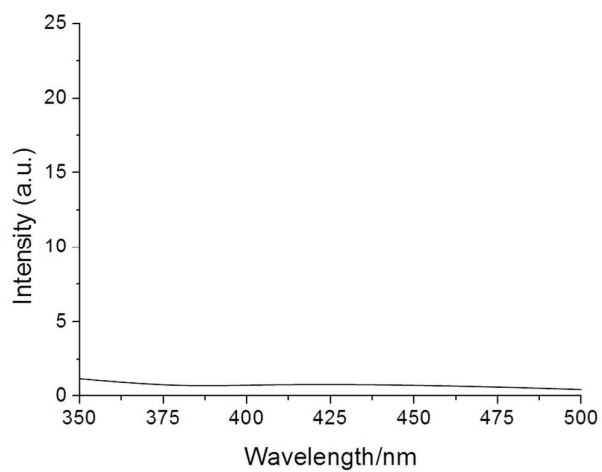
**Figure S3.** Fluorescent response of dityrosine obtained for optimizing the duration (a) and (b) range of potential scanning of the electrochemically controlled probe cleavage and protein cross-linking. Insets shows the peak response varied with the parameter under optimization, error bars indicate standard deviation (n=3).



**Figure S4.** Fluorescent response of dityrosine obtained for optimizing the incubation for probe-target recognition and cross-coupling. The meaning of curves and data points is the same as in Figure S3.



**Figure S5.** Fluorescent response of dityrosine obtained in detecting 10 nM target protein in serially diluted serum samples. Inset shows the peak response varied with the fold of dilution, error bars indicate standard deviation (n=3).



**Figure S6.** Fluorescent response of dityrosine obtained in a control using 100  $\mu$ M synthesized amyloid beta 1~42, with the only tyrosine residual replaced by alanine, as an interfering protein without tyrosine residue, in the detection of 10 nM target protein; no evident signal readout can be observed.