

Supporting Information for

**H₂SO₄-Catalyzed One-Step Synthesis of
β,β-Carbon-Bridged bisBODIPYs from BODIPYs
and Aldehydes**

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1. Figure S1

BODIPYs **B1-3** (Figure S1) were synthesized according to literature procedures.¹⁻³

Compounds **3a-h** (Figure S1) are commercial reagents.

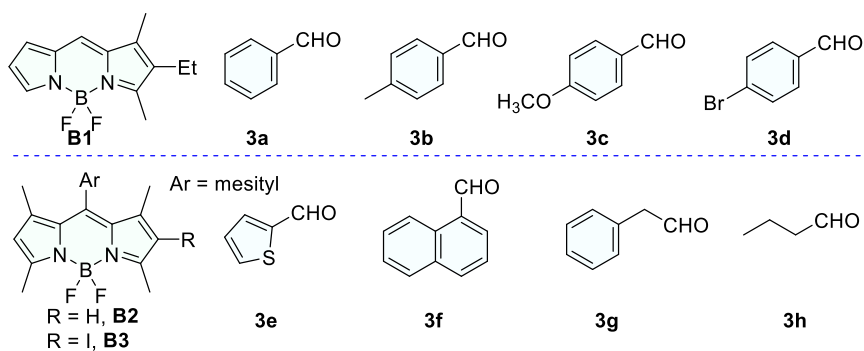
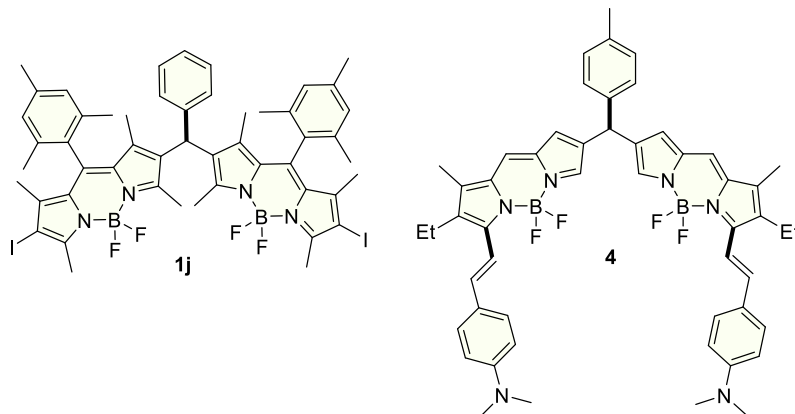


Figure S1. Chemical structure of BODIPYs **B1-3** and various aldehydes **3a-h**

2. Photophysical properties

2.1 Table S1: Photophysical properties of **1j** and **4** in different solvents at room temperature.



dyes	solvent	$\lambda_{\text{abs}}^{\text{max}}$ (nm)	$\epsilon_{\text{abs}}^{\text{max}}$ [$\text{M}^{-1} \text{cm}^{-1}$] ^a	$\lambda_{\text{em}}^{\text{max}}$ (nm)	Φ^{b}	SS (cm^{-1}) ^c
1j	cyclohexane	549	184800	558	0.310	290
	Toluene	551	166300	563	0.300	390
	THF	548	163500	562	0.280	450
	CH ₃ CN	545	166200	557	0.097	400
	DMSO	551	141500	563	0.093	390
4	cyclohexane	638	90400	651	0.077	310
	Toluene	649	155600	678	0.380	660
	THF	645	140500	691	0.015	820
	CH ₃ CN	644	123300	717	0.007	1600
	DMSO	657	123400	726	0.005	1450

^aMolar absorption coefficient values rounded to the nearest $100 \text{ M}^{-1} \text{ cm}^{-1}$.

^bFluorescence quantum yields determined using Rhodamine B ($\Phi = 0.49$ in ethanol) as reference.

^cStokes shift values rounded to nearest 10 cm^{-1} .

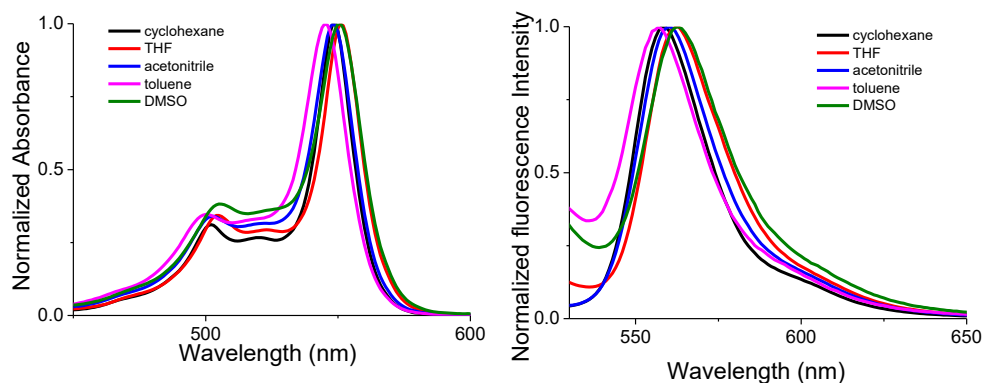


Figure S2. Absorption (left) and fluorescence emission (right) spectra of **1j** recorded in different solvents (excitation at 500 nm).

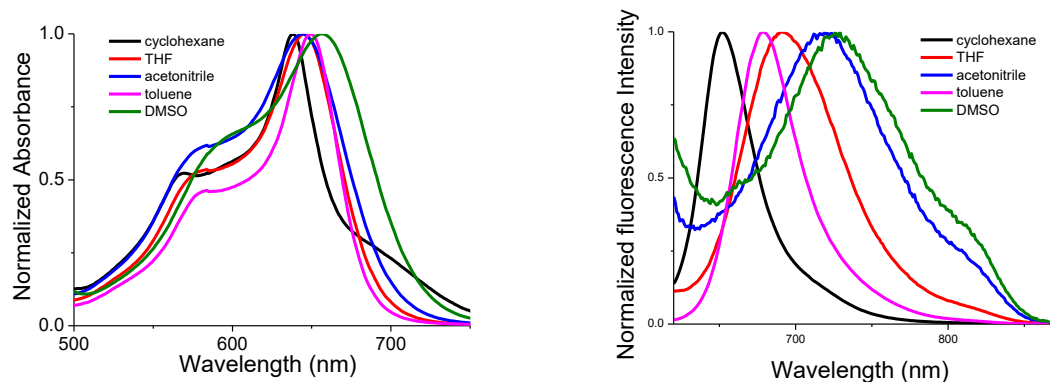


Figure S3. Absorption (left) and fluorescence emission (right) spectra of **4** recorded in different solvents (excitation at 600 nm).

2.2 UV-Vis absorption and fluorescence emission spectra in CH₂Cl₂.

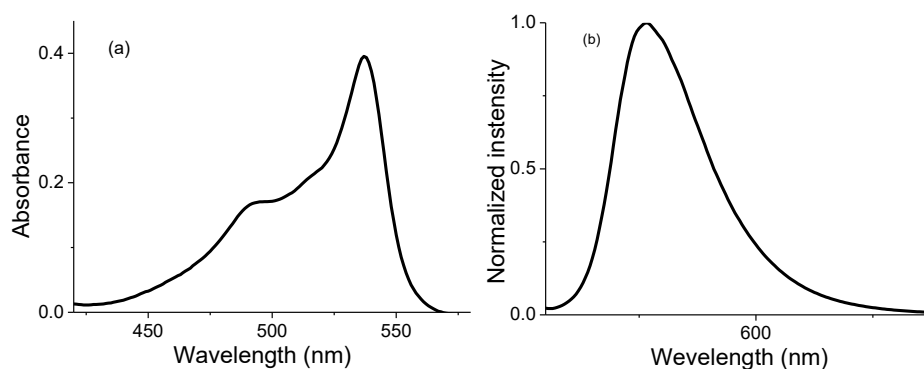


Figure S4. Absorption (a) and fluorescence emission (b) spectra of **1a** recorded in CH₂Cl₂ (excitation at 500 nm).

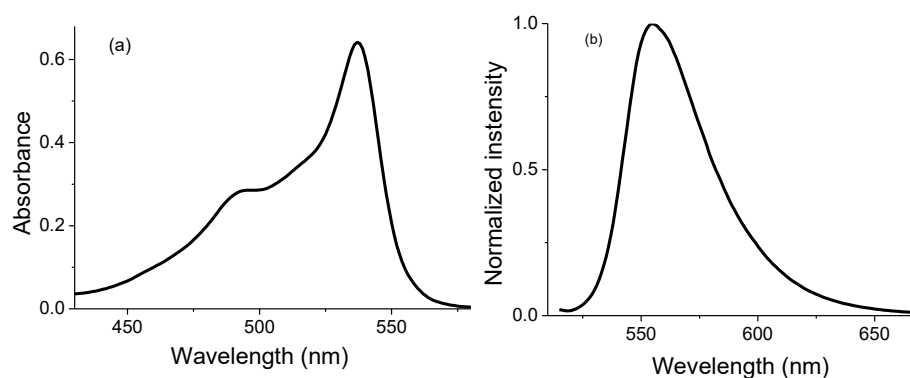


Figure S5. Absorption (a) and fluorescence emission (b) spectra of **1b** recorded in CH₂Cl₂ (excitation at 500 nm).

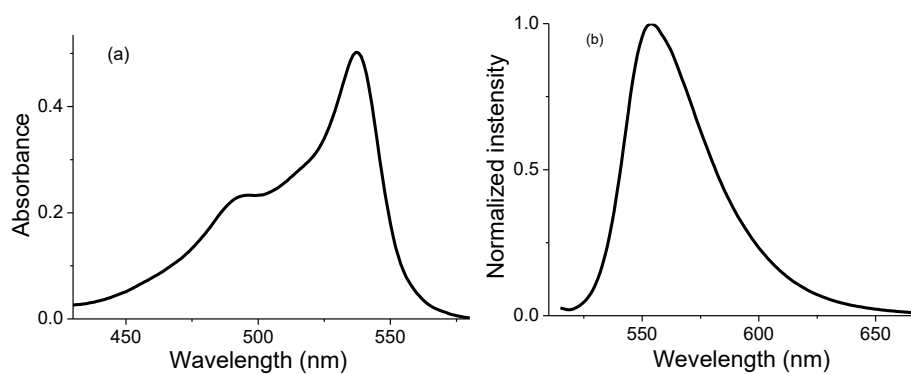


Figure S6. Absorption (a) and fluorescence emission (b) spectra of **1c** recorded in CH₂Cl₂ (excitation at 500 nm).

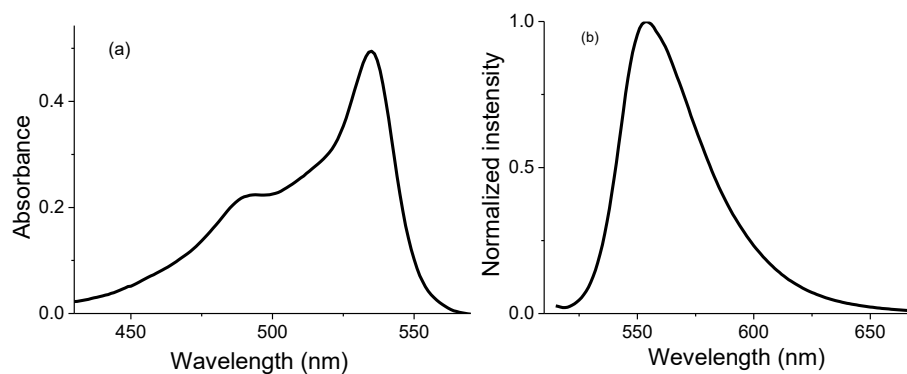


Figure S7. Absorption (a) and fluorescence emission (b) spectra of **1d** recorded in CH_2Cl_2 (excitation at 500 nm).

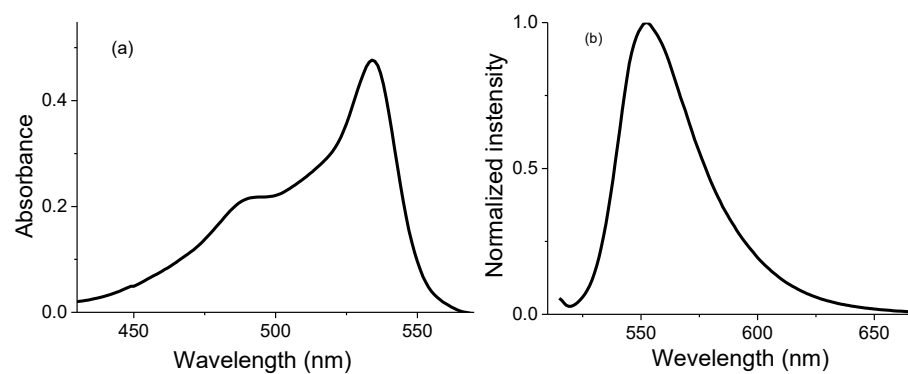


Figure S8. Absorption (a) and fluorescence emission (b) spectra of **1e** recorded in CH_2Cl_2 (excitation at 500 nm).

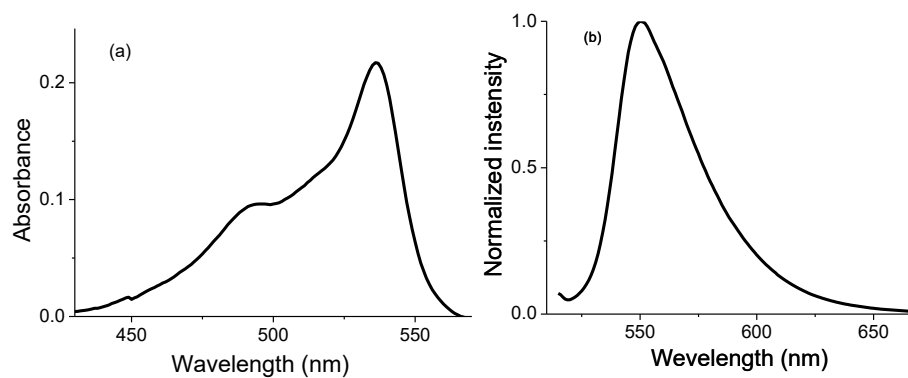


Figure S9. Absorption (a) and fluorescence emission (b) spectra of **1f** recorded in CH_2Cl_2 (excitation at 500 nm).

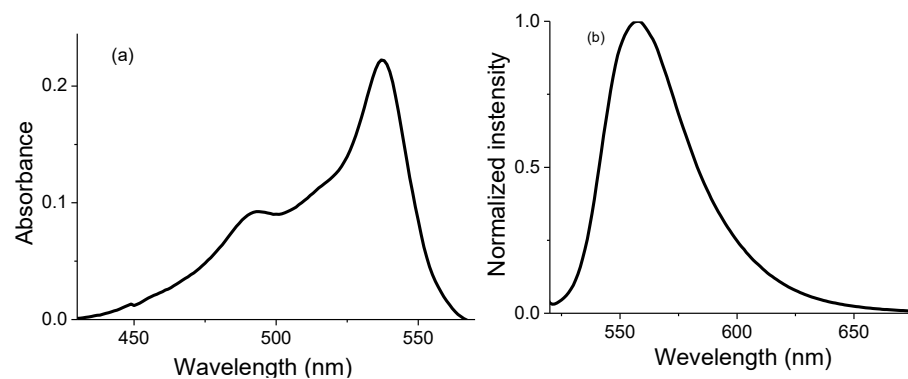


Figure S10. Absorption (a) and fluorescence emission (b) spectra of **1g** recorded in CH₂Cl₂ (excitation at 500 nm).

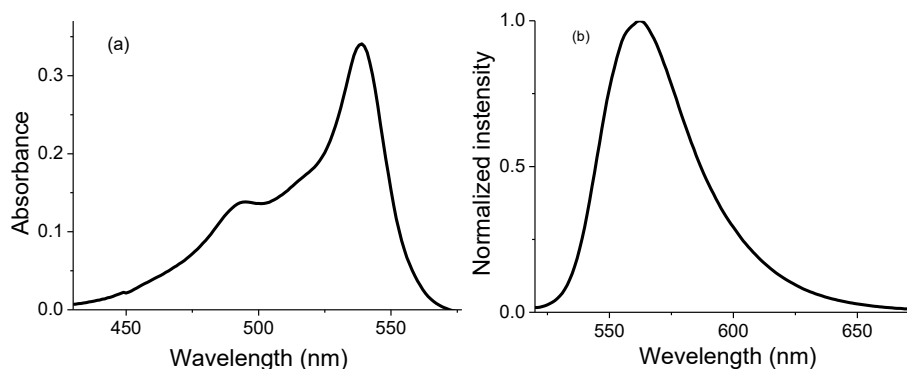


Figure S11. Absorption (a) and fluorescence emission (b) spectra of **1h** recorded in CH₂Cl₂ (excitation at 500 nm).

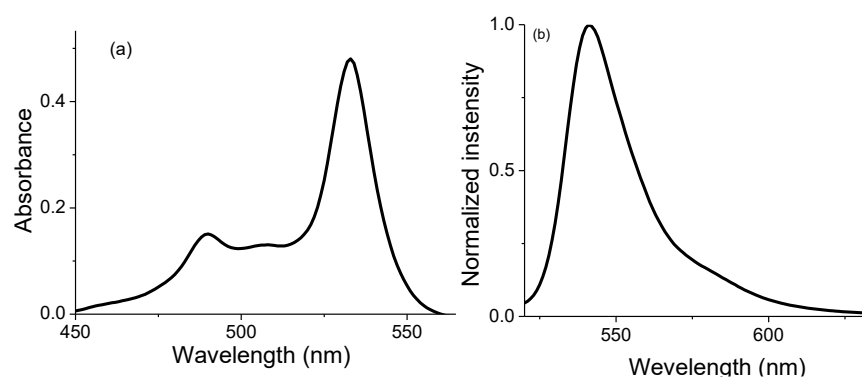


Figure S12. Absorption (a) and fluorescence emission (b) spectra of **1i** recorded in CH₂Cl₂ (excitation at 500 nm).

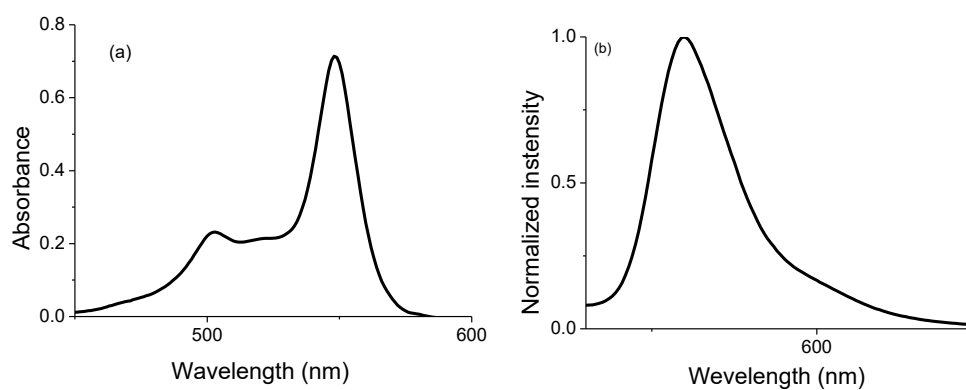


Figure S13. Absorption (a) and fluorescence emission (b) spectra of **1j** recorded in CH₂Cl₂ (excitation at 500 nm).

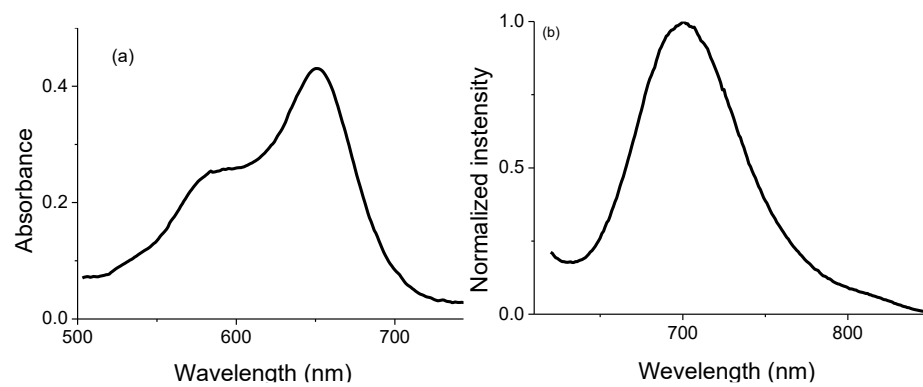


Figure S14. Absorption (a) and fluorescence emission (b) spectra of **4** recorded in CH_2Cl_2 (excitation at 600 nm).

3. Singlet oxygen generation efficiency

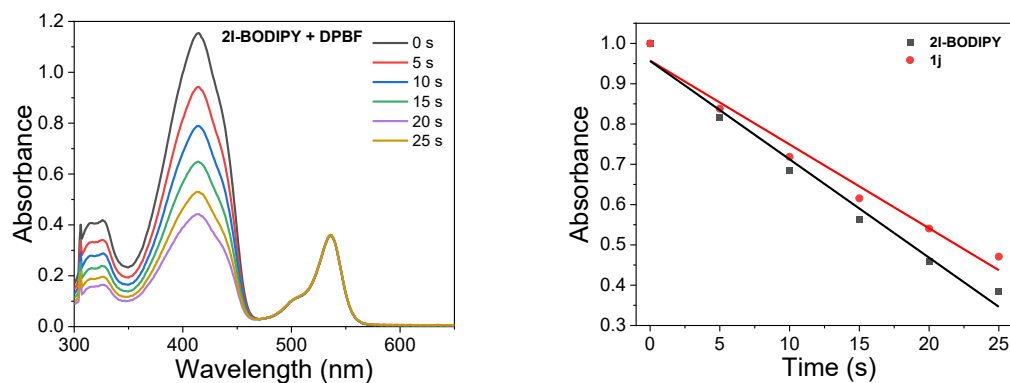
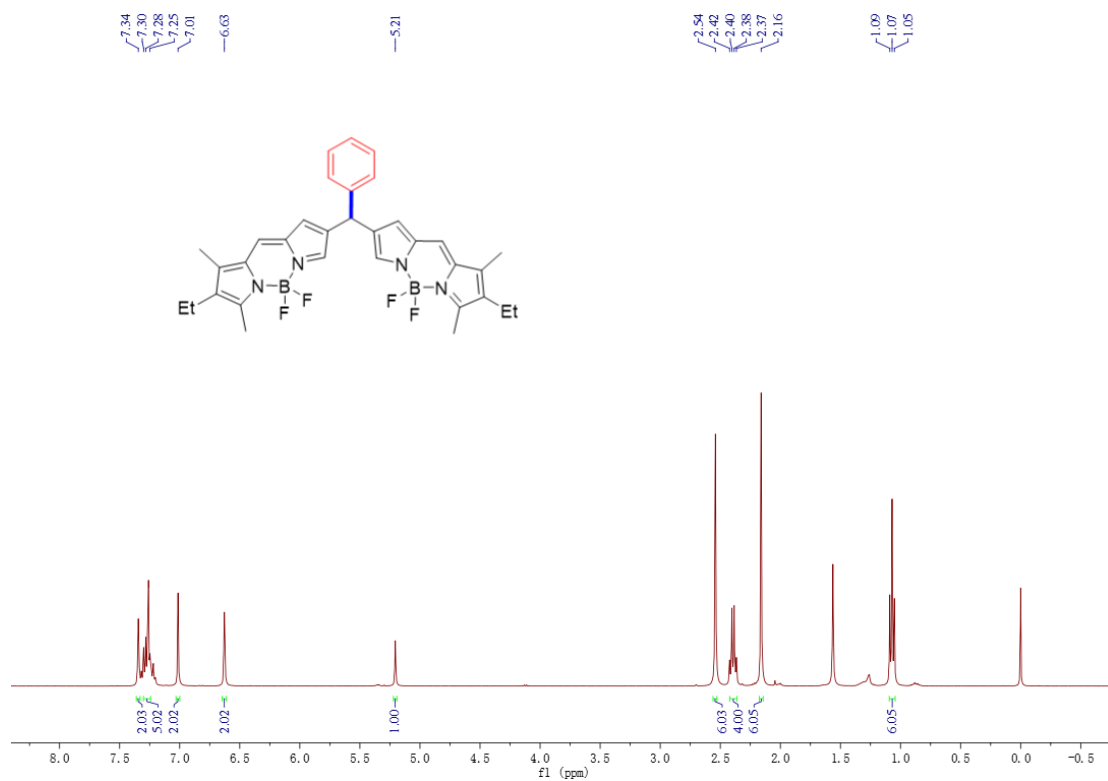


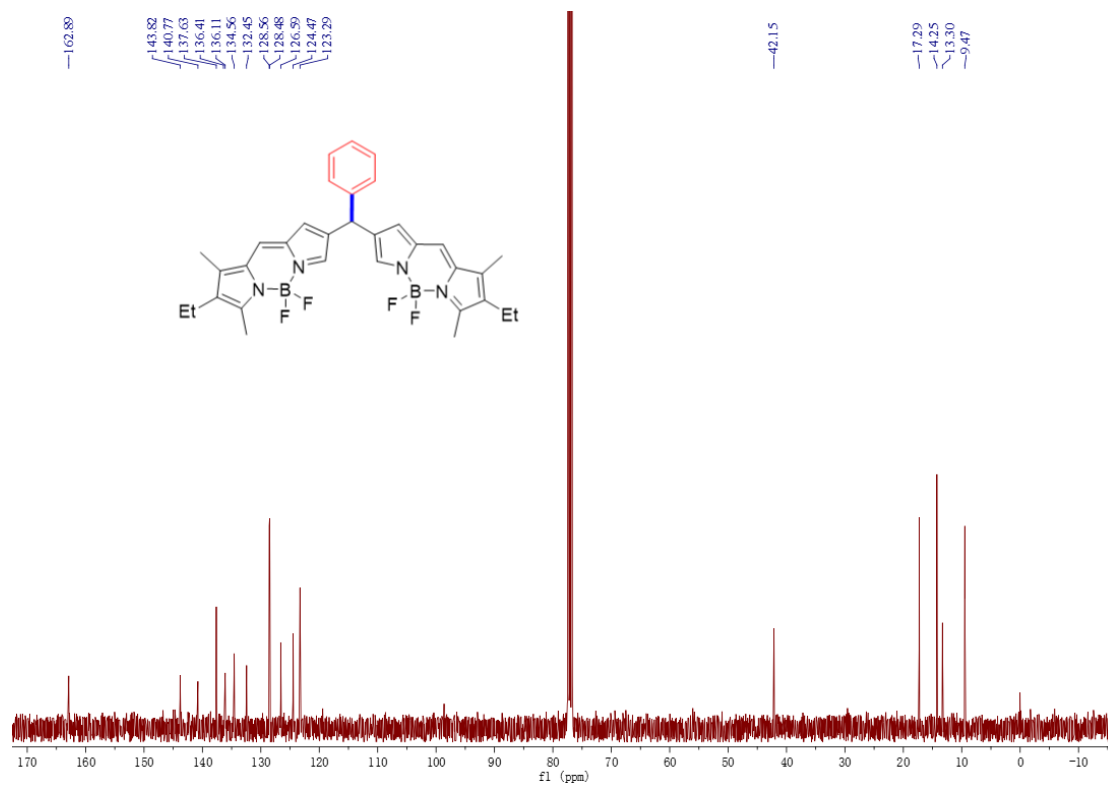
Figure S15. (a) Changes in the absorption spectrum of **DPBF** upon irradiation in the presence of **2I-BODIPY** in toluene (recorded at 5 s intervals). (b) Plot of change in absorbance of DPBF at 410 nm vs irradiation time in the presence of bisBODIPY **1j** in toluene against **2I-BODIPY** as the standard in toluene.

4. ^1H , ^{13}C NMR and HRMS spectra of all new compounds

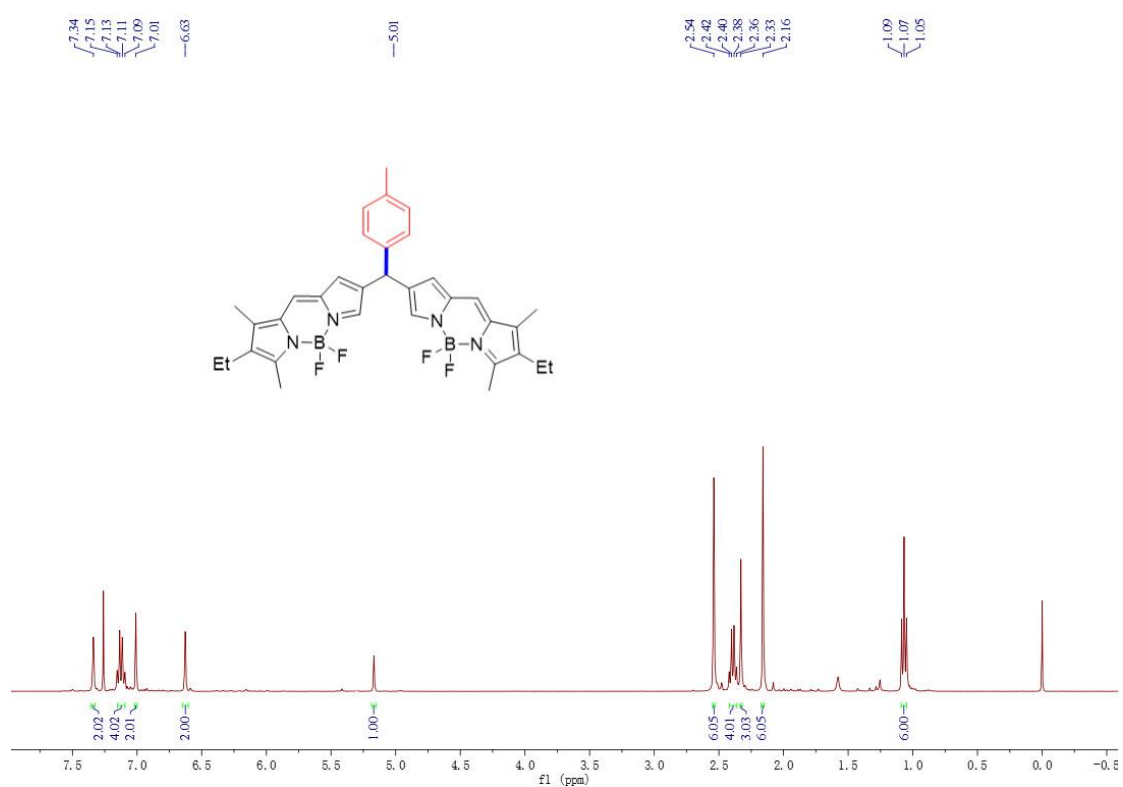
^1H NMR spectrum of **1a** in CDCl_3



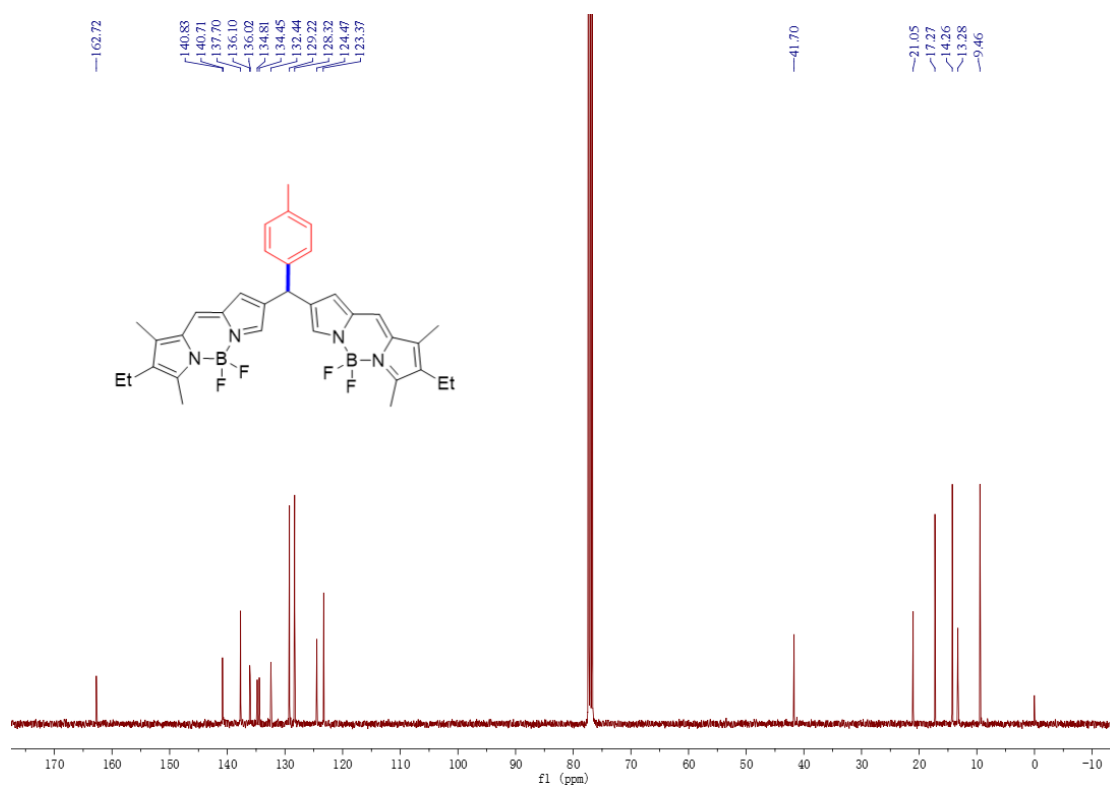
^{13}C NMR spectrum of **1a** in CDCl_3



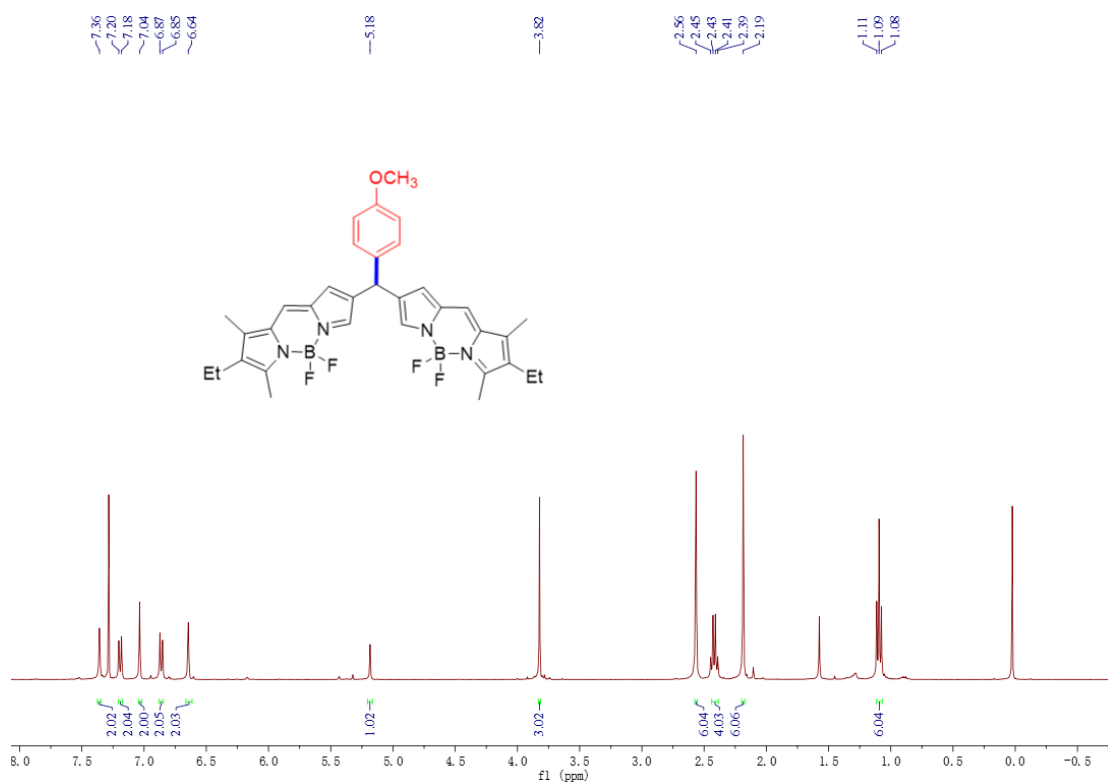
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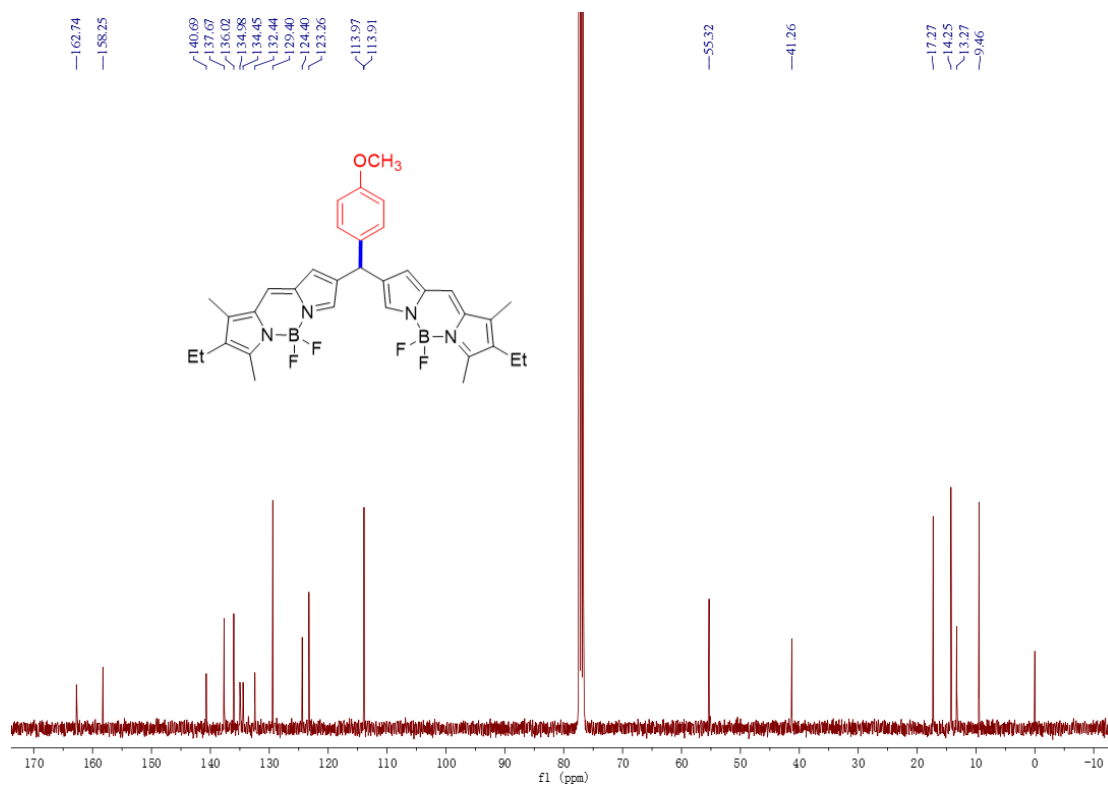
^{13}C NMR spectrum of **1b** in CDCl_3



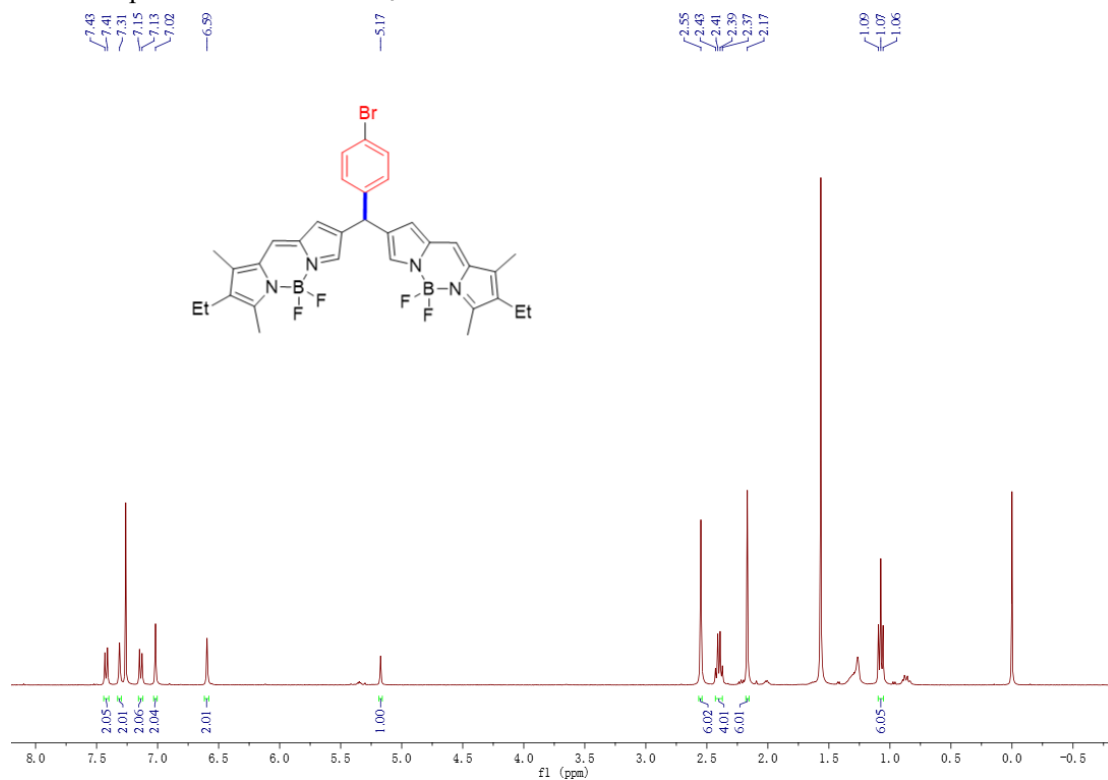
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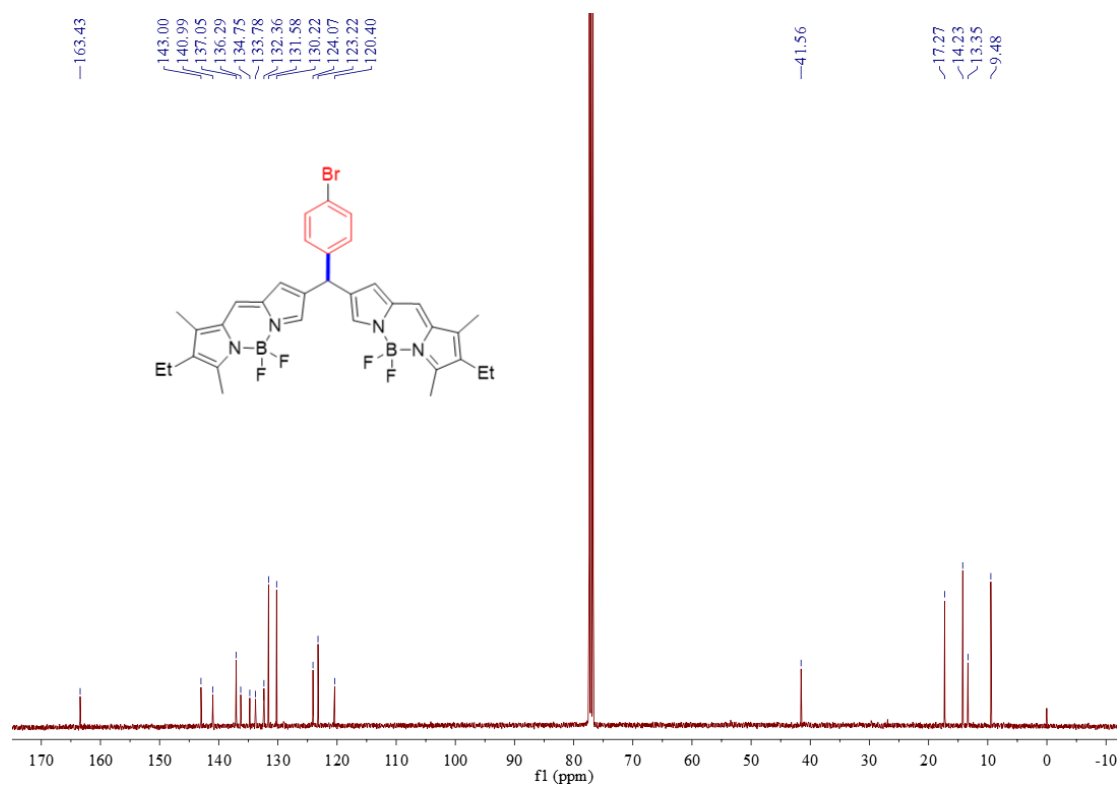
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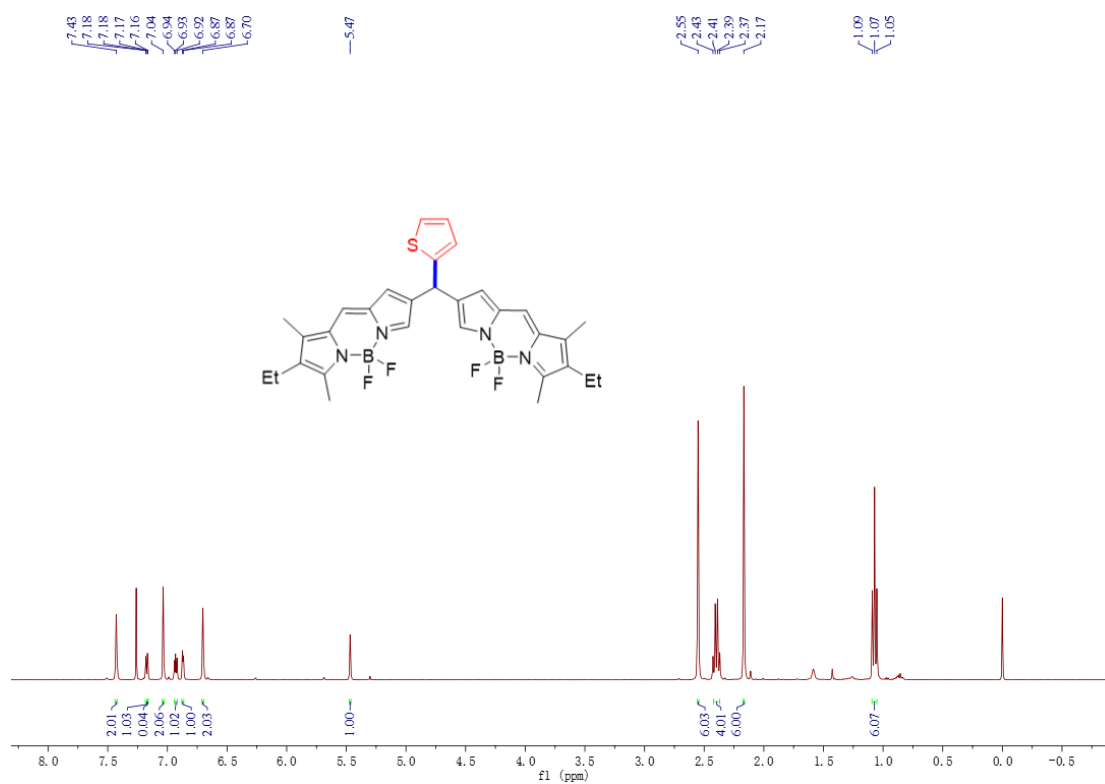
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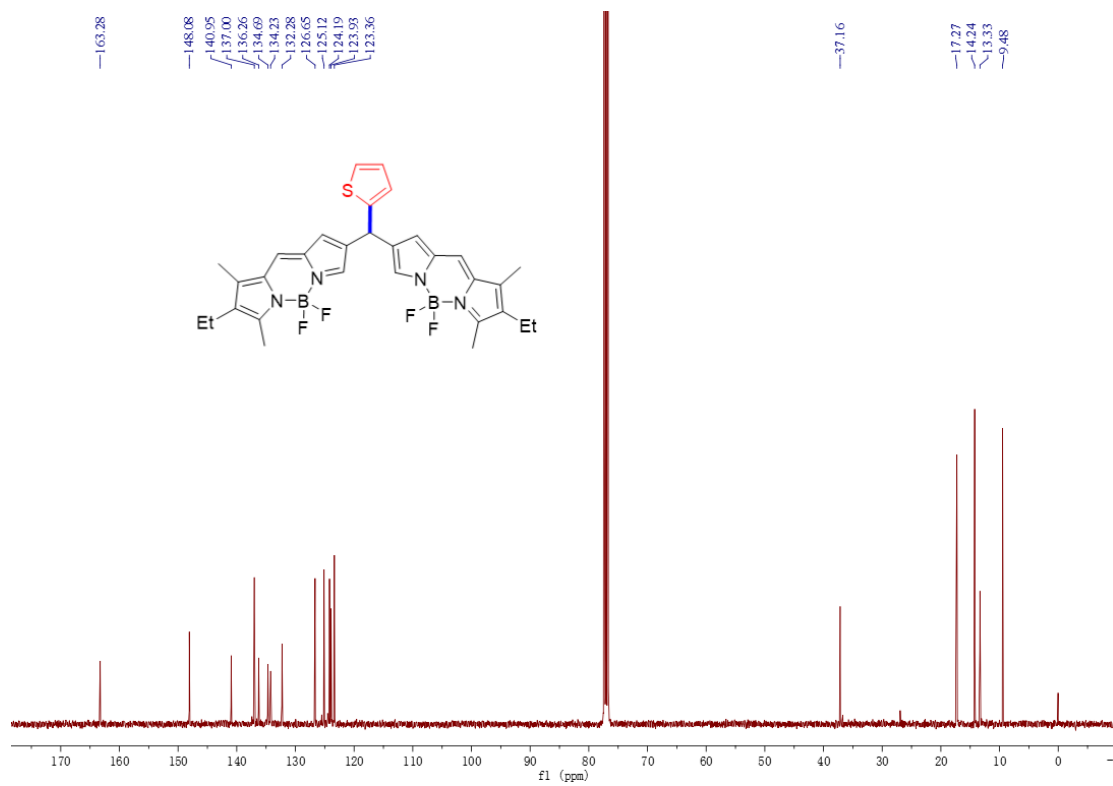
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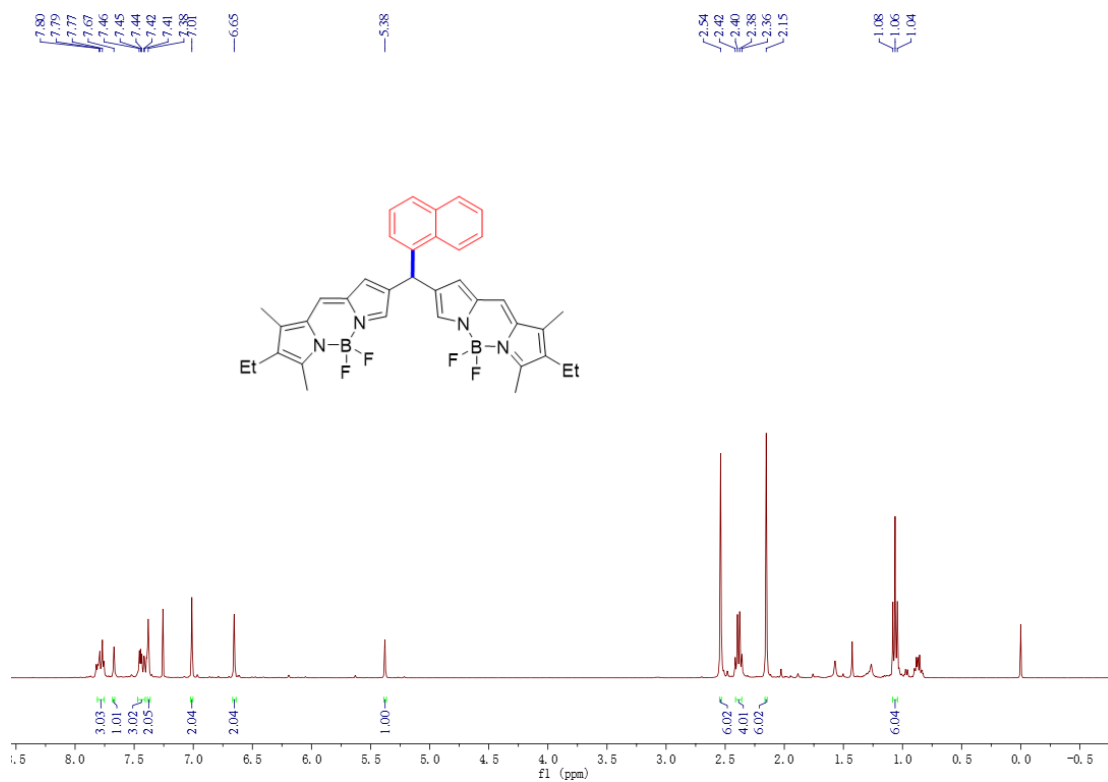
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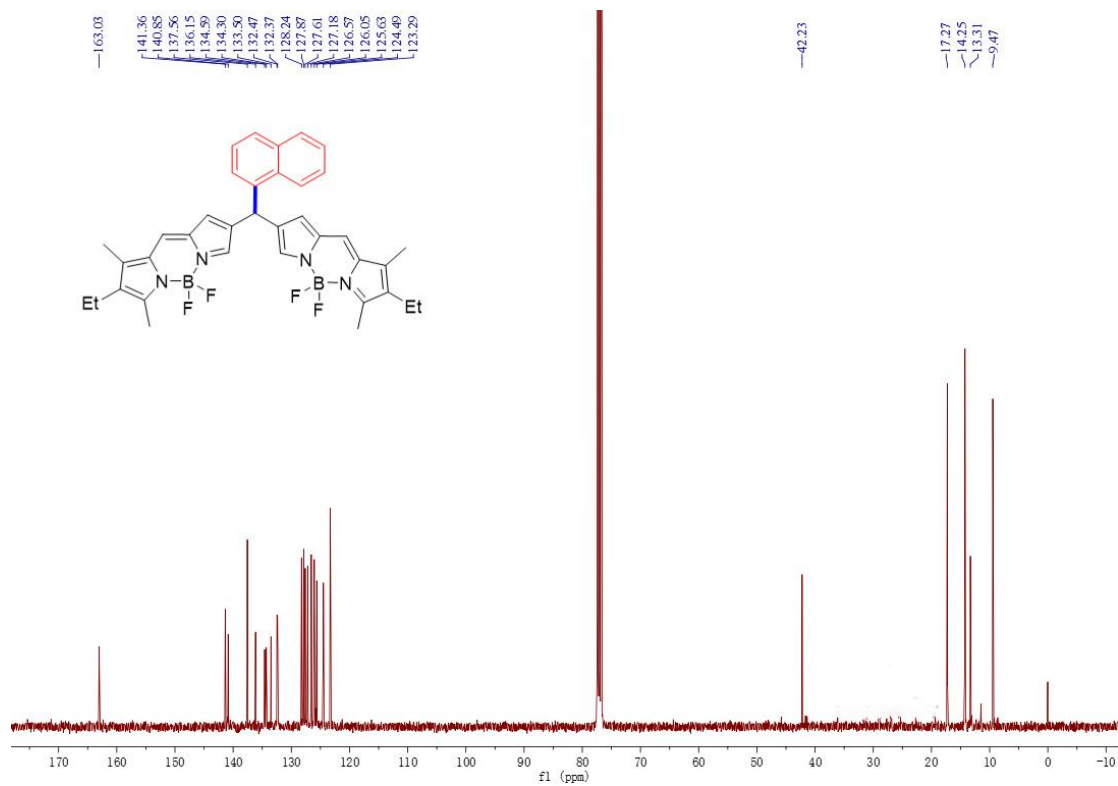
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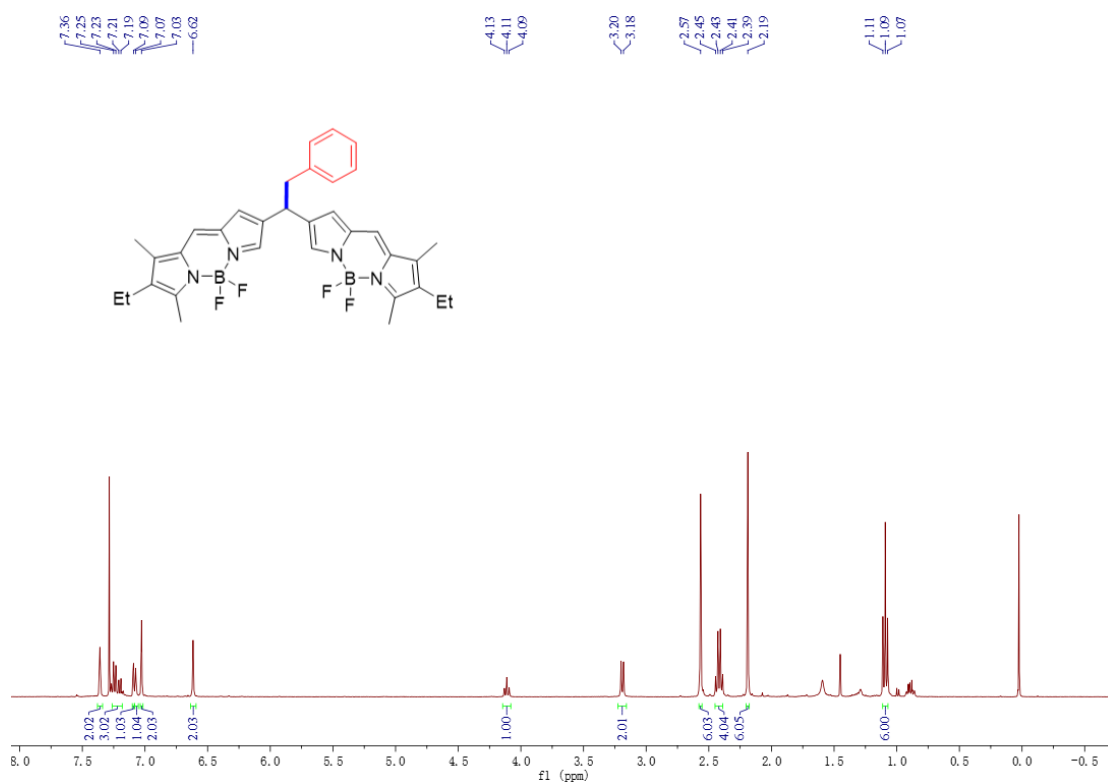
^1H NMR spectrum of **1f** in CDCl_3



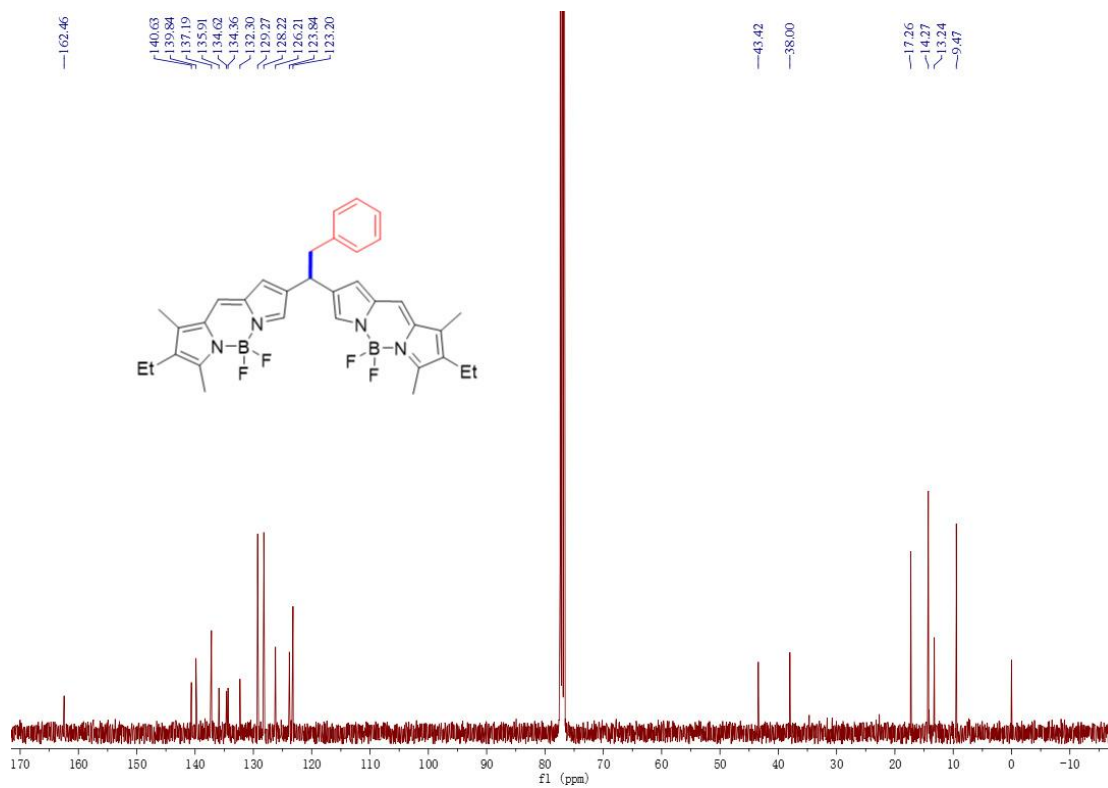
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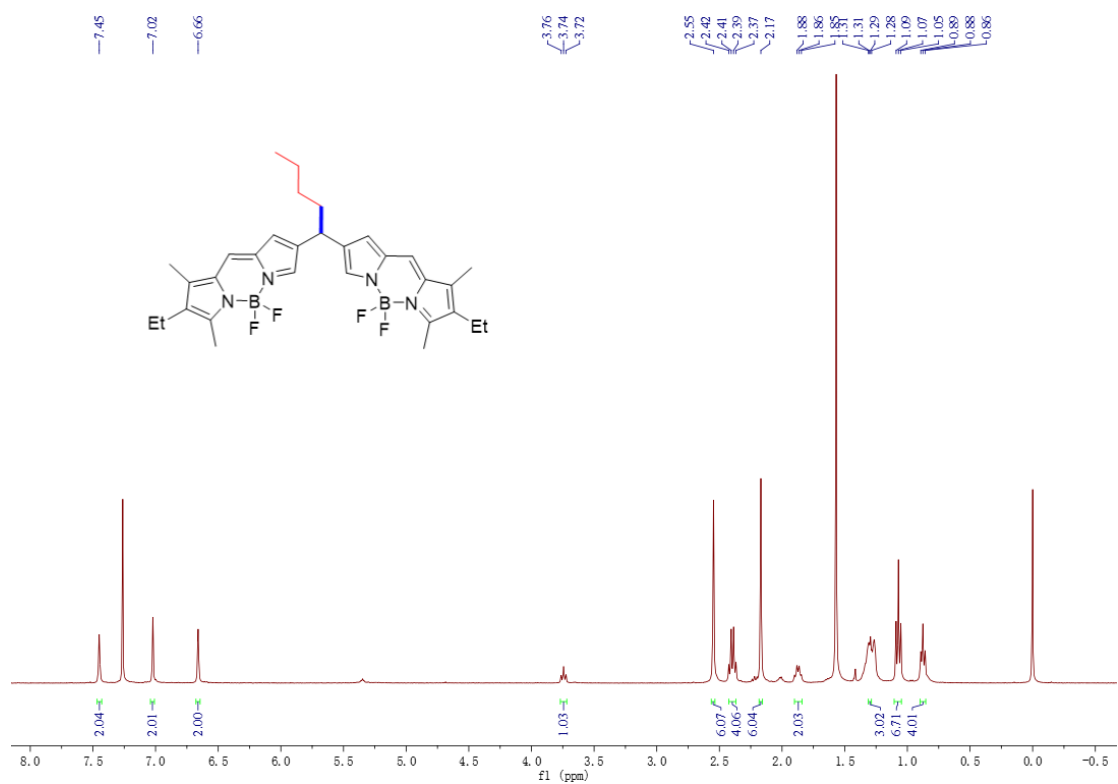
^1H NMR spectrum of **1g** in CDCl_3



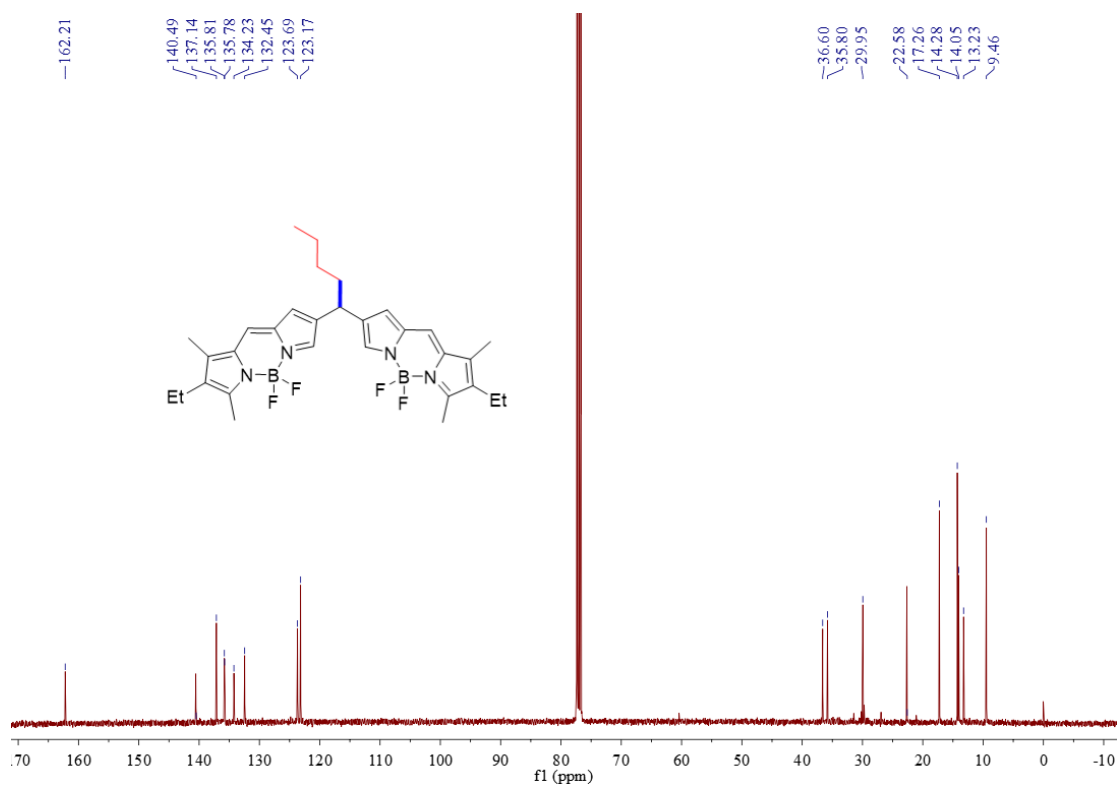
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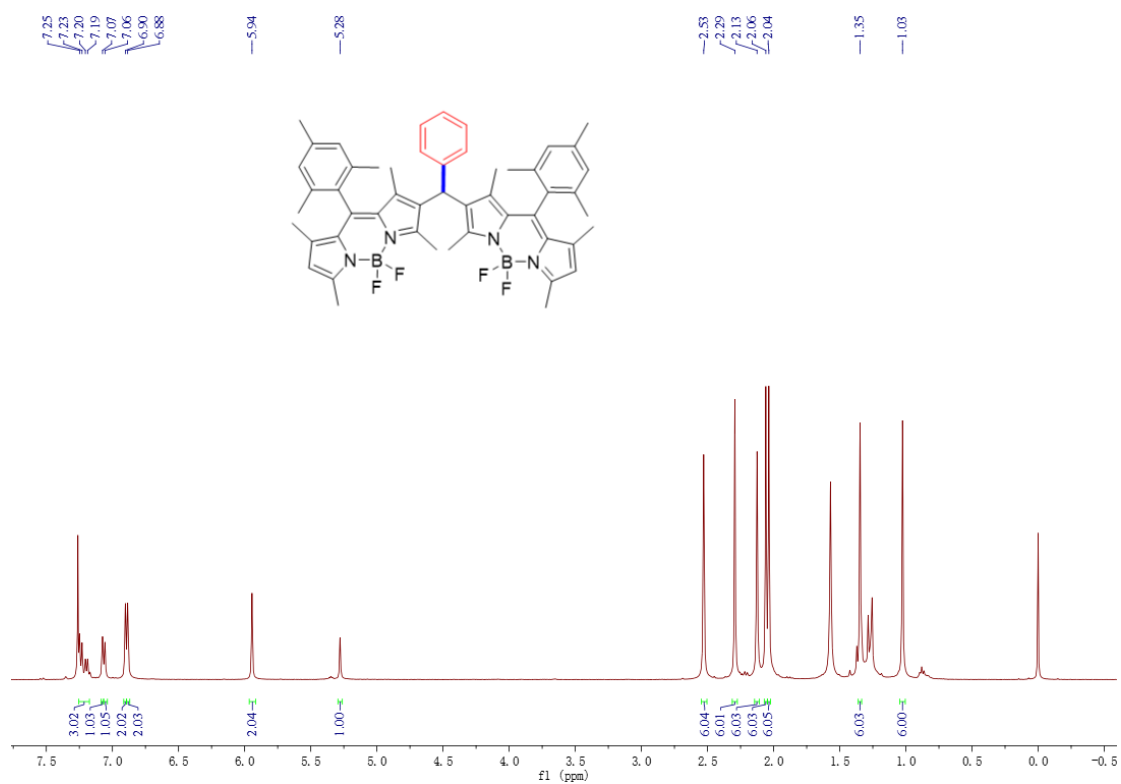
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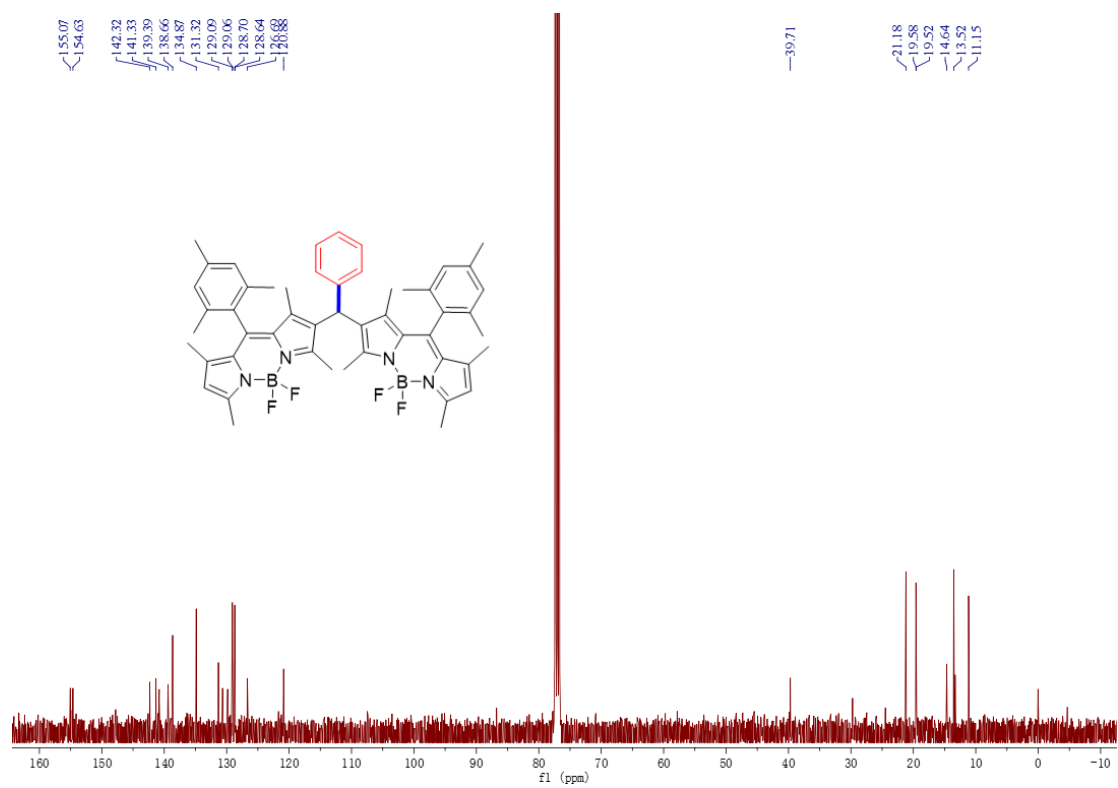
^{13}C NMR spectrum of **1h** in CDCl_3



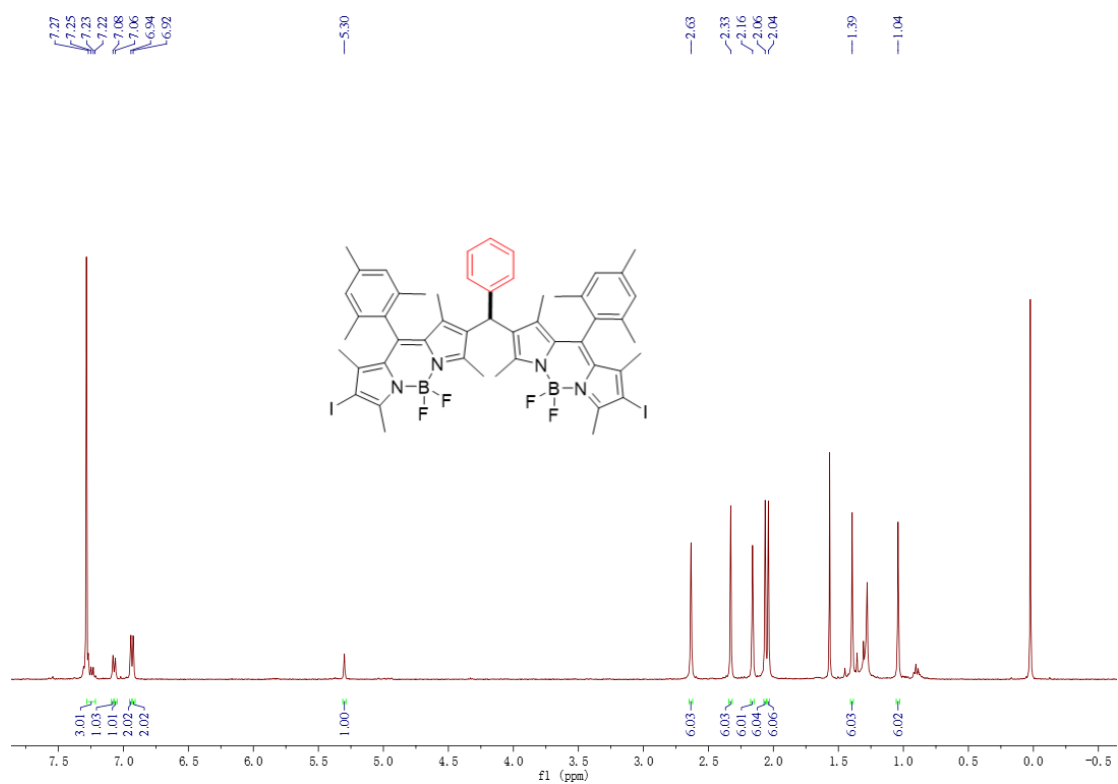
^1H NMR spectrum of **1i** in CDCl_3



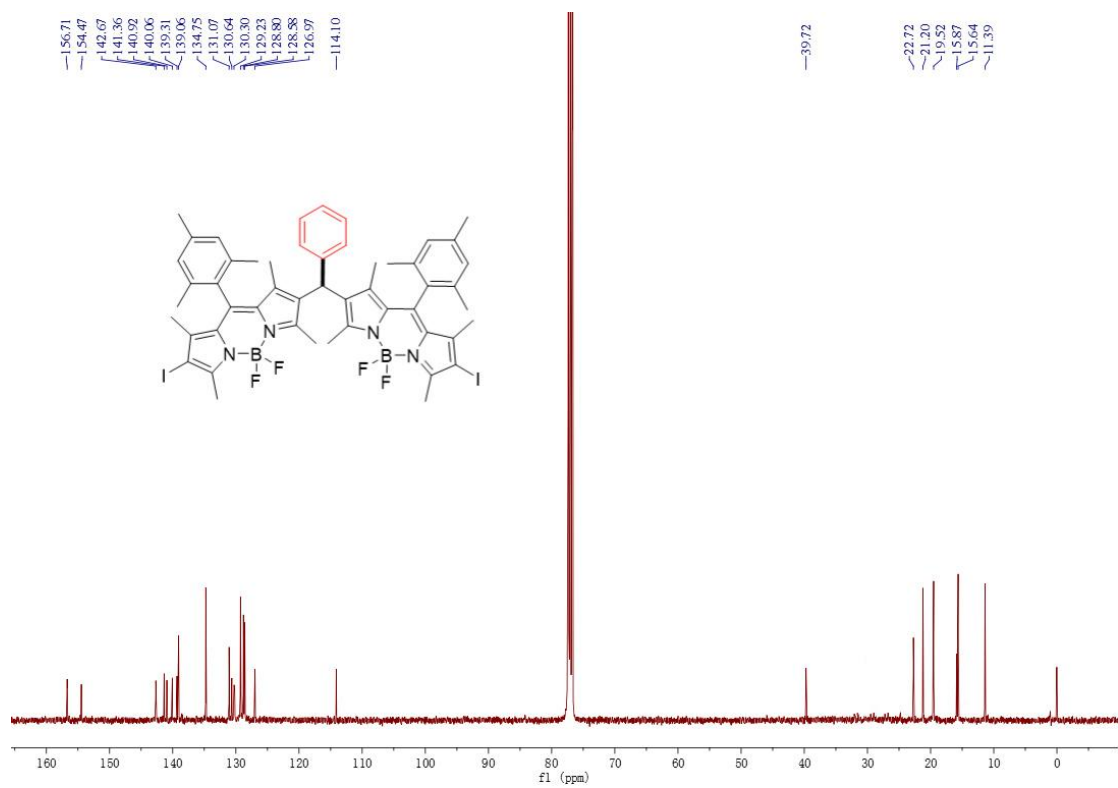
^{13}C NMR spectrum of **1i** in CDCl_3



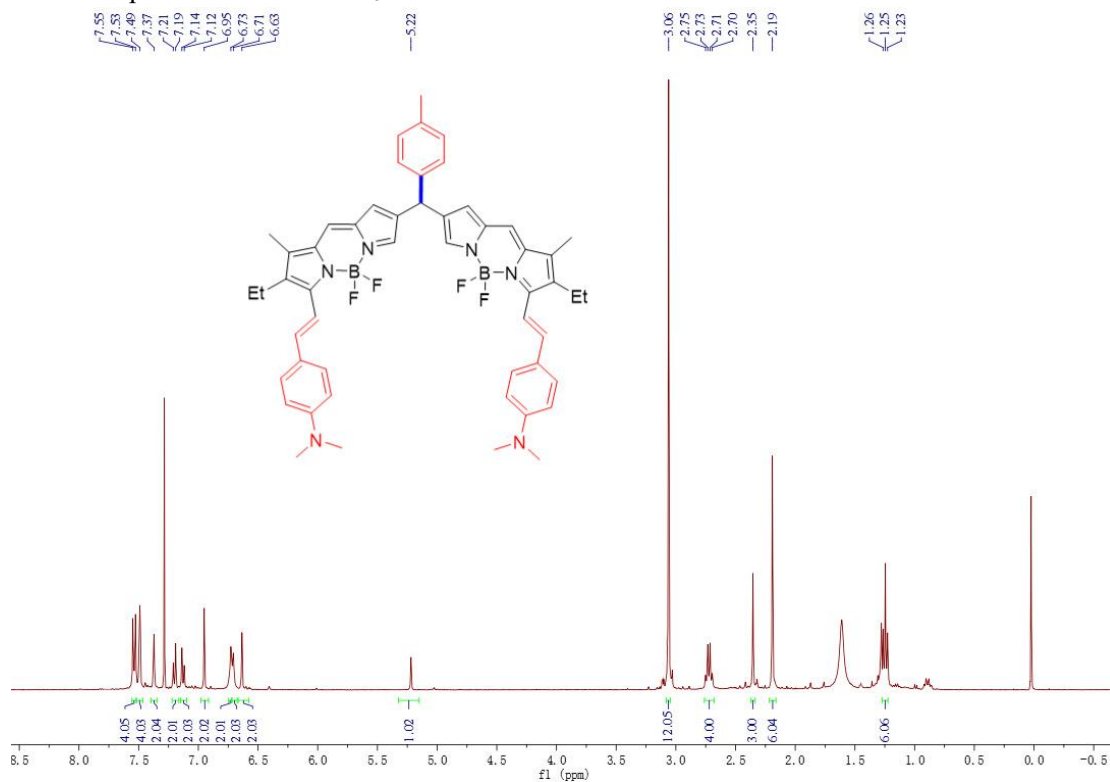
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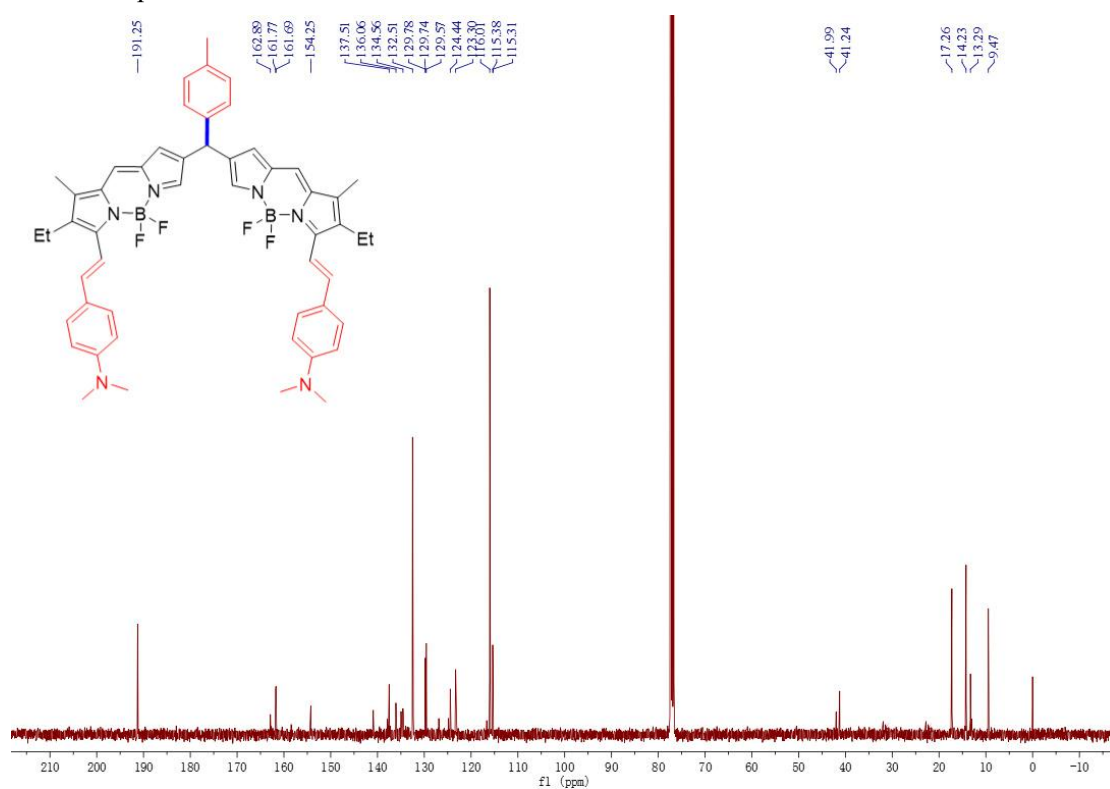
^{13}C NMR spectrum of **1j** in CDCl_3



¹H NMR spectrum of **4** in CDCl₃

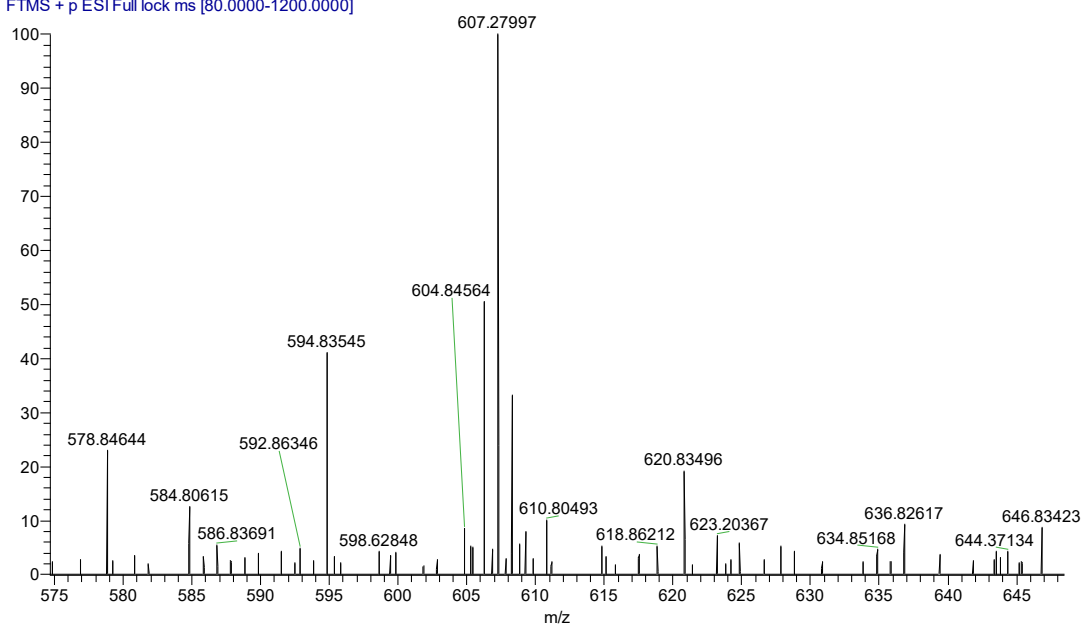


¹³C NMR spectrum of **4** in CDCl₃

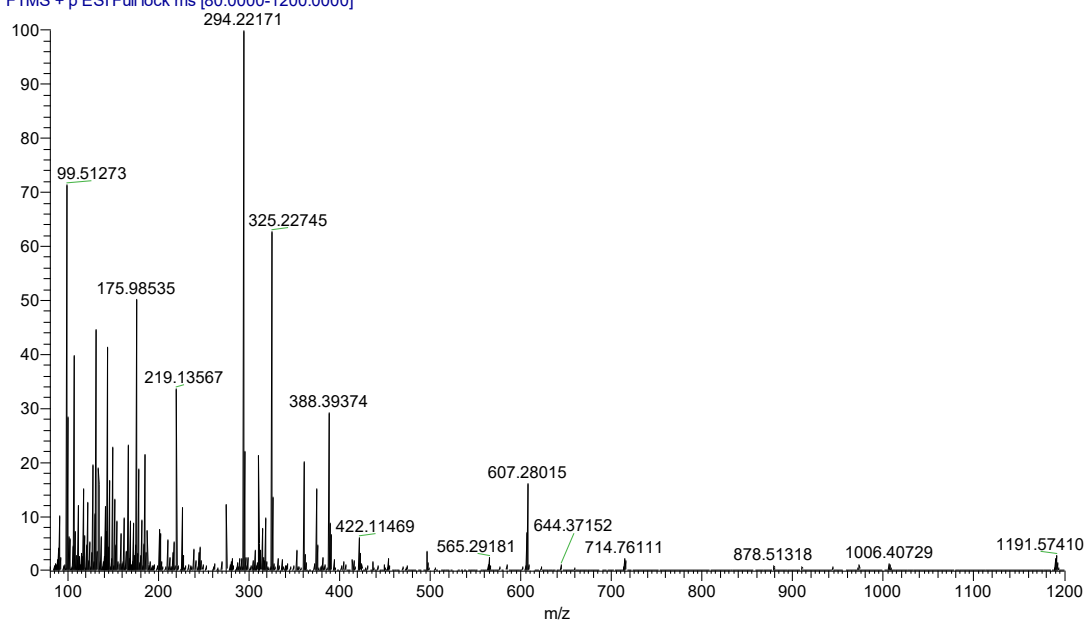


HRMS spectra of 1a

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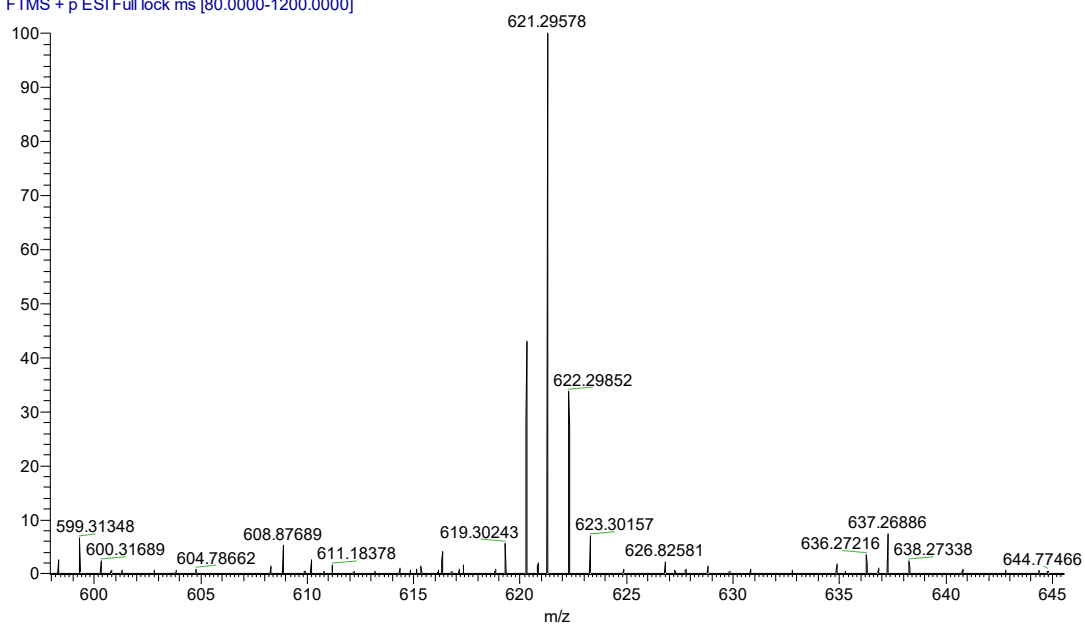


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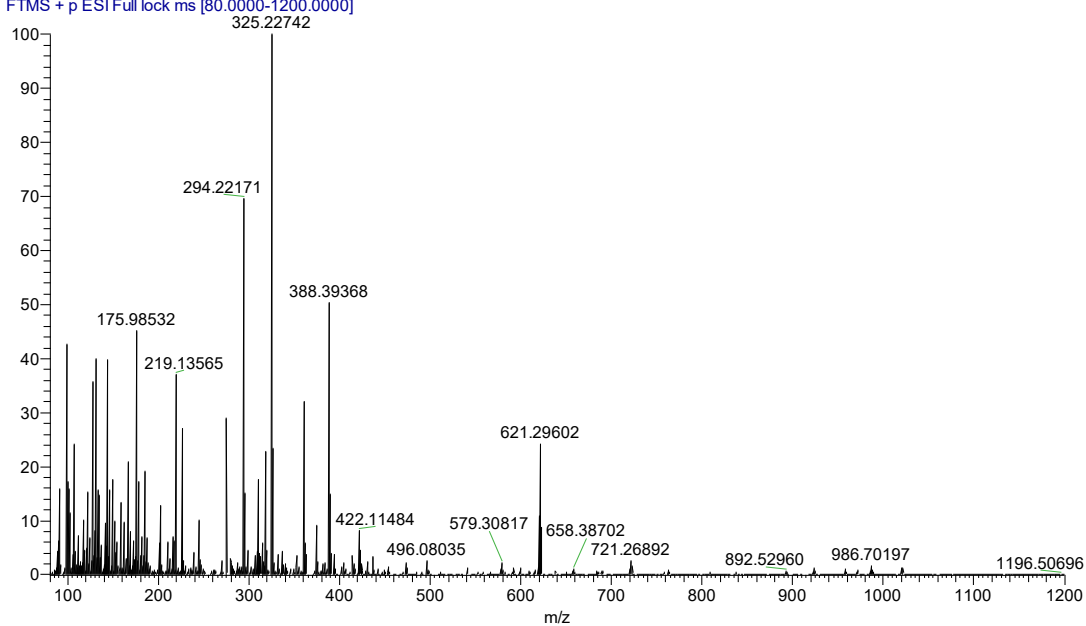


HRMS spectra of **1b**

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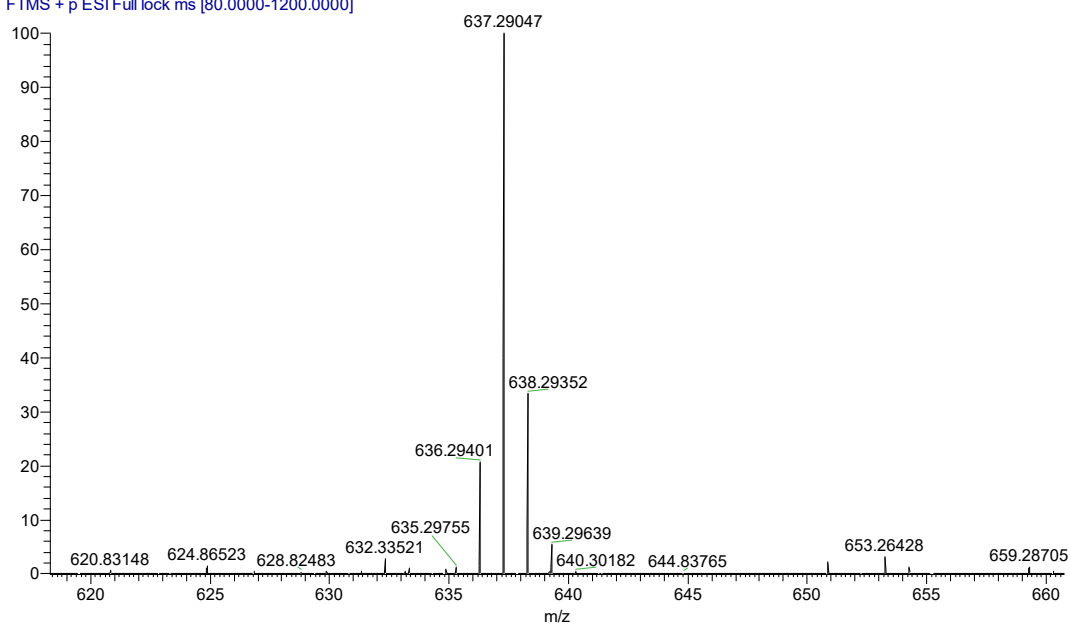


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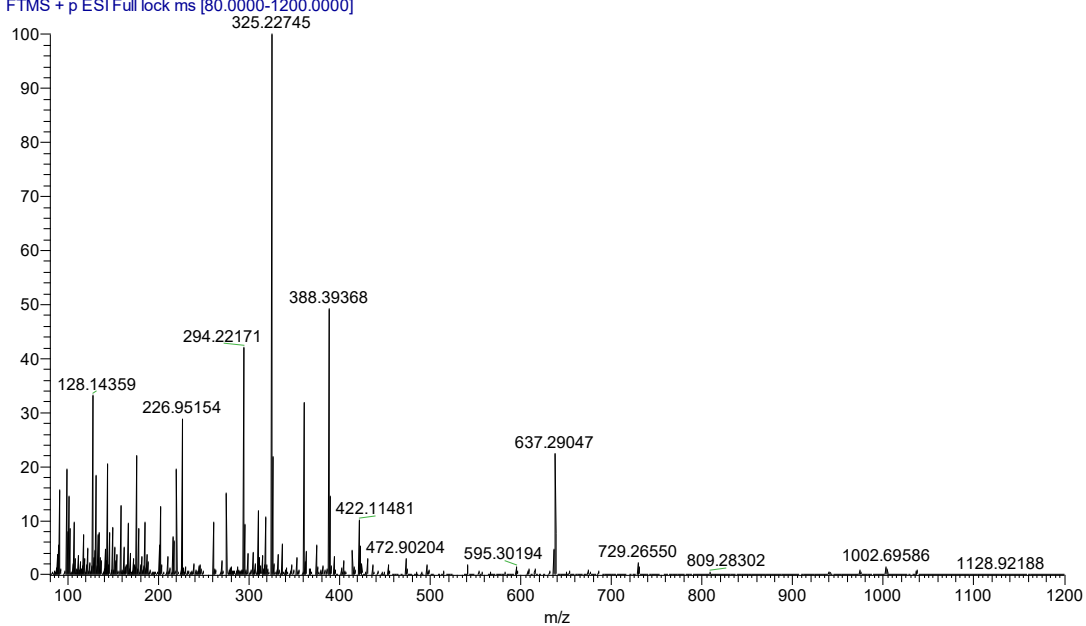


HRMS spectra of 1c

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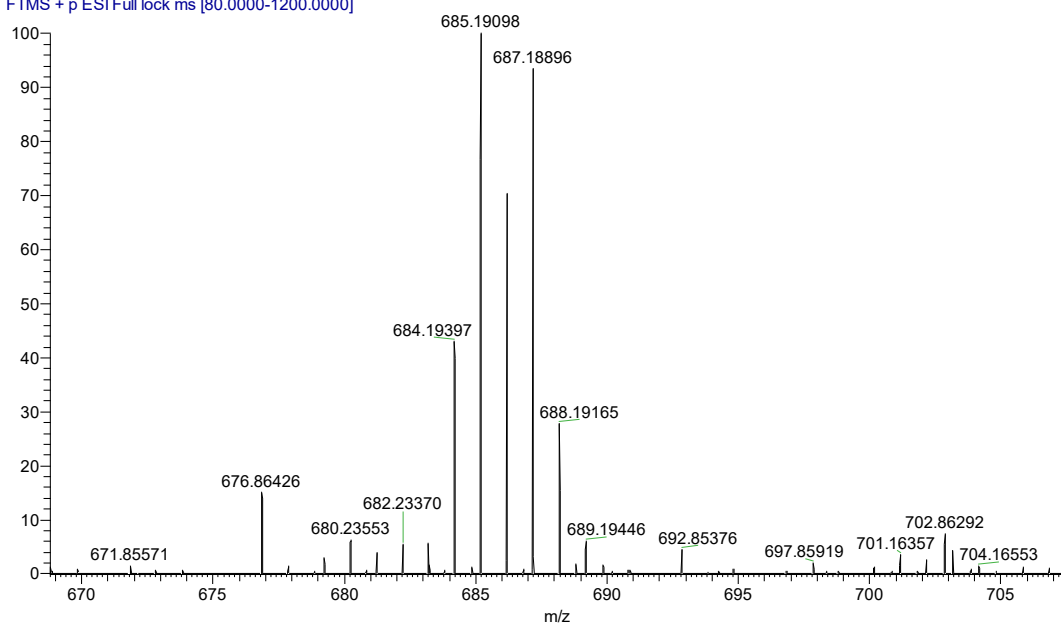


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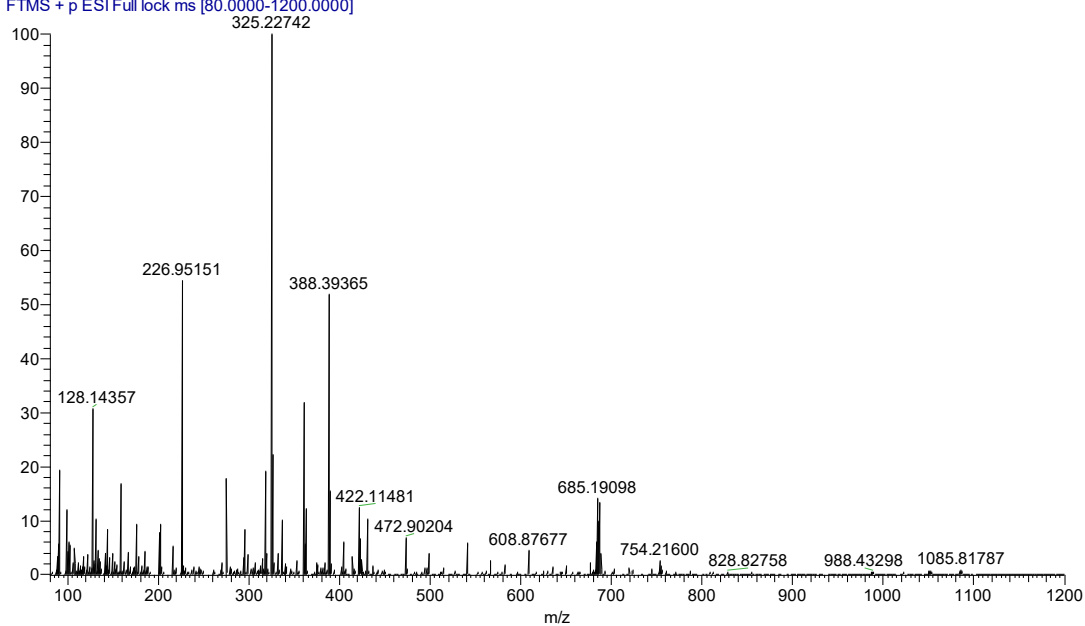


HRMS spectra of 1d

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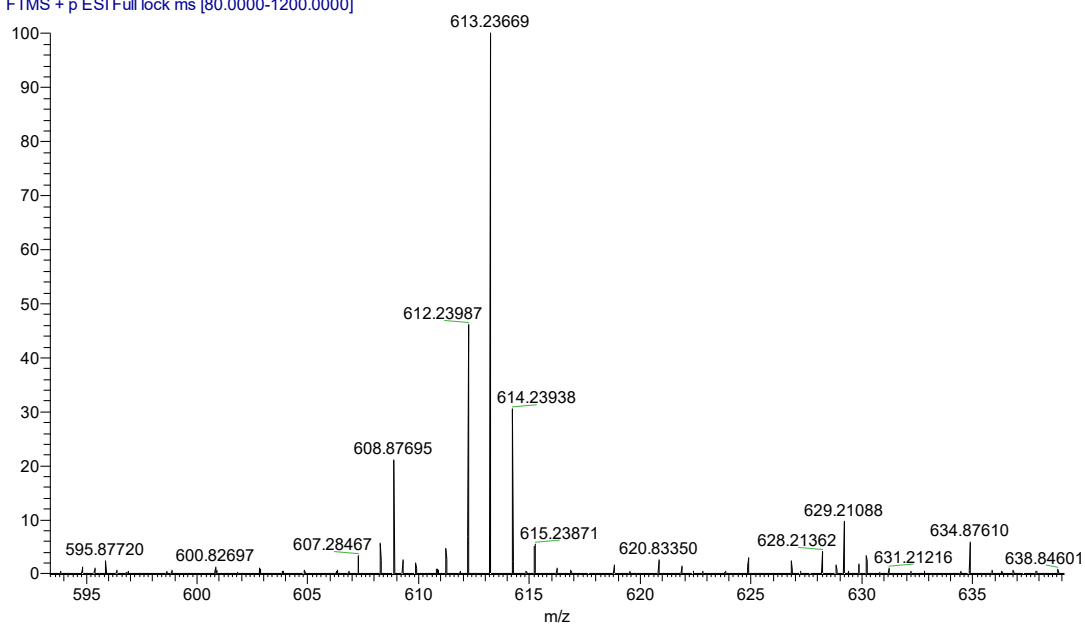


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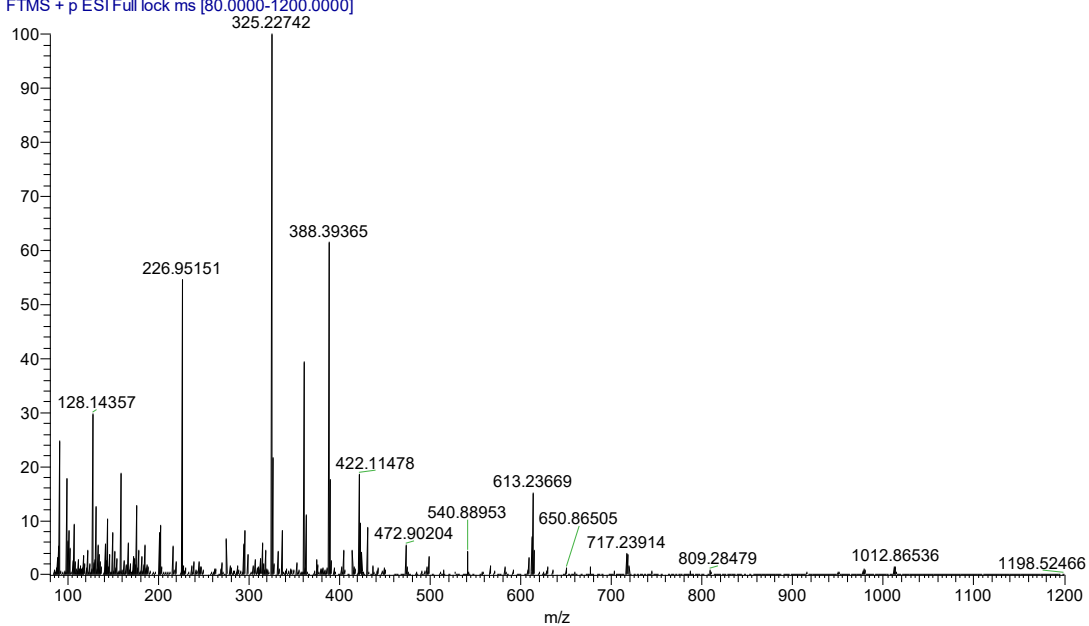


HRMS spectra of 1e

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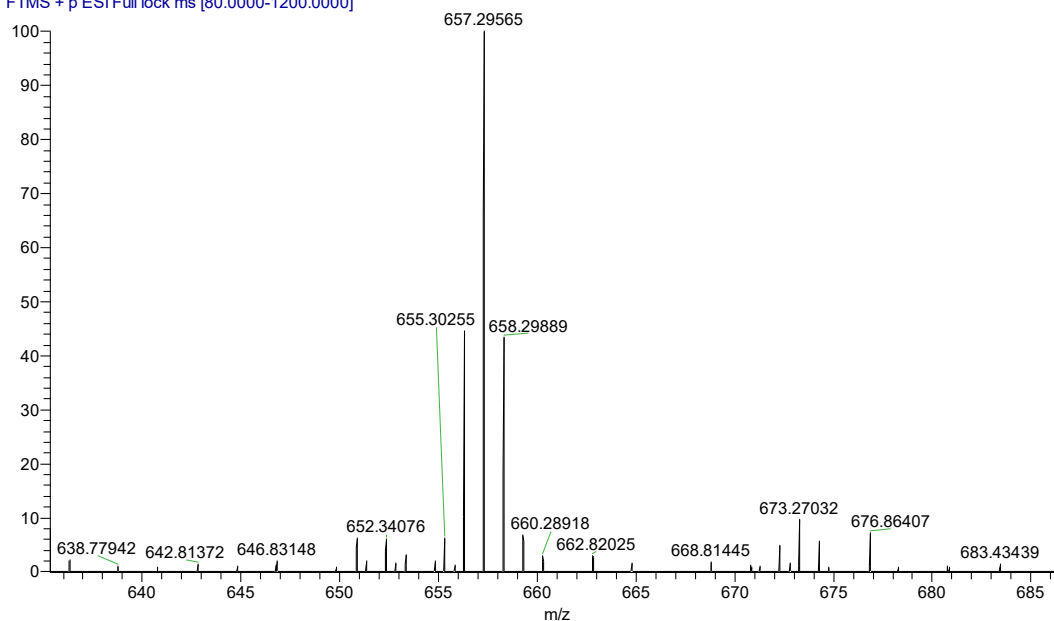


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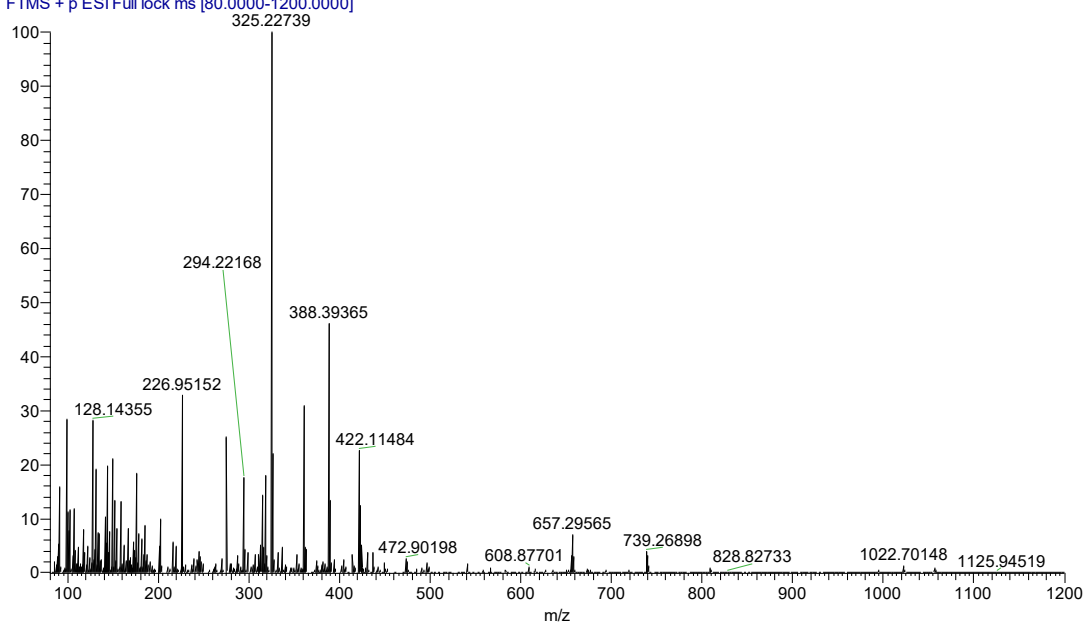


HRMS spectra of 1f

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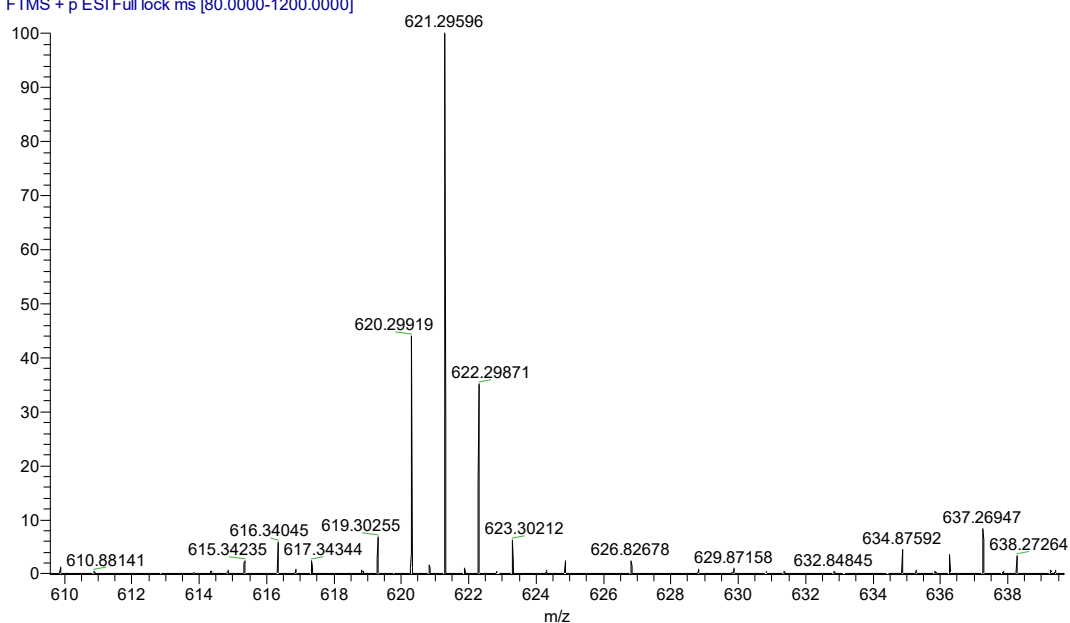


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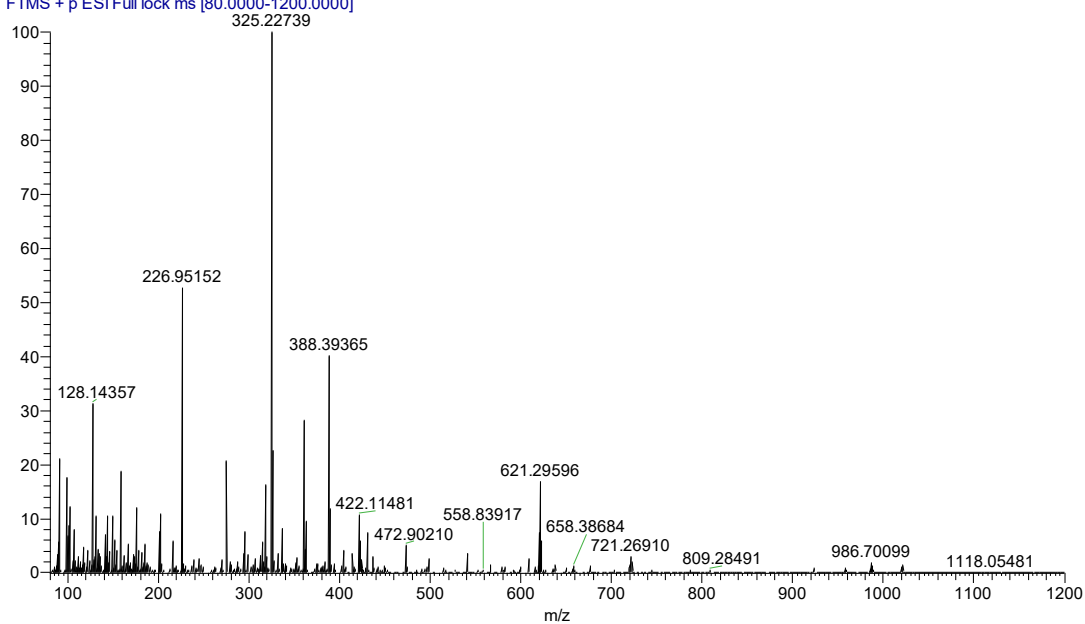


HRMS spectra of 1g

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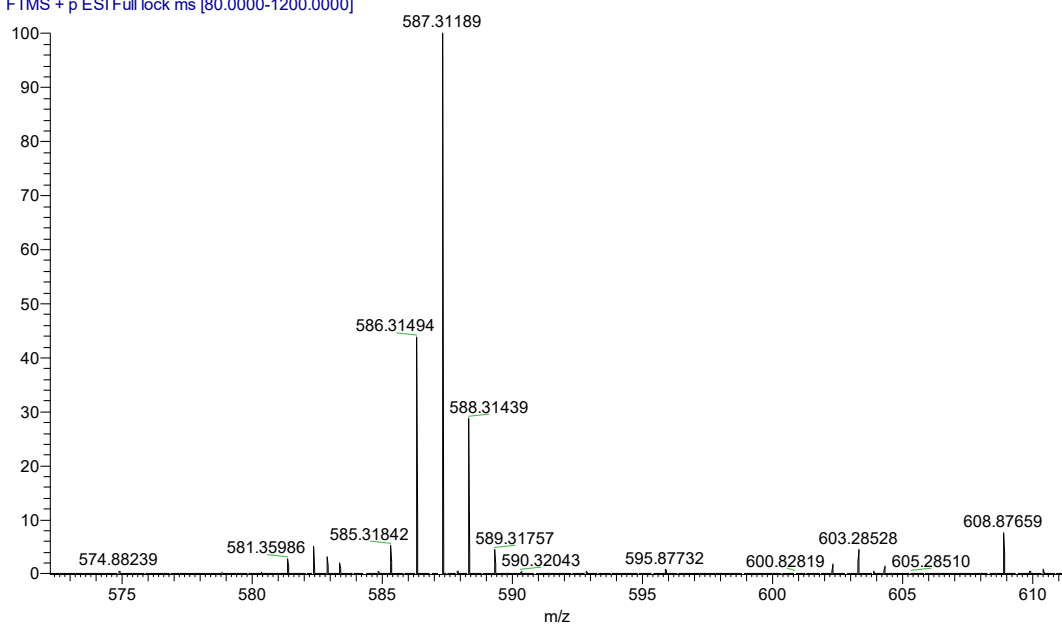


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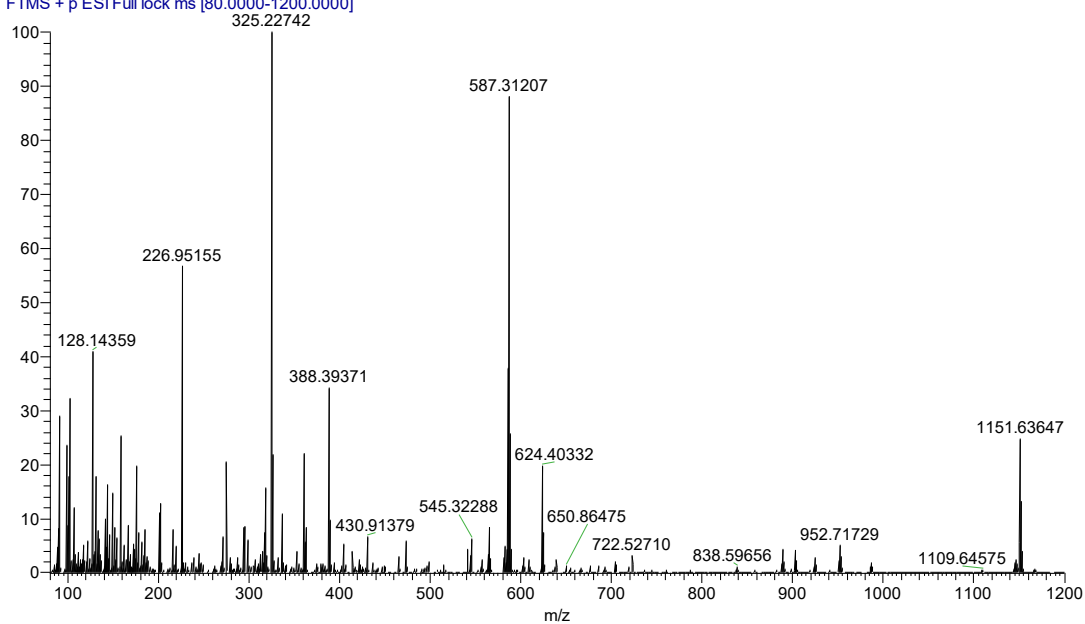


HRMS spectra of 1h

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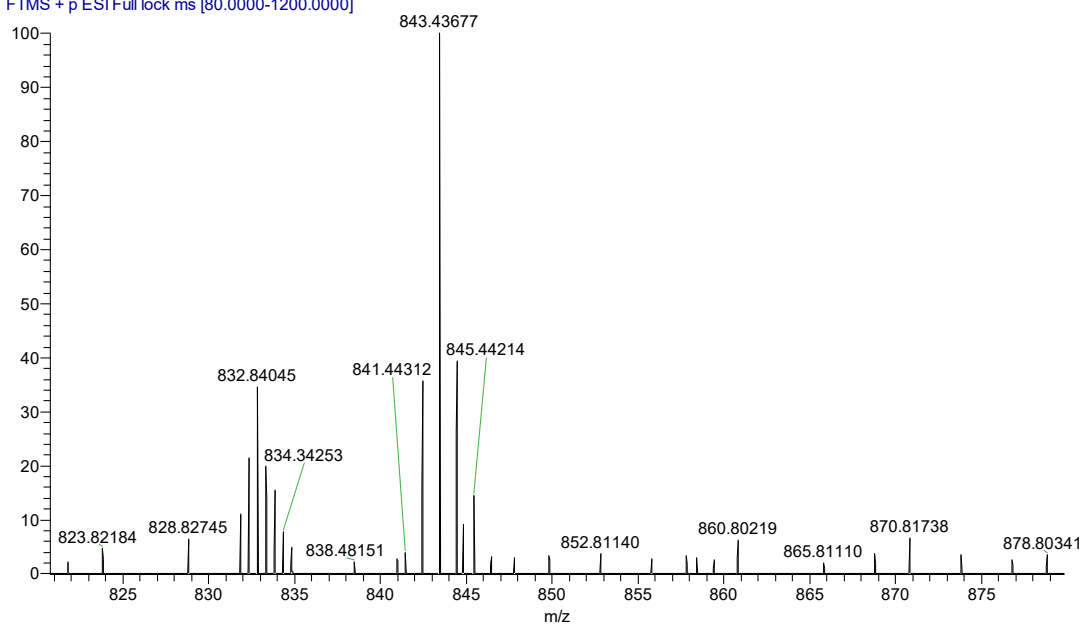


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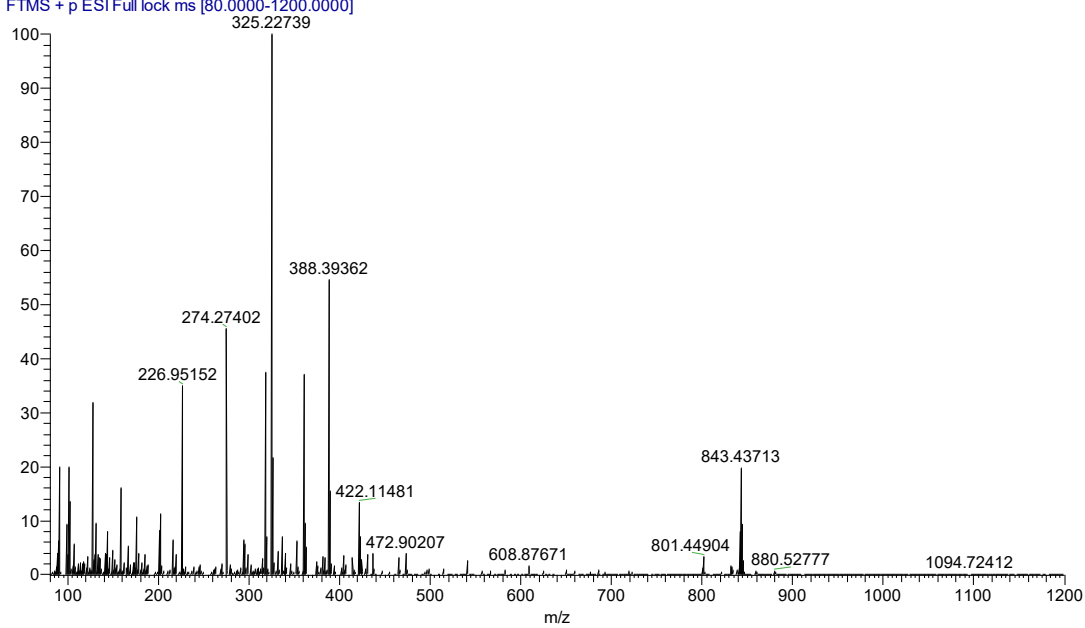


HRMS spectra of **1i**

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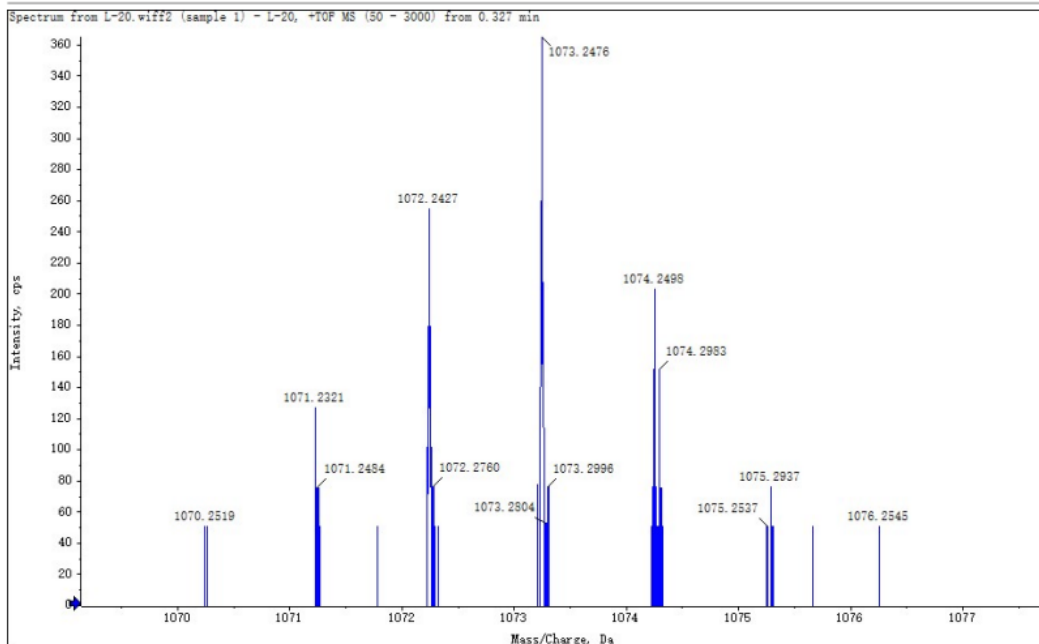
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HRMS spectra of 1j

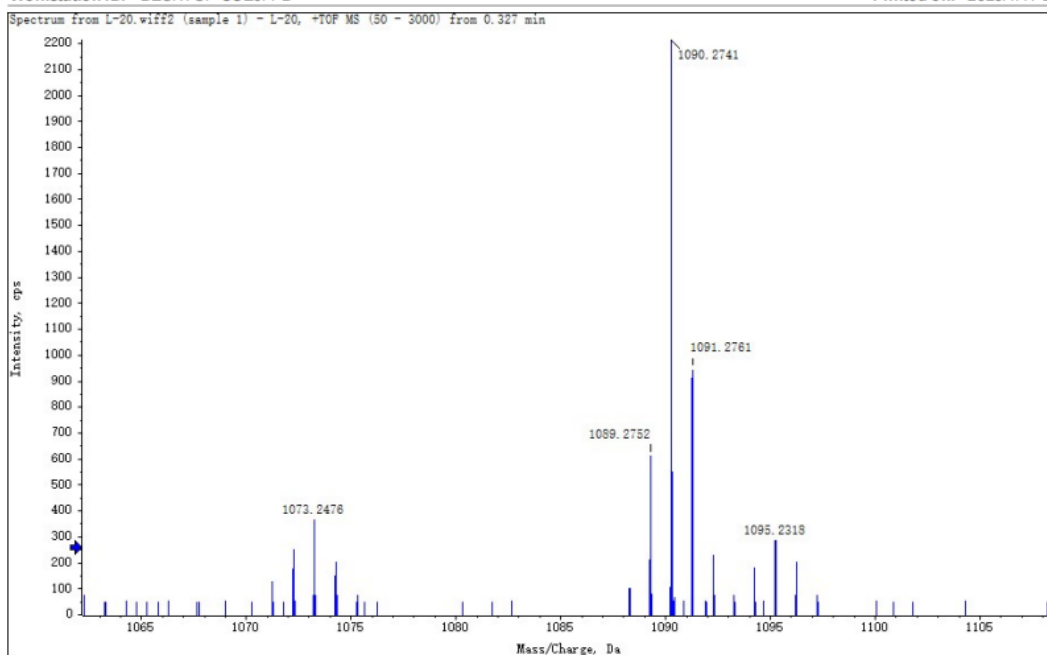
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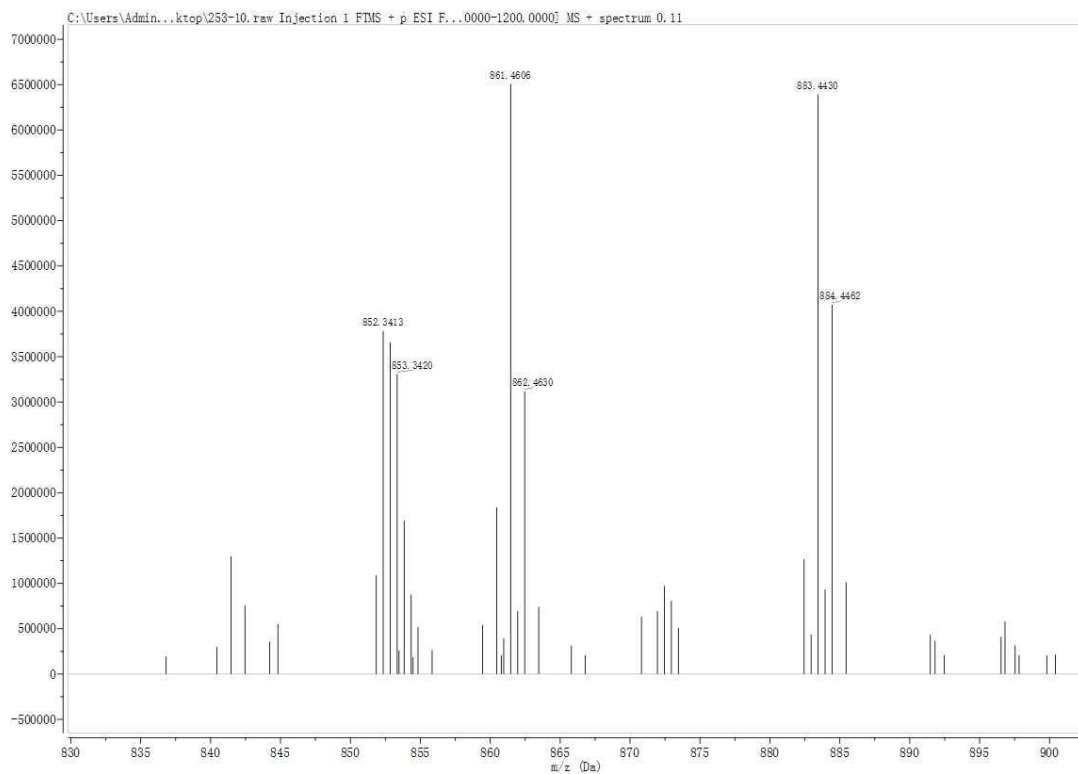


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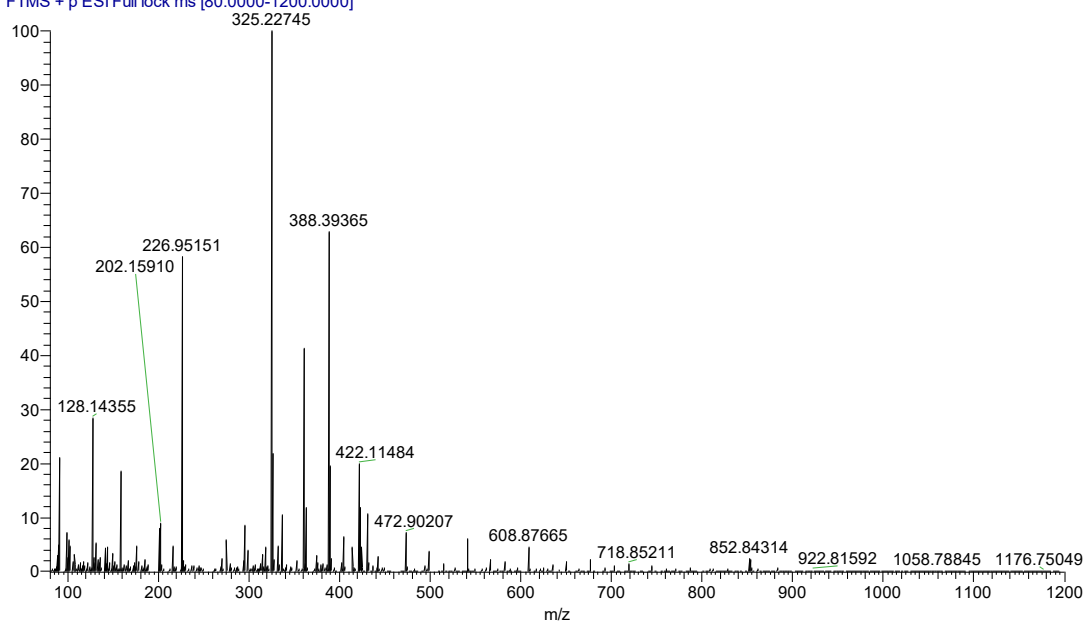
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HRMS spectra of 4



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5. References

1. Vos de Wale, E.; Pardoën, J. A.; van Koeveeringe, J. A.; Lugtenburg, J. *Recl. Trav. Chim. Pays-Bas.*, 1977, **96**, 306.
2. T. Li, W. Gu, C. Yu, X. Lv, H. Wang, E. Hao and L. Jiao, *Chin. J. Chem.*, 2016, **34**, 989.
3. L. Gai, J. Mack, H. Lu, H. Yamada, D. Kuzuhara, G. Lai, Z. Li and Z. Shen, *Chem. Eur. J.*, 2014, **20**, 1091.