

1 Experimental Procedures

Table S1. AIE-type fluorescent probes for the detection of A β amyloid.

Probe name	Type	Maximum absorption / emission wavelength (nm)	Water-solubility	Emission enhancement (Folds) ¹	Amyloid	K _d (nM) ²	Mapping of A β plaque ³	Reference
Cur-N-BF ₂	<i>Light-up</i>	470 / 572	PBS buffer solution	-	A β ₁₋₄₂ fibrils	-	-	[1]
QM-FN-SO ₃	<i>Off-on</i>	500 / 720	PBS buffer solution	50	A β ₄₂ fibrils	170	in situ brain sections	[2]
FB	<i>Turn-on</i>	565 / 605	PBS buffer solution	20	A β ₁₋₄₂ fibrils	47.91	brain sections	[3]
TM-1	<i>Turn-on</i>	500 / 680	1% DMSO in	106	A β	35	brain	[4]
TM-2		430 / 650	PBS buffer solution	96	aggregates	92	sections	
TMNL	<i>Turn-on</i>	360 / 645	PBS buffer solution	47	A β ₁₋₄₂ fibrils	410.4	in situ brain sections	This probe

¹ "-" meant not mentioned.

² "-" meant not measured.

³ "-" meant not mentioned.

Table S2. The selectivity comparison of AIE-type fluorescent probes for the detection of A β amyloid.

Probe name	Graph	Selectivity	Reference
Cur-N-BF ₂	<p>PL intensity (au)</p> <p>Aβ fibrils, HSA, Transferrin, Insulin, Lysozyme, Pepsin, Trypsin</p>	A β ₁₋₄₂ fibrils	[1]
QM-FN-SO ₃	<p>High selectivity</p> <p>I₆₆₀ nm</p> <p>1 peanut agglutinin, 2 pepsin, 3 lysozyme, 4 tyrosinase, 5 α-KA, 6 D-(+)-mannose, 7 D-galactose, 8 Leu, 9 Glu, 10 Phe, 11 Pro, 12 Thr, 13 Tro, 14 Tyr</p>	A β ₄₂ fibrils	[2]
FB	<p>(a) Relative FL (%)</p> <p>Probe, Aβ aggregates, Aβ monomer, Aβ oligomer, BSA, HSA</p> <p>(b) Fluorescence intensity (a.u.)</p> <p>Blank, Aβ aggregates, Ala, Arg, Asp, Glu, Cys, Ile, Lys, Met, GSH</p>	A β ₁₋₄₂ fibrils	[3]
TM-1 TM-2	<p>Normalized FL intensity</p> <p>A</p> <p>1 Control, 2 Glycine, 3 Alanine, 4 Leucine, 5 Valine, 6 Proline, 7 prion, 8 Aβ₄₂ aggregates</p> <p>B</p> <p>1 Control, 2 Glycine, 3 Alanine, 4 Leucine, 5 Valine, 6 Proline, 7 prion, 8 Aβ₄₂ aggregates</p>	A β aggregates	[4]

Table S3. The molecular structures of the corresponding probes in Table S1-S2.

Probe name	Structure
Cur-N-BF ₂	
QM-FN-SO ₃	
FB	
TM-1	
TM-2	

2 ^1H NMR, ^{13}C NMR and HRMS Spectrum of New Compounds

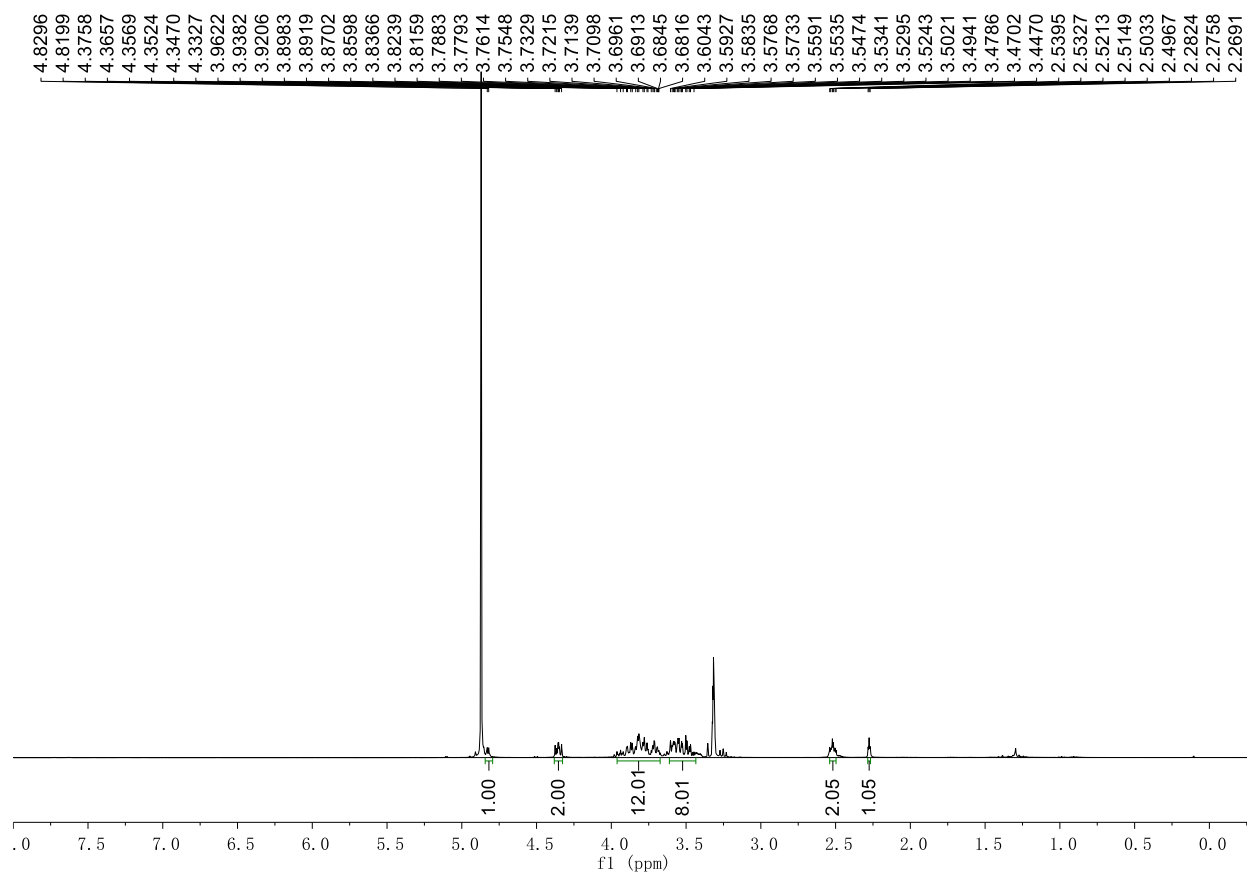


Figure S1. ^1H NMR spectra of compound 4.

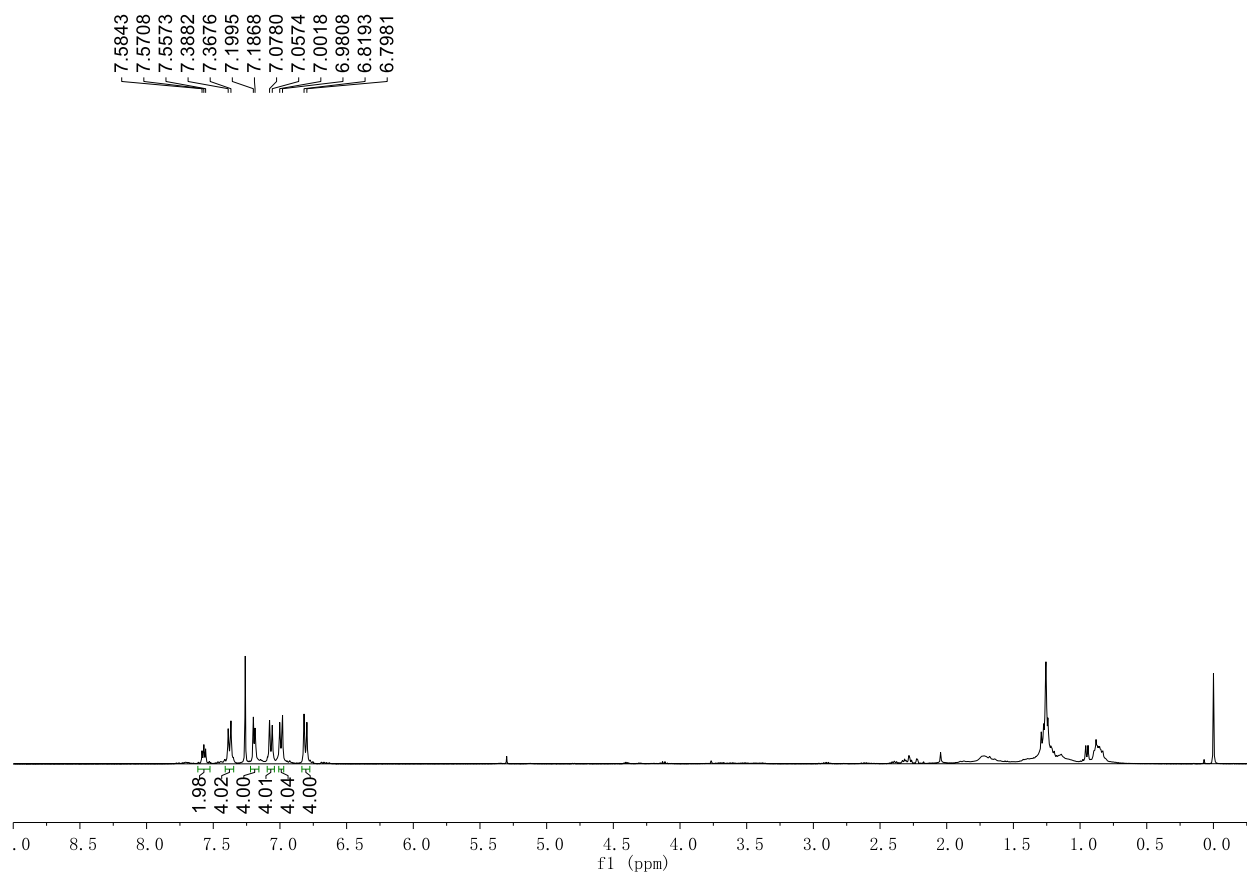


Figure S2. ^1H NMR spectra of compound 13.

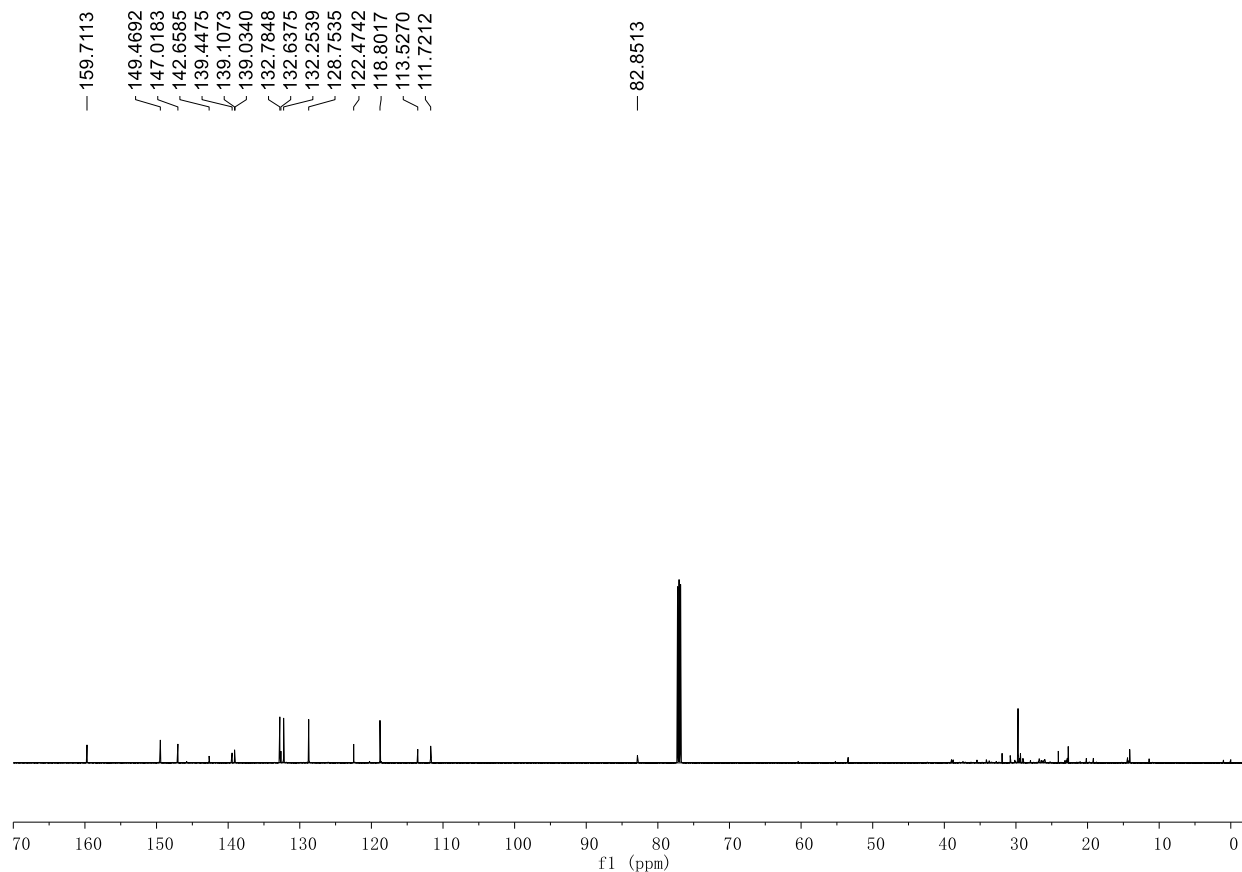


Figure S3. ^{13}C NMR spectra of compound 13.

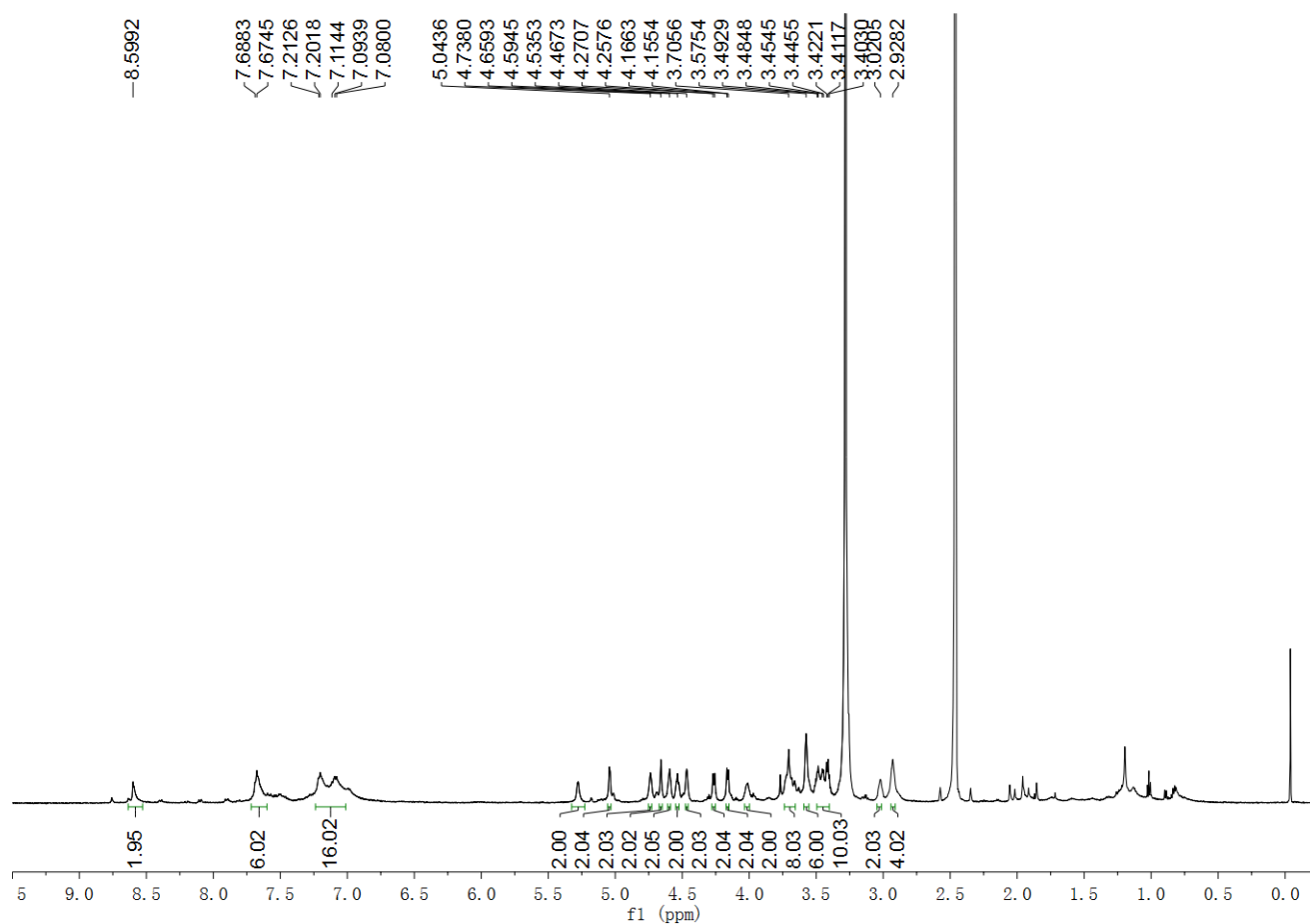


Figure S4. ^1H NMR spectra of TMNL.

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Sample Name: NM-C-2CN-1009
Sample Number: N/A

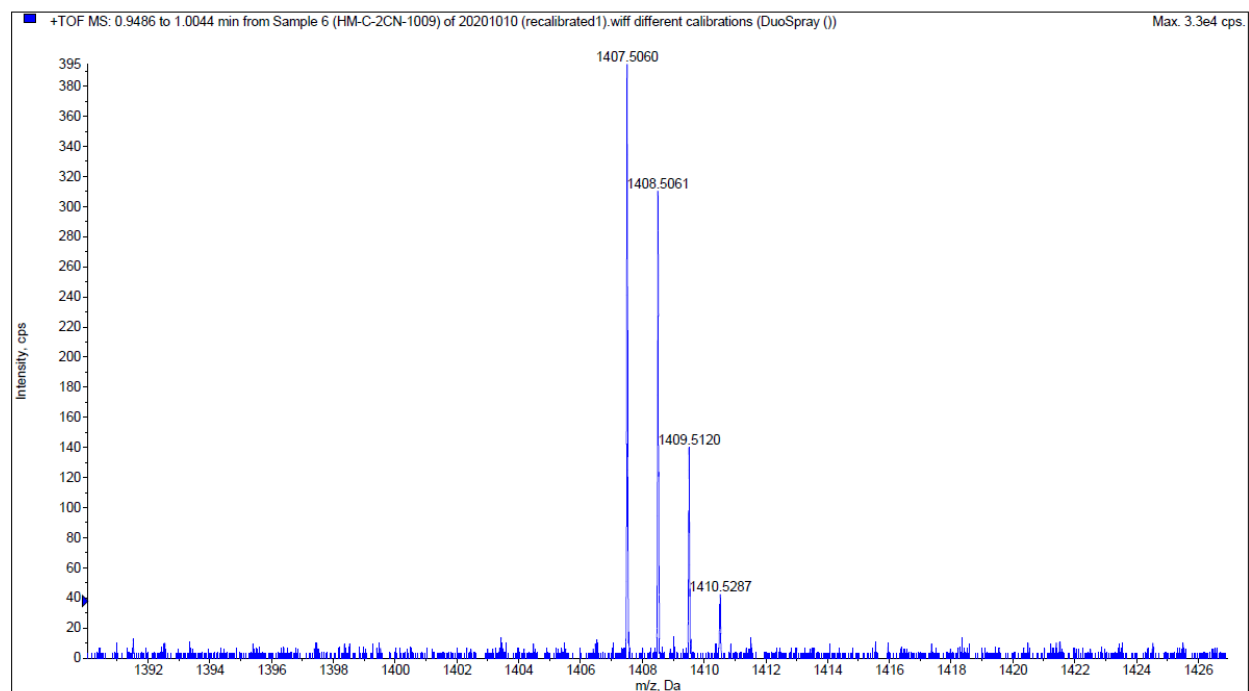


Figure S5. HRMS spectra of TMNL.

3 Reference

- [1] Yang Y.; Li S.; Zhang Q.; Kuang Y.; Qin A.; Gao M.; Li F.; Tang B.Z. An AIE-active theranostic probe for light-up detection of A β aggregates and protection of neuronal cells. *J. Mater. Chem. B* **2019**, 7, 2434-2441.
- [2] Fu W.; Yan C.; Guo Z.; Zhang J.; Zhang H.; Tian H.; Zhu W.H. Rational Design of Near-Infrared Aggregation-Induced-Emission-Active Probes: In Situ Mapping of Amyloid- β Plaques with Ultrasensitivity and High-Fidelity. *J. Am. Chem. Soc.* **2019**, 141, 3171-3177.
- [3] Wang Y.; Qiu Y.; Sun A.; Xiong Y.; Tan H.; Shi Y.; Yu P.; Roy G.; Zhang L.; Yan J. Dual-functional AIE fluorescent probes for imaging β -amyloid plaques and lipid droplets. *Anal. Chim. Acta* **2020**, 1133, 109-118.
- [4] Xu M.; Li R.; Li X.; Lv G.; Li S.; Sun A.; Zhou Y.; Yi T. NIR fluorescent probes with good water-solubility for detection of amyloid beta aggregates in Alzheimer's disease. *J. Mater. Chem. B* **2019**, 7, 5535-5540.