

Supporting Information

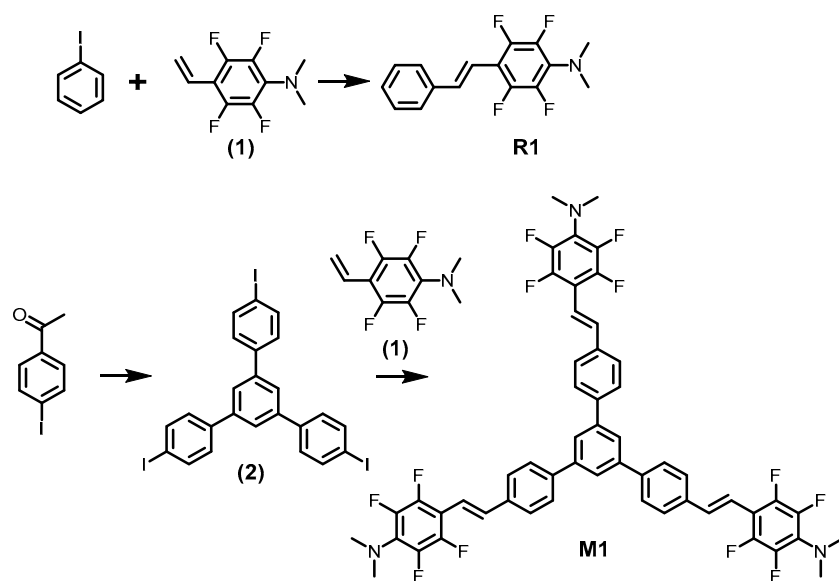
Interfacial Synthesis of an Ultrathin Two-Dimensional Polymer Film via [2 + 2] Photocycloaddition

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Scheme S1. Synthesis of molecules **R1** and **M1**.

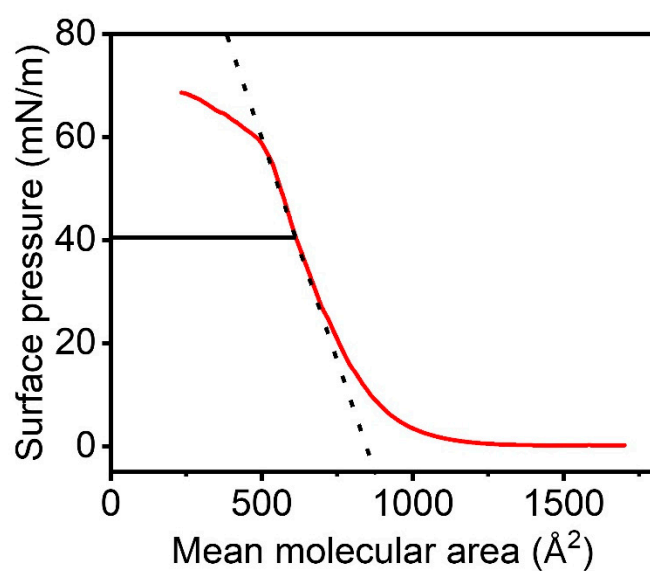


Figure S1. Surface pressure-mean molecular area (SP-MMA) isotherm of compressing **M1** with a compression rate of 3 mm/min.

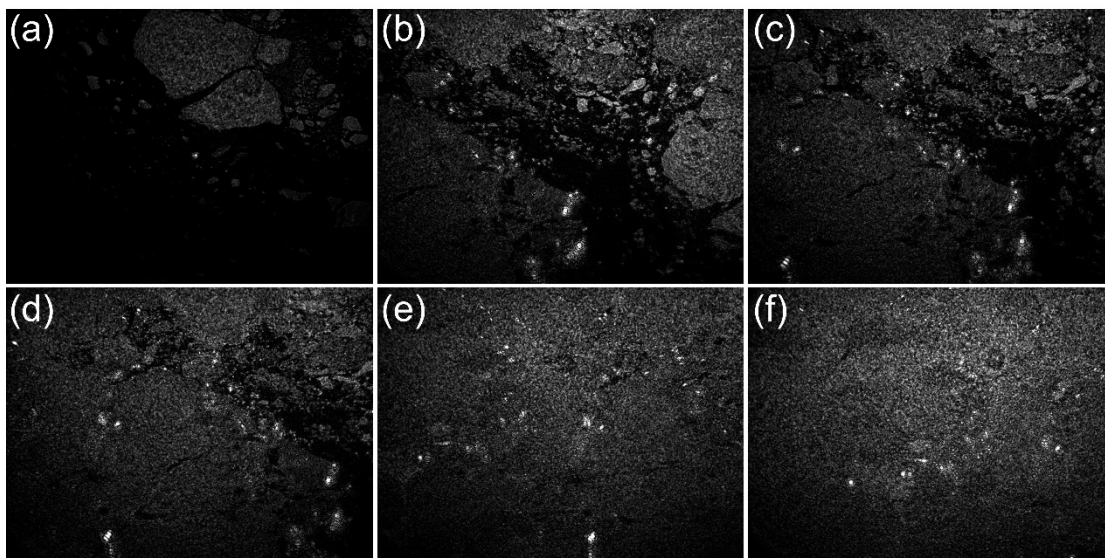


Figure S2. BAM images of the process of compressing **M1** in the SP-MMA isotherm study.

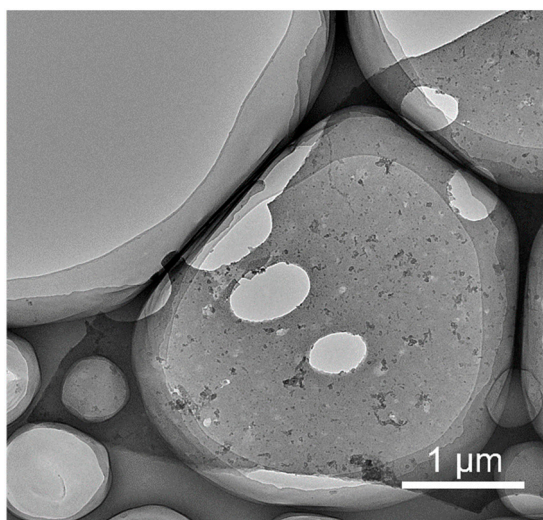


Figure S3. Free-standing **P1** film on a copper TEM grid.

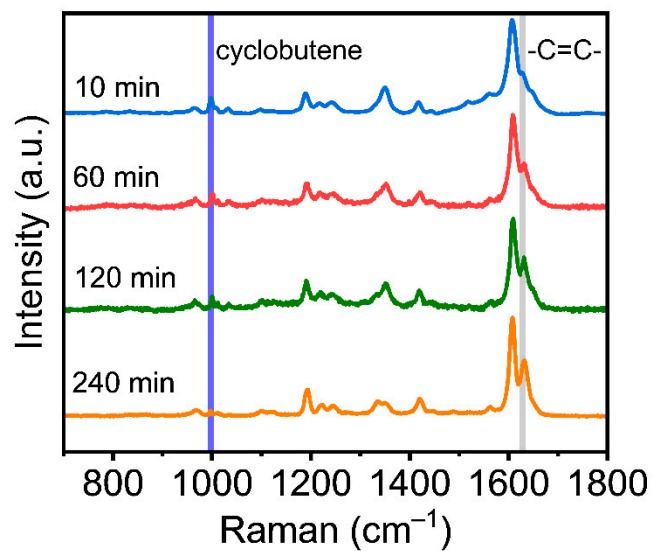


Figure S4. Raman spectrum of the depolymerization process of **P1**.

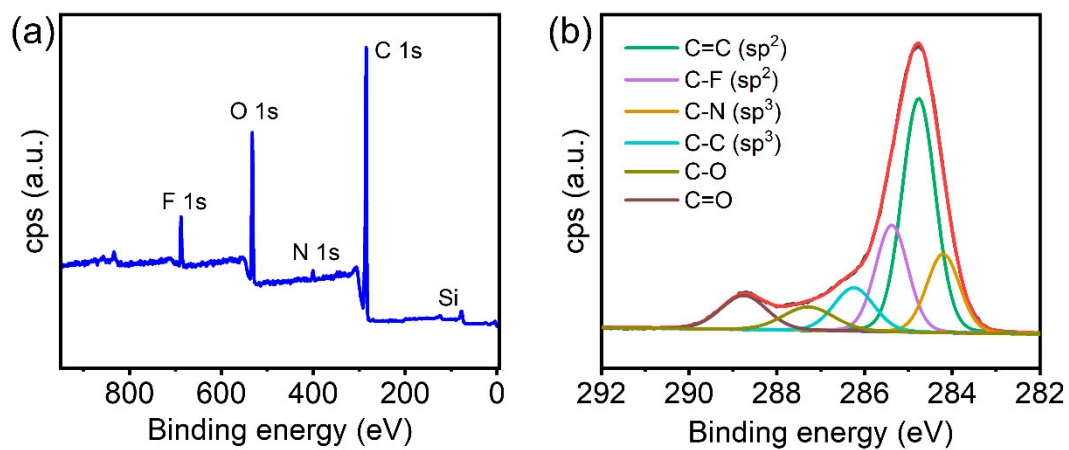


Figure S5. (a) XPS survey spectra of **P1** and (b) XPS C 1s spectra of **P1** on SiO₂/Si wafer.

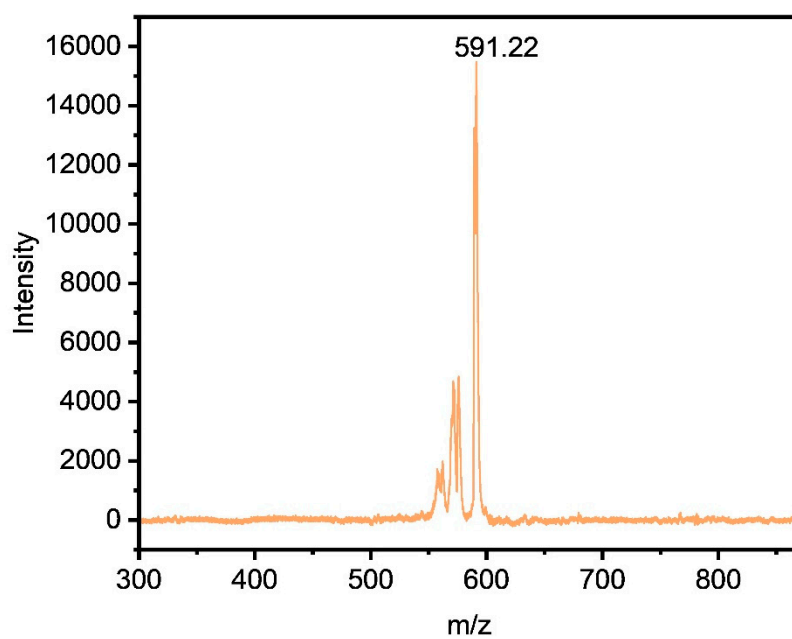


Figure S6. Mass spectrum of the compound **D1**.

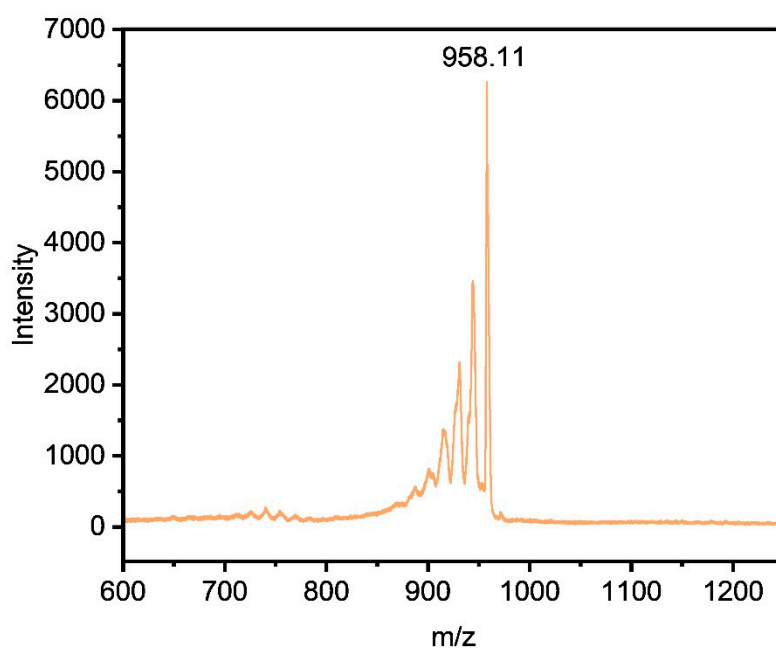


Figure S7. Mass spectrum of the compound **M1**.

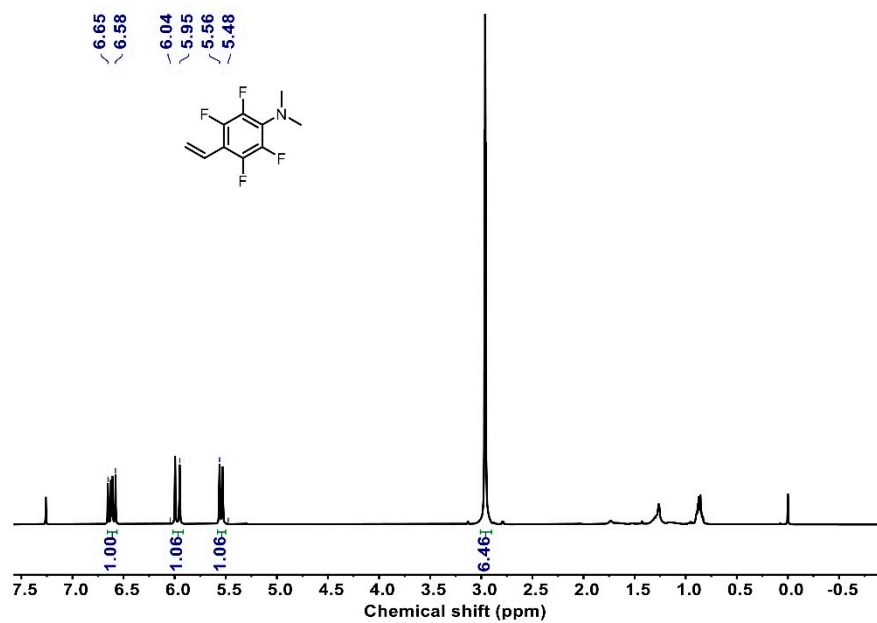


Figure S8. ¹H NMR spectrum of compound (1).

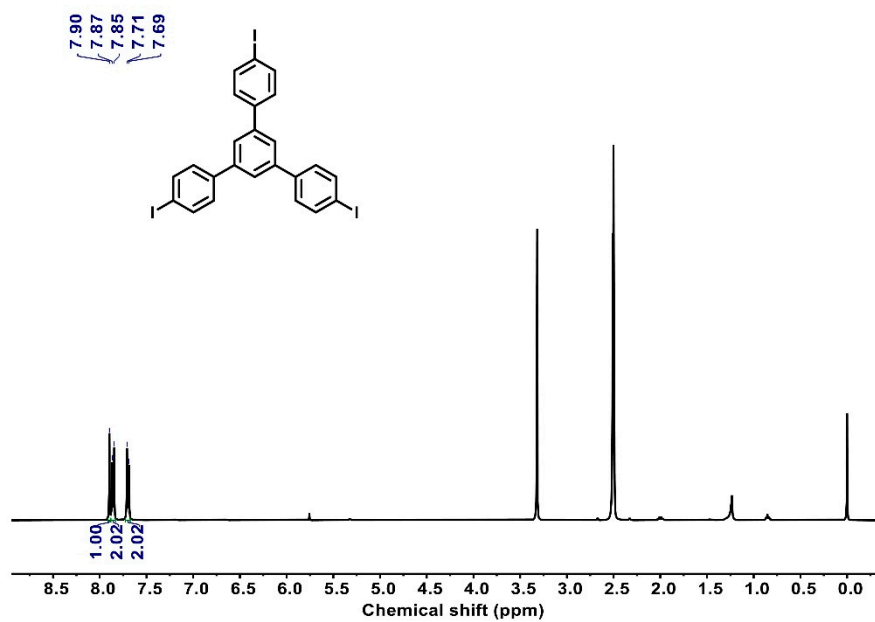


Figure S9. ¹H NMR spectrum of compound (2).

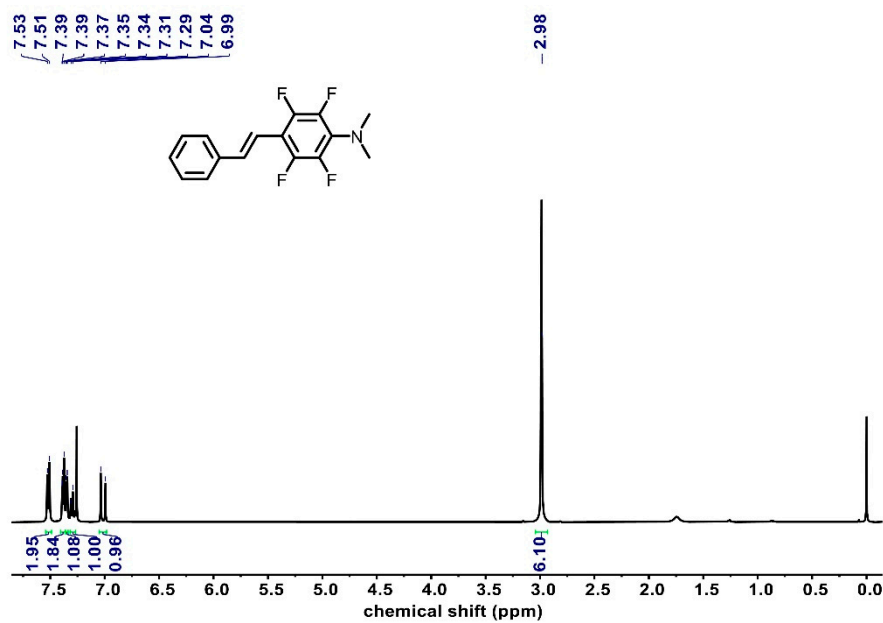


Figure S10. ¹H NMR spectrum of the compound **R1**.

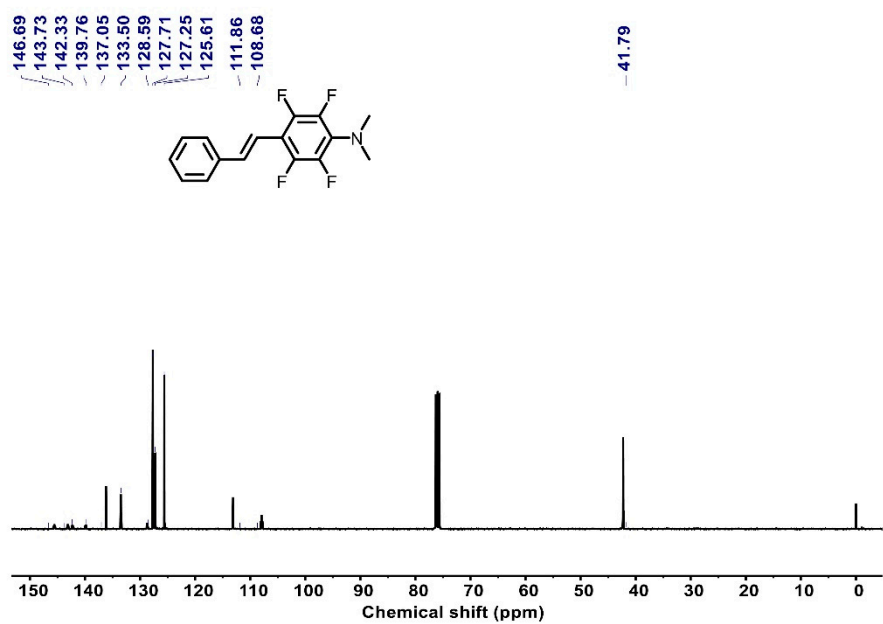


Figure. S11. ¹³C NMR spectrum of the compound **R1**.

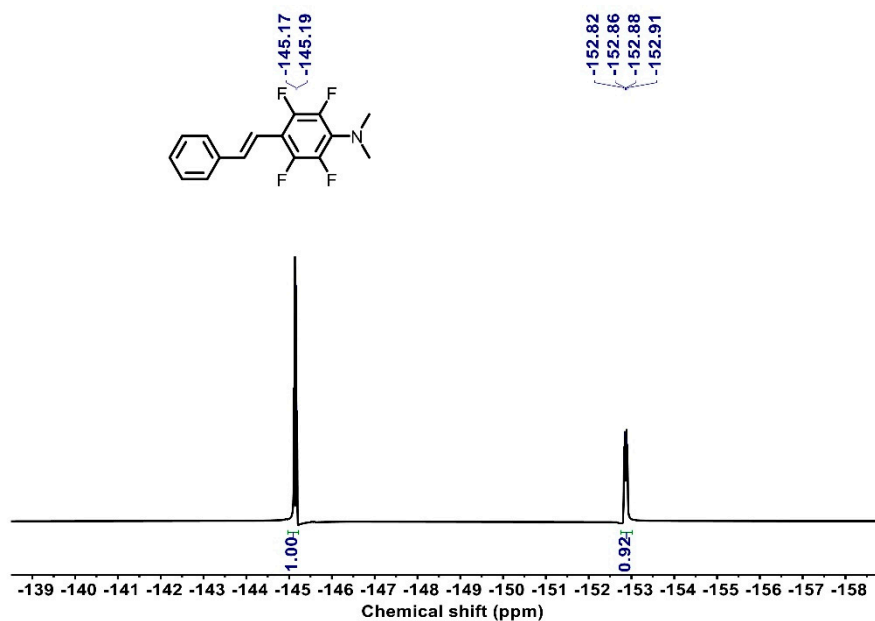


Figure S12. ^{19}F NMR spectrum of the compound **R1**.

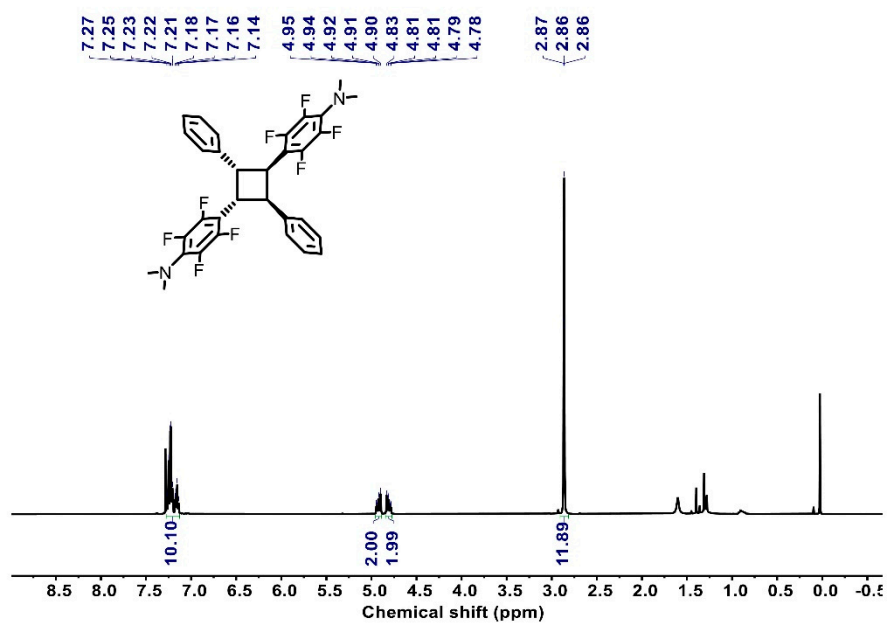


Figure S13. ^1H NMR spectrum of the compound **D1**.

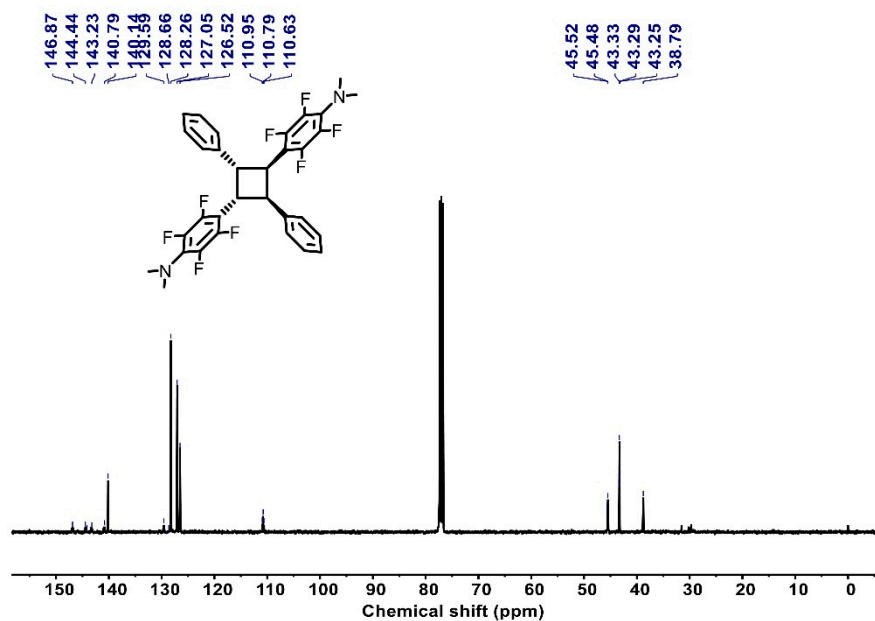


Figure S14. ¹³C NMR spectrum of the compound D1.

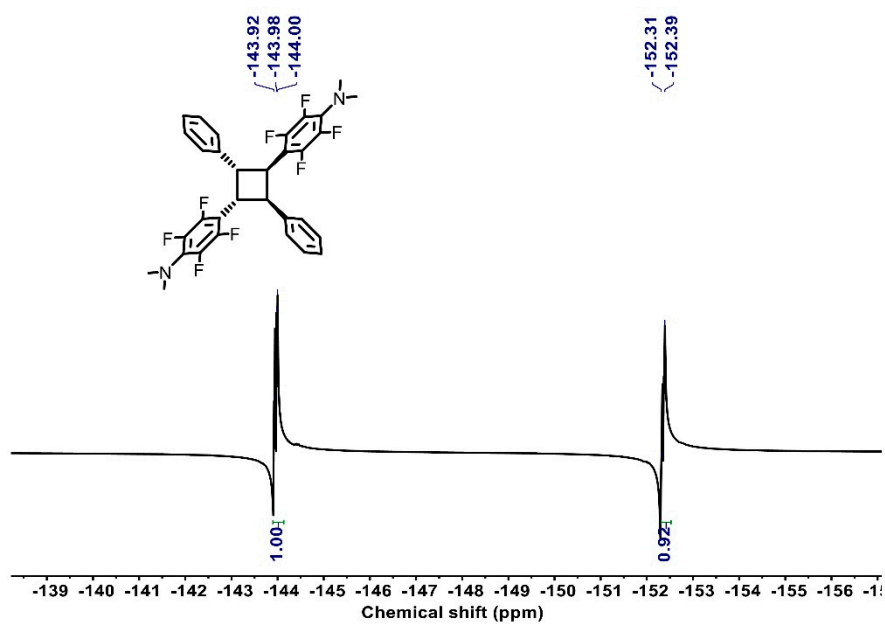


Figure S15. ¹⁹F NMR spectrum of the compound R1.

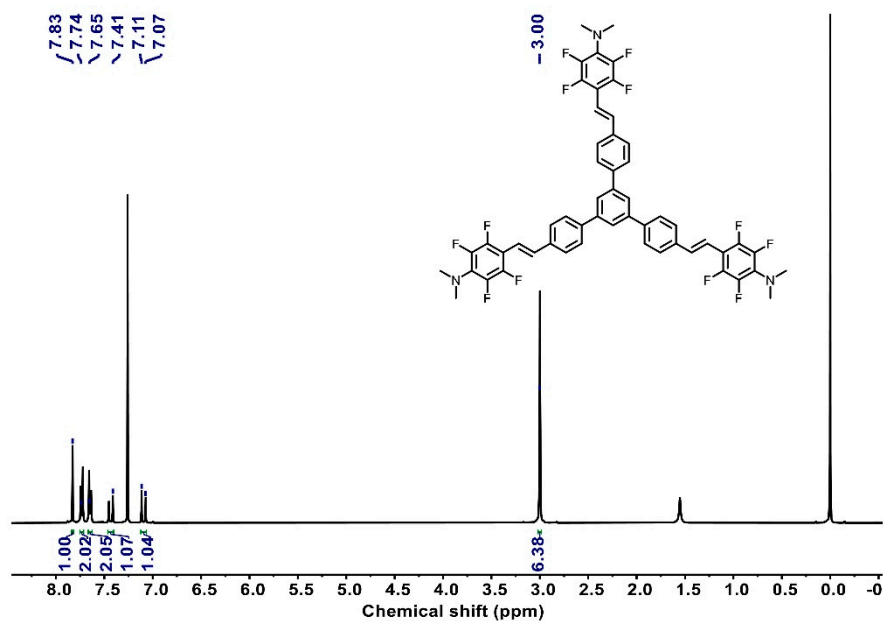


Figure S16. ¹H NMR spectrum of the compound **M1**.

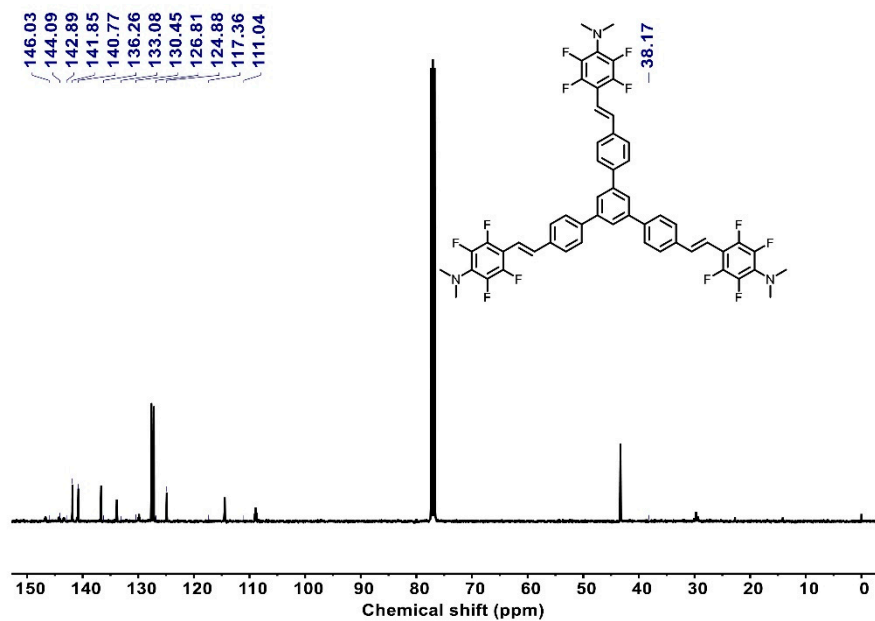


Figure S17. ¹³C NMR spectrum of the compound **M1**.

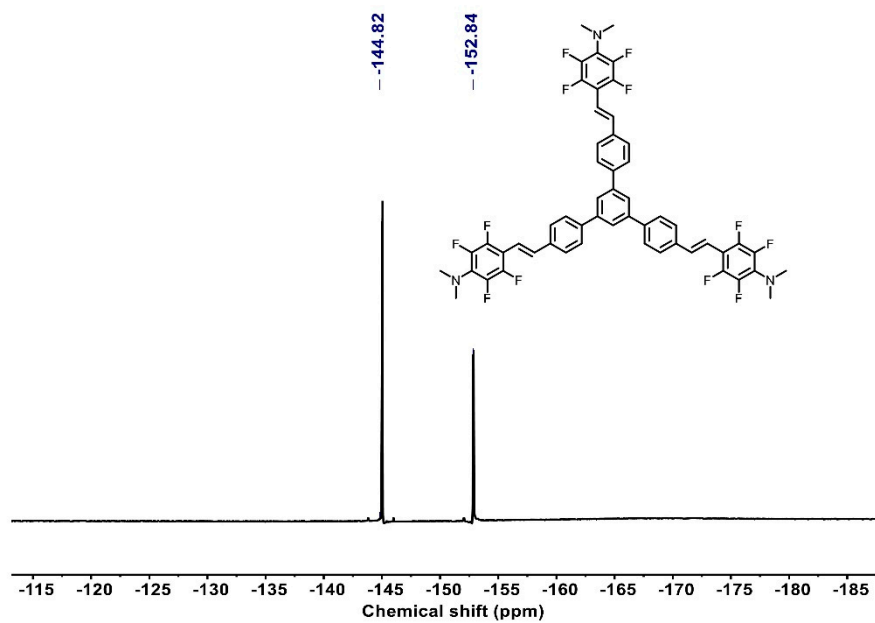


Figure S18. ^{19}F NMR spectrum of the compound **M1**.

Table S1: The single crystal data of **R1**.

Identification code	MX8069
CCDC number	2226830
Empirical formula	C ₁₆ H ₁₃ F ₄ N
Formula weight	295.27
Temperature/K	170.00(10)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	10.4213(4)
b/Å	7.5800(3)
c/Å	17.4234(7)
α /°	90
β /°	101.852(4)
γ /°	90
Volume/Å ³	1346.99(9)
Z	4
ρ _{calc} /cm ³	1.456
μ /mm ⁻¹	0.124
F(000)	608.0
Crystal size/mm ³	0.58 × 0.43 × 0.35
Radiation	Mo K α (λ = 0.71073)
2 Θ range for data collection/	5.562 to 60.534
Index ranges	-14 ≤ h ≤ 9, -7 ≤ k ≤ 10, -23 ≤ l ≤ 22
Reflections collected	10583
Independent reflections	3592 [R _{int} = 0.0141, R _{sigma} = 0.0154]
Data/restraints/parameters	3592/0/211
Goodness-of-fit on F ²	1.079
Final R indexes [I ≥ 2 σ (I)]	R ₁ = 0.0682, wR ₂ = 0.1801
Final R indexes [all data]	R ₁ = 0.0814, wR ₂ = 0.1895
Largest diff. peak/hole / e Å ⁻³	0.52/-0.82