Synthesis

Reviews and Full Papers in Chemical Synthesis

Supporting Information
for DOI: 10.1055/a-2097-0092

Georg Thieme Verlag KG, Rüdigerstraße 14, 70469 Stuttgart, Germany

Thieme

Supporting Information

for manuscript entitled

Kinetic or Thermodynamic Product? Case Studies on the Formation of Regioisomers of Tetraphenyladamantanes

by Tim Berking, Wolfgang Frey and Clemens Richert

1. NMR Spectra

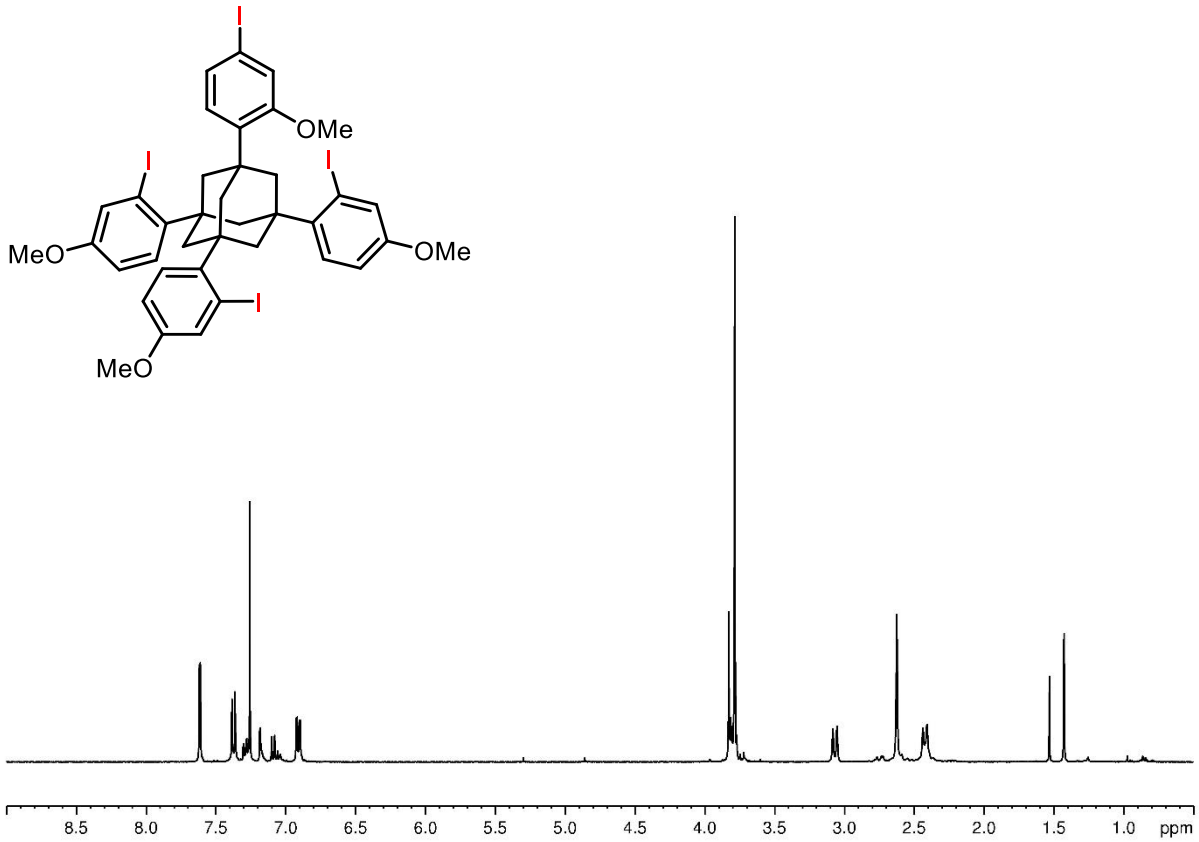

Figure S1. ^{1}H-NMR spectrum of compound **11** ($CDCl_3$, 400 MHz).

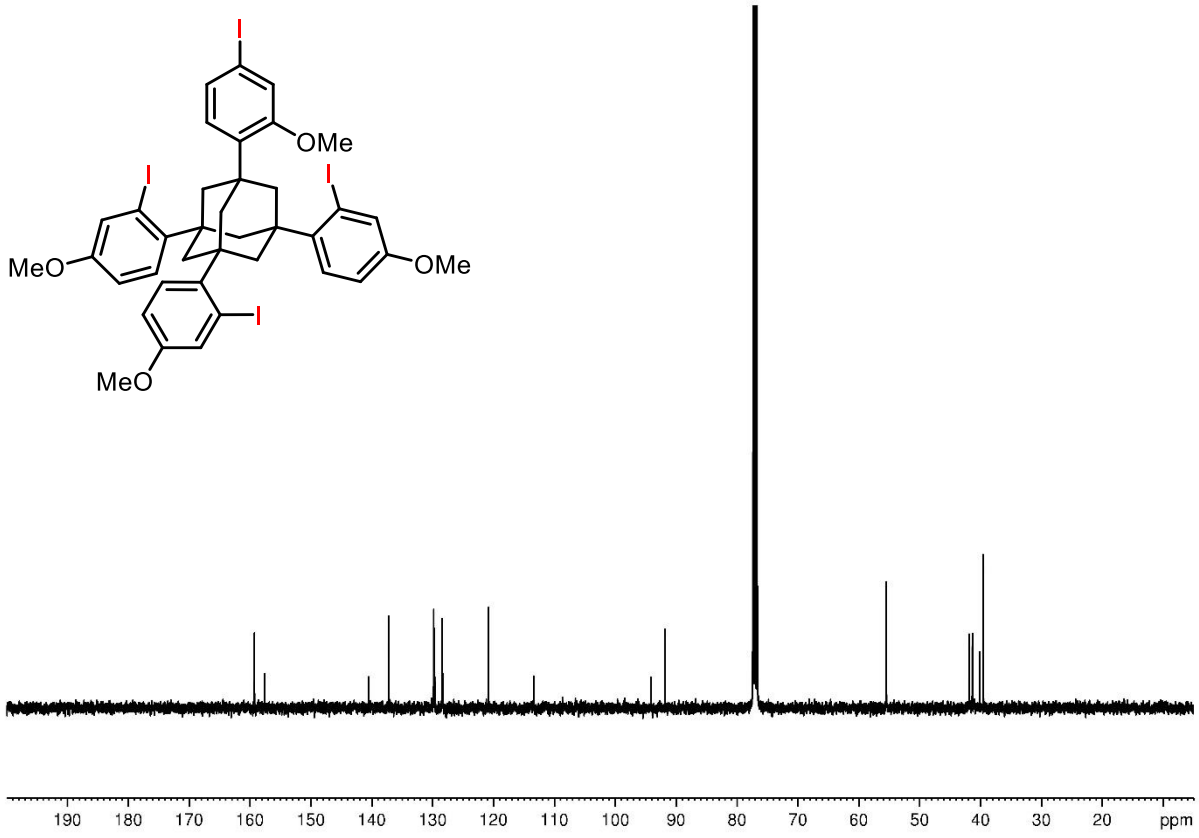

Figure S2. ^{13}C-NMR spectrum of compound **11** ($CDCl_3$, 101 MHz).

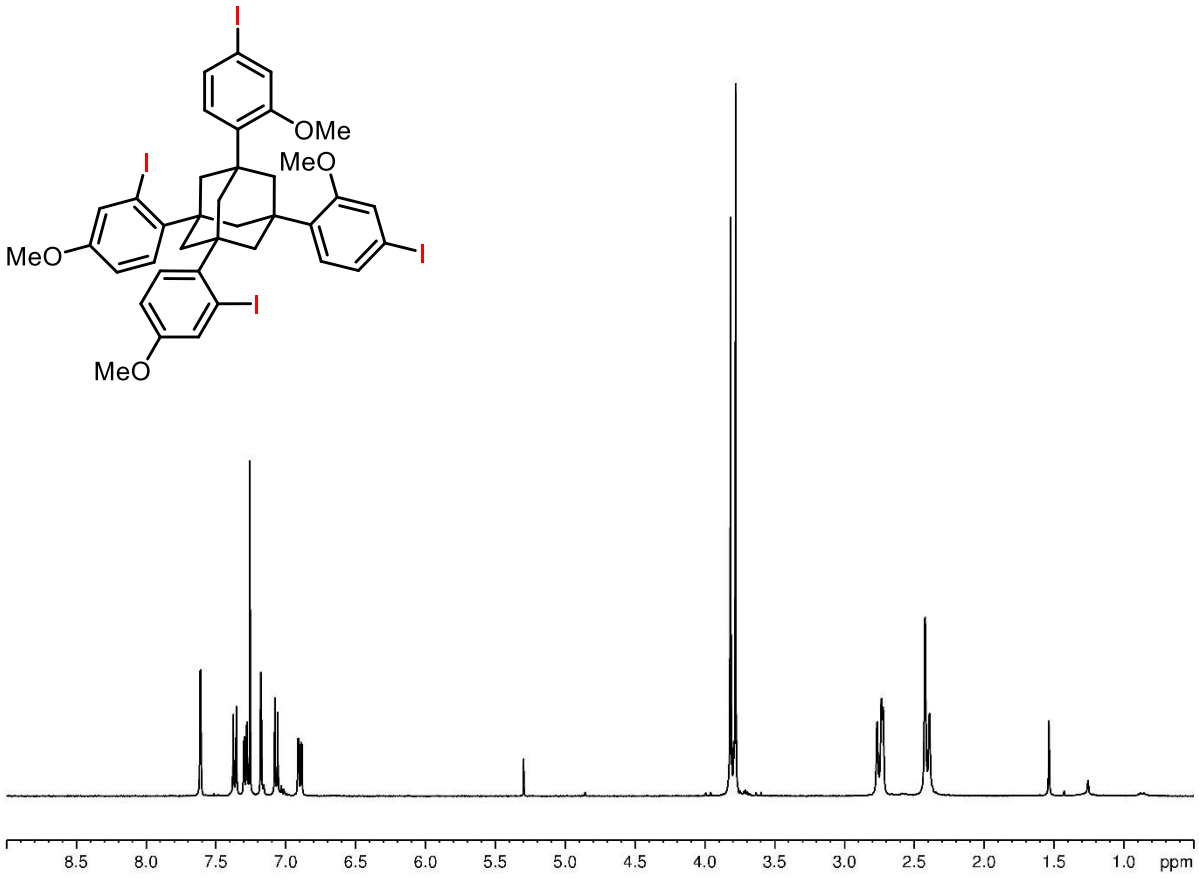

Figure S3. ^{1}H-NMR spectrum of compound **12** ($CDCl_3$, 400 MHz).

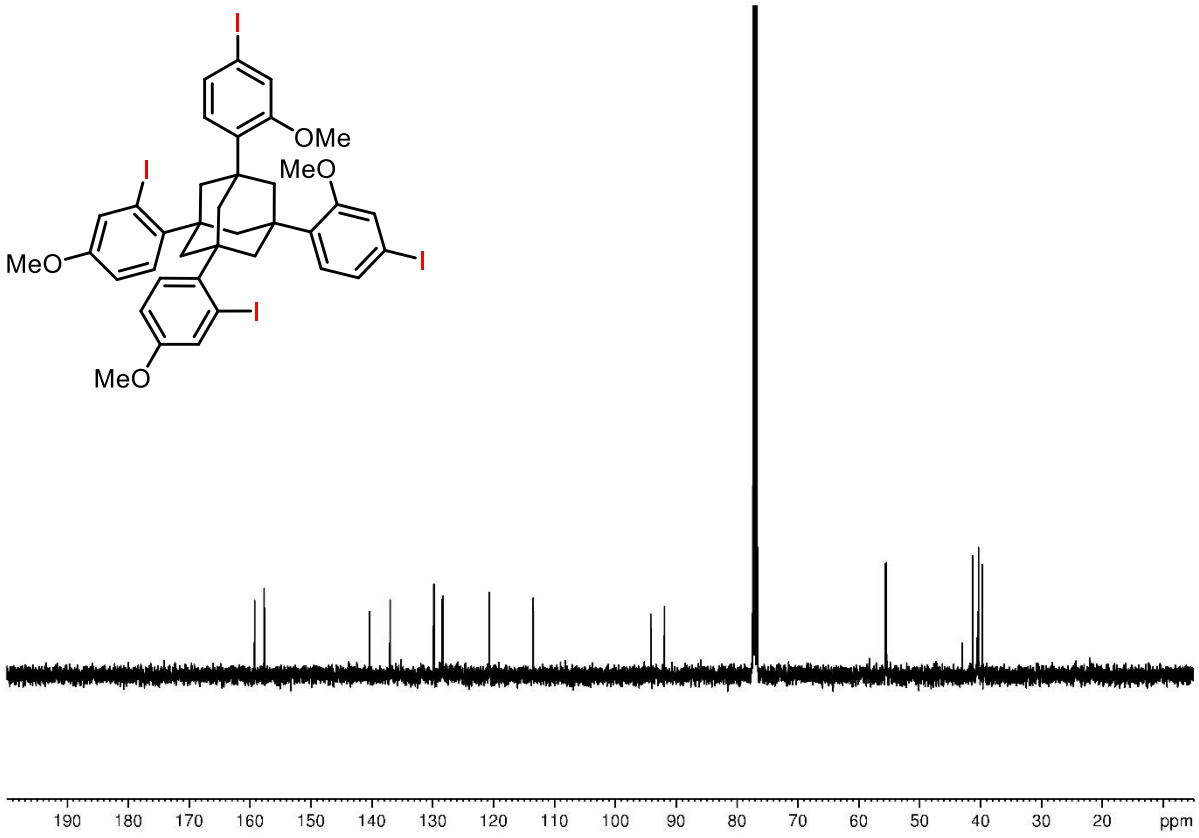

Figure S4. ^{13}C-NMR spectrum of compound **12** ($CDCl_3$, 101 MHz).

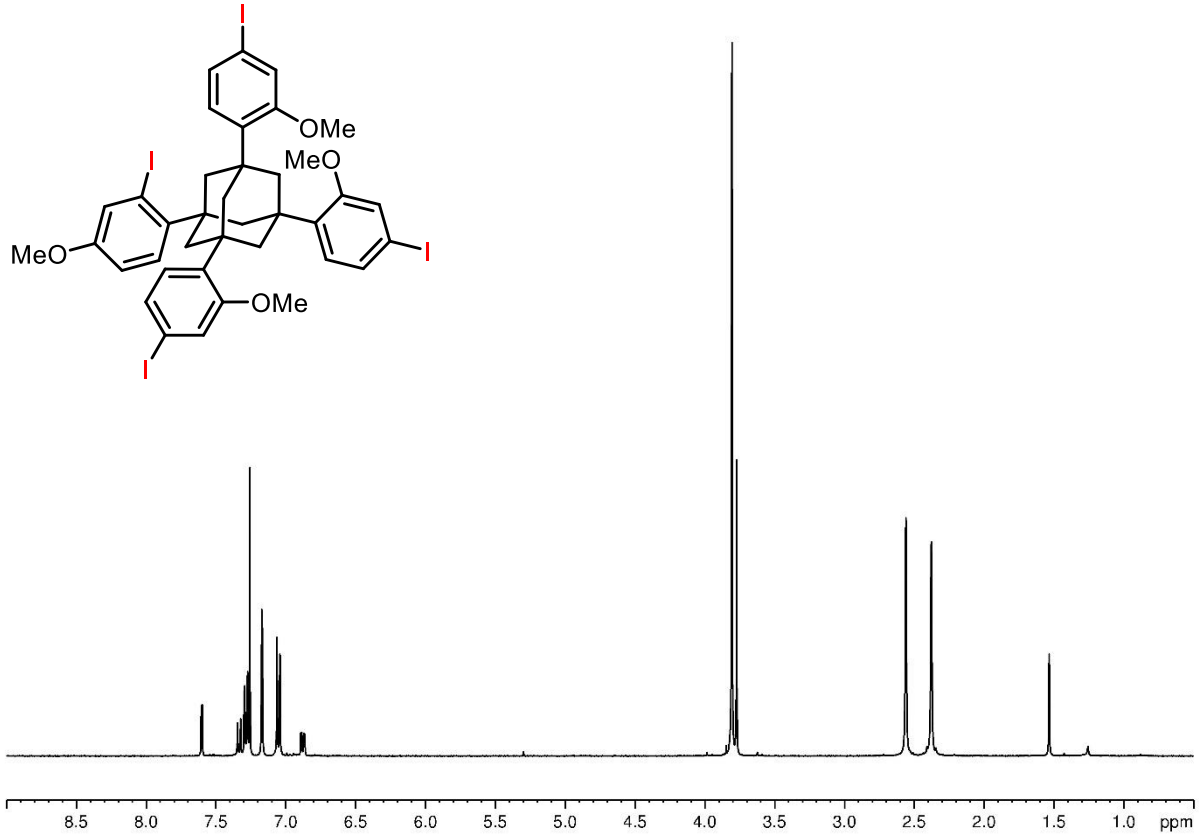

Figure S5. ^{1}H-NMR spectrum of compound **13 (**$CDCl_3$, 400 MHz).

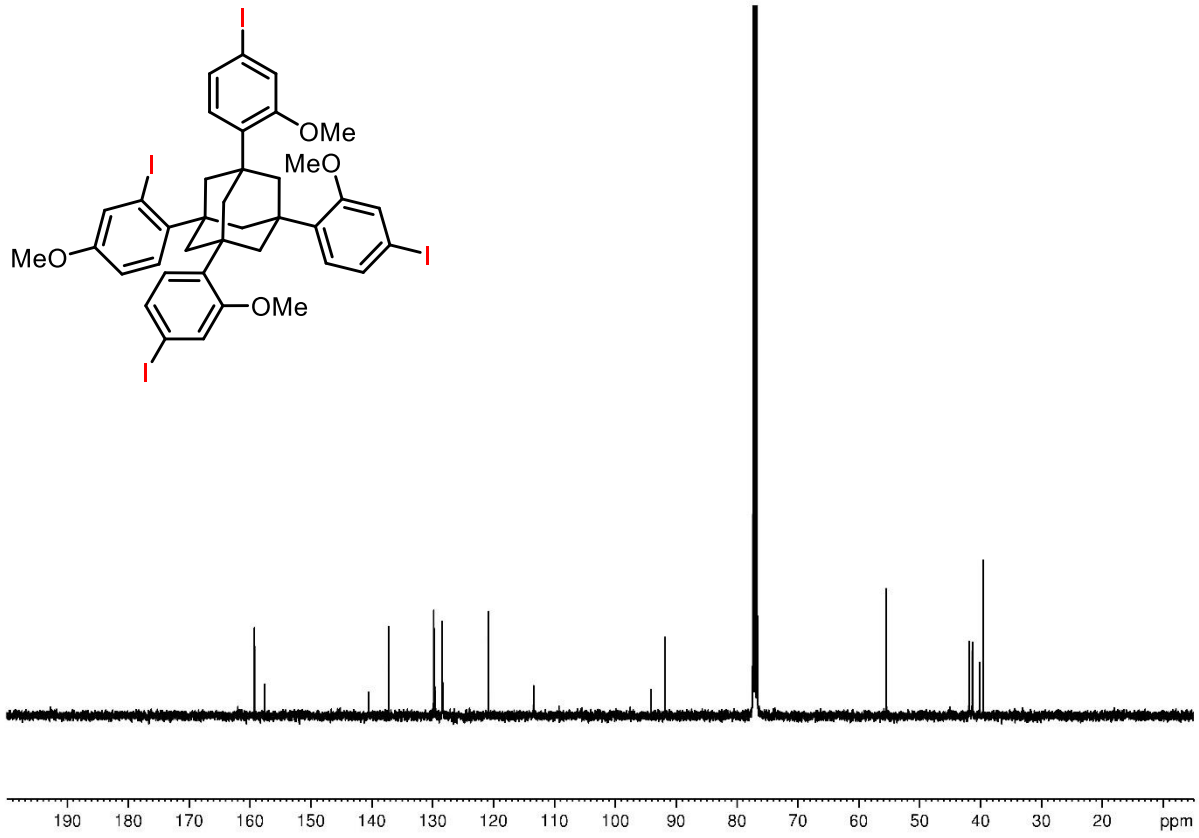

Figure S6. ^{13}C-NMR spectrum of compound **13** ($CDCl_3$, 101 MHz).

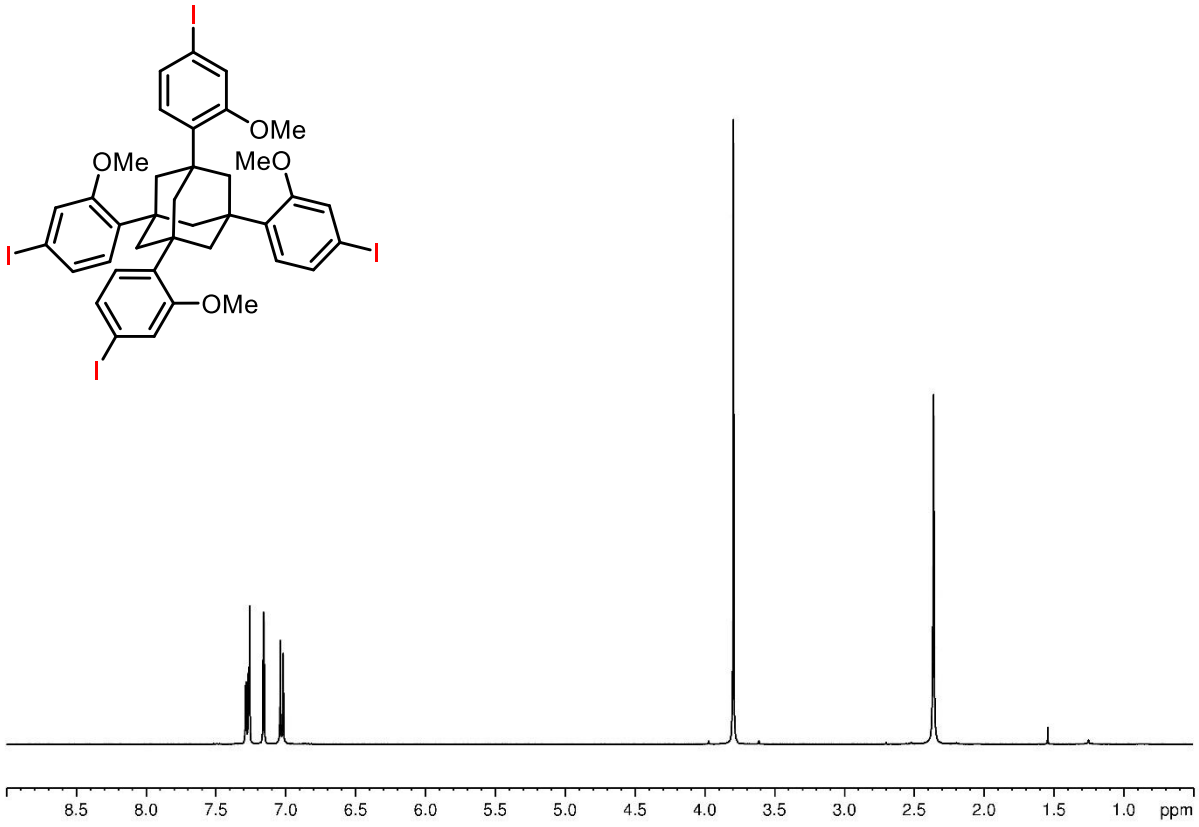

Figure S7. ^{1}H-NMR spectrum of iTIM ($CDCl_3$, 400 MHz).

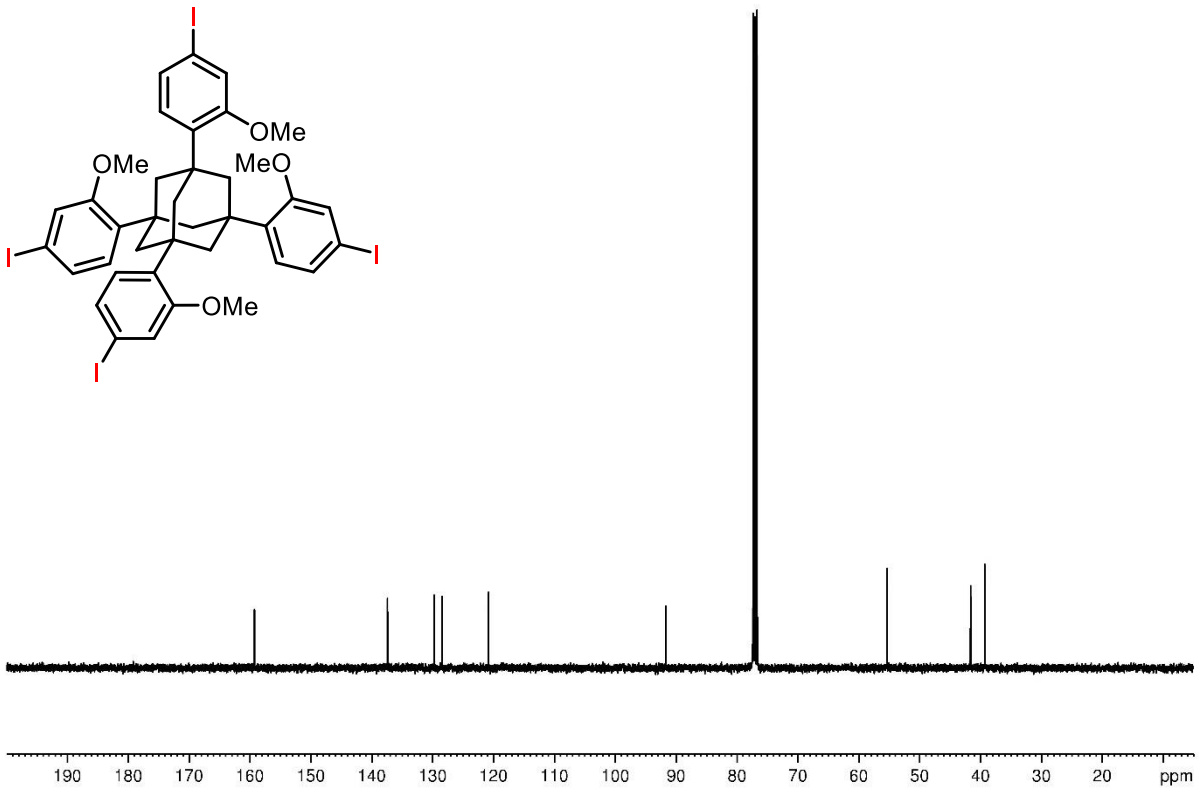

Figure S8. ^{13}C-NMR spectrum of iTIM ($CDCl_3$, 101 MHz).

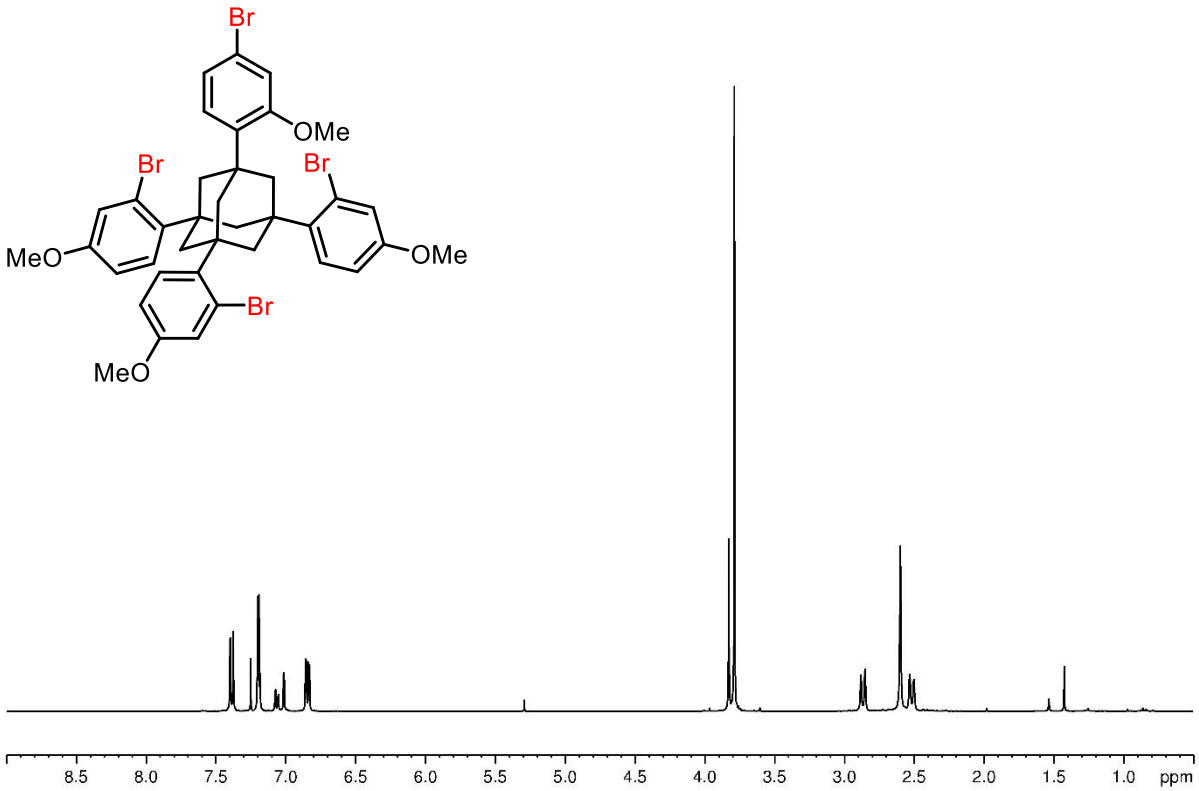

Figure S9. ^{1}H-NMR spectrum of compound **5** ($CDCl_3$, 400 MHz).

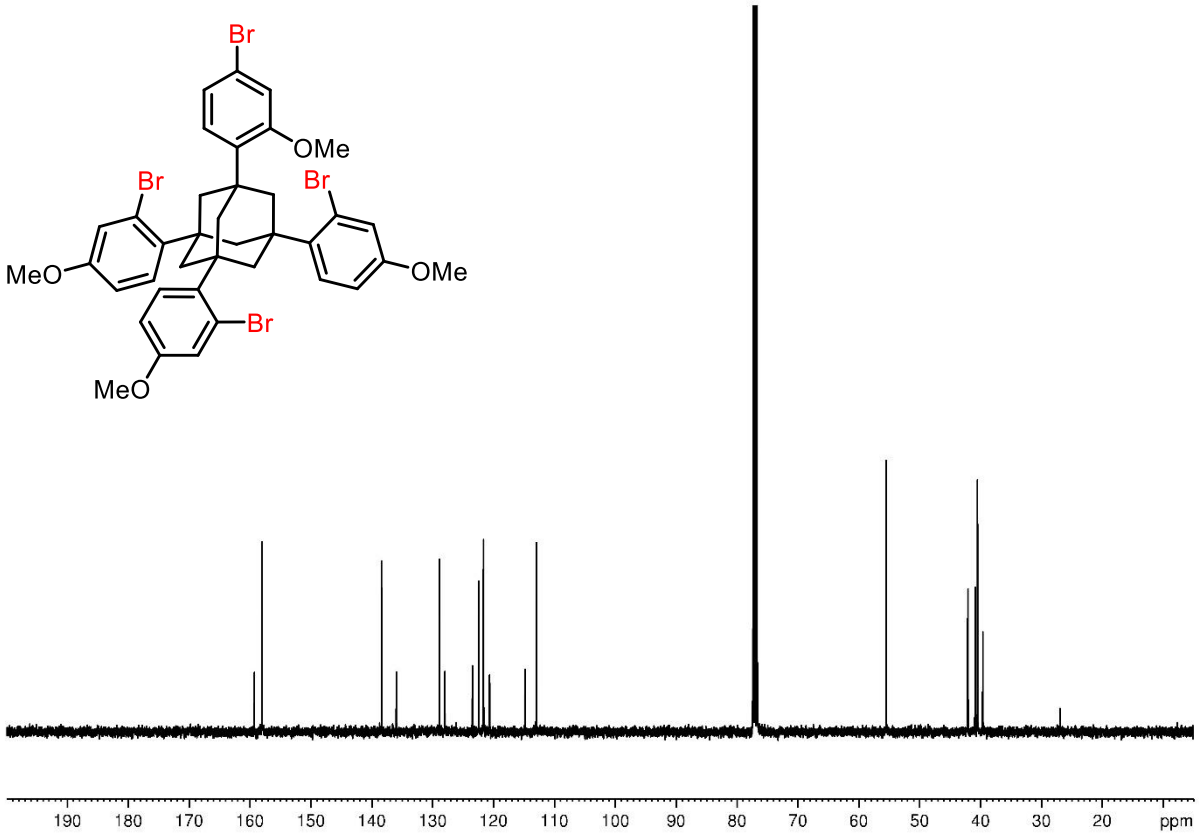

Figure S10. ^{13}C-NMR spectrum of compound **5** ($CDCl_3$, 101 MHz).

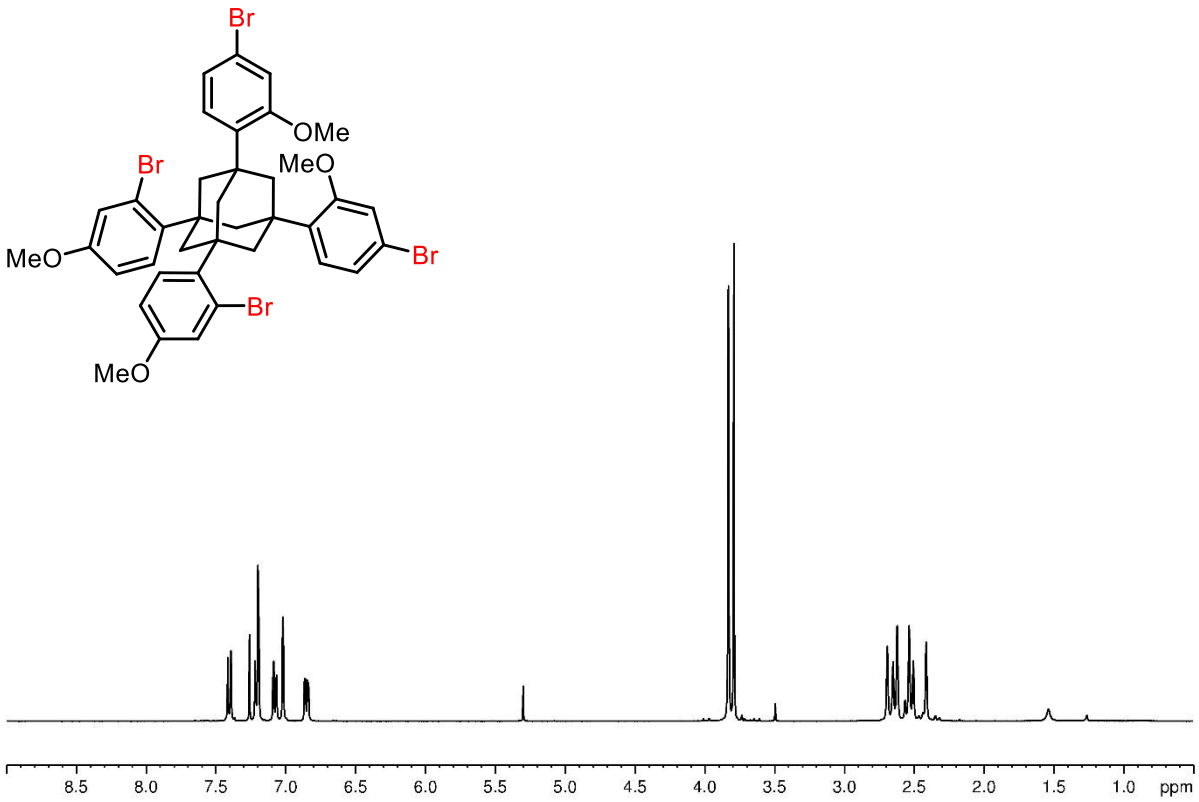

Figure S11. ^{1}H-NMR spectrum of compound **6** ($CDCl_3$, 400 MHz).

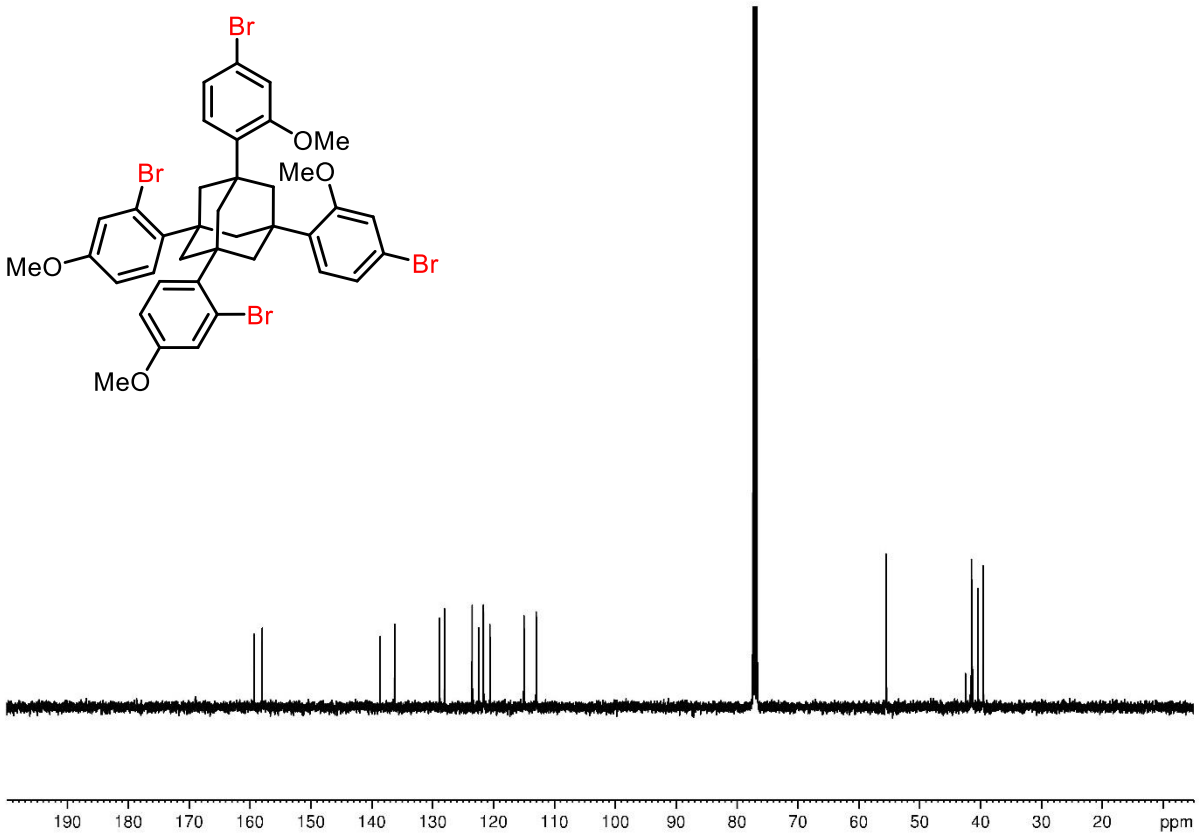

Figure S12. ^{13}C-NMR spectrum of compound **6** ($CDCl_3$, 101 MHz).

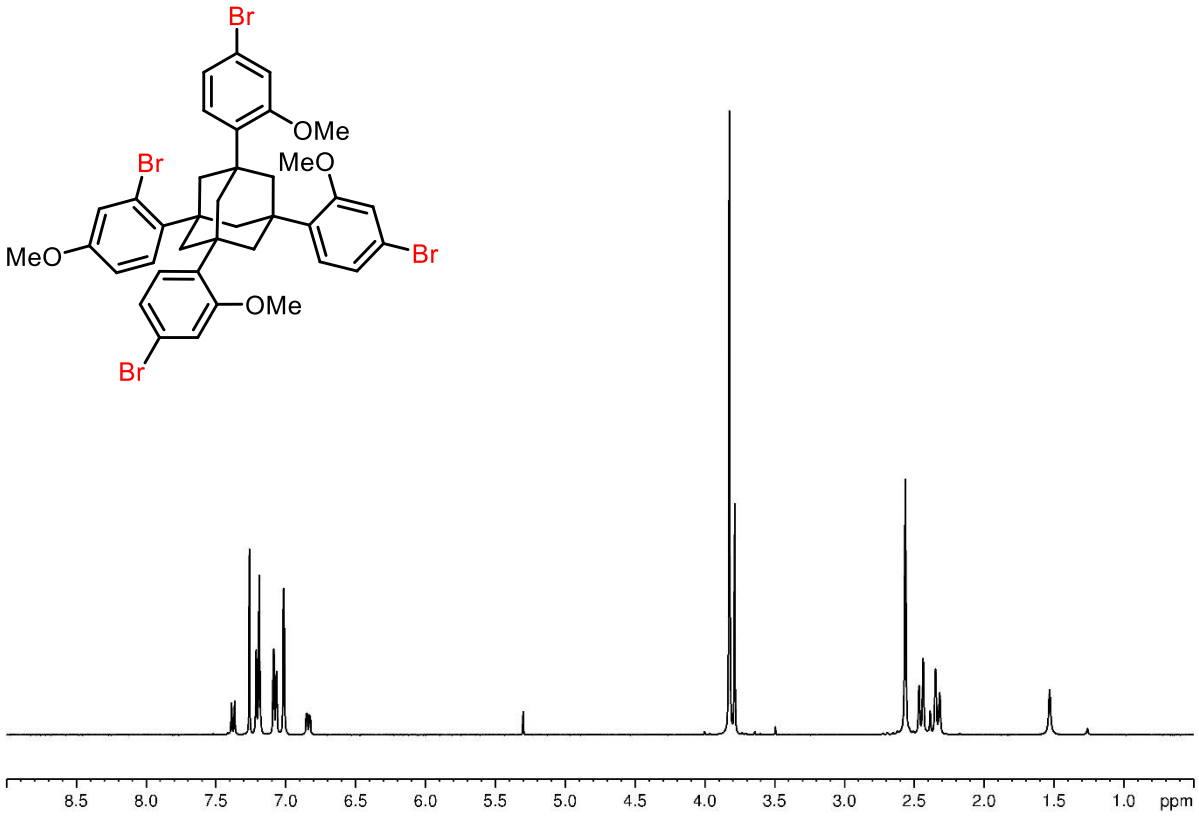

Figure S13. ^{1}H-NMR spectrum of compound **7** ($CDCl_3$, 400 MHz).

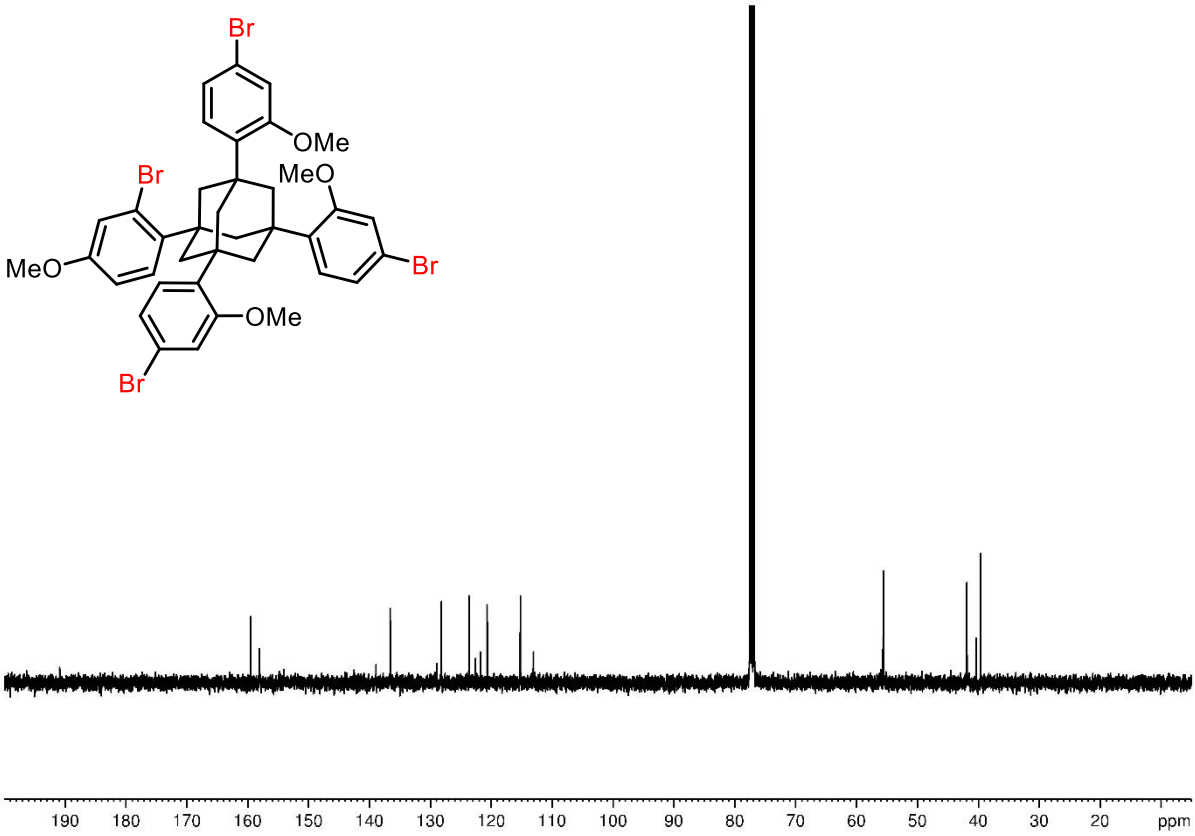

Figure S14. ^{13}C-NMR spectrum of compound **7** ($CDCl_3$, 101 MHz).

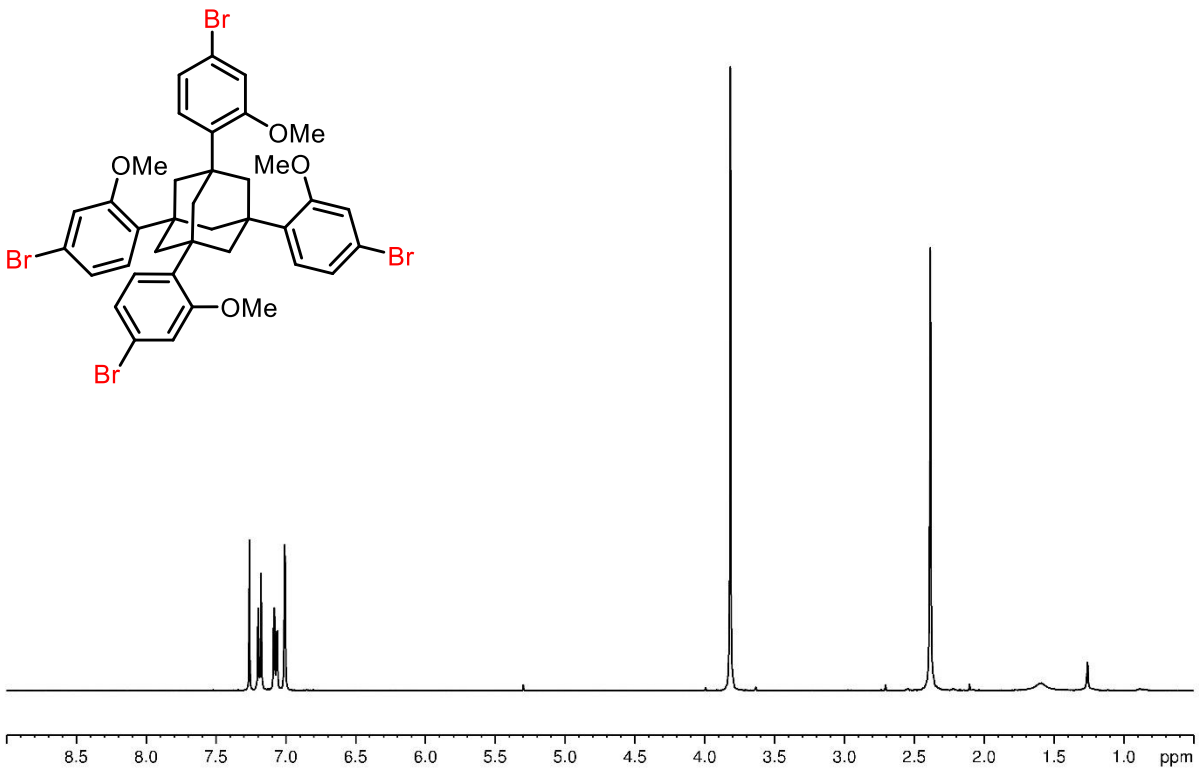

Figure S15. ^{1}H-NMR spectrum of iTBro ($CDCl_3$, 400 MHz).

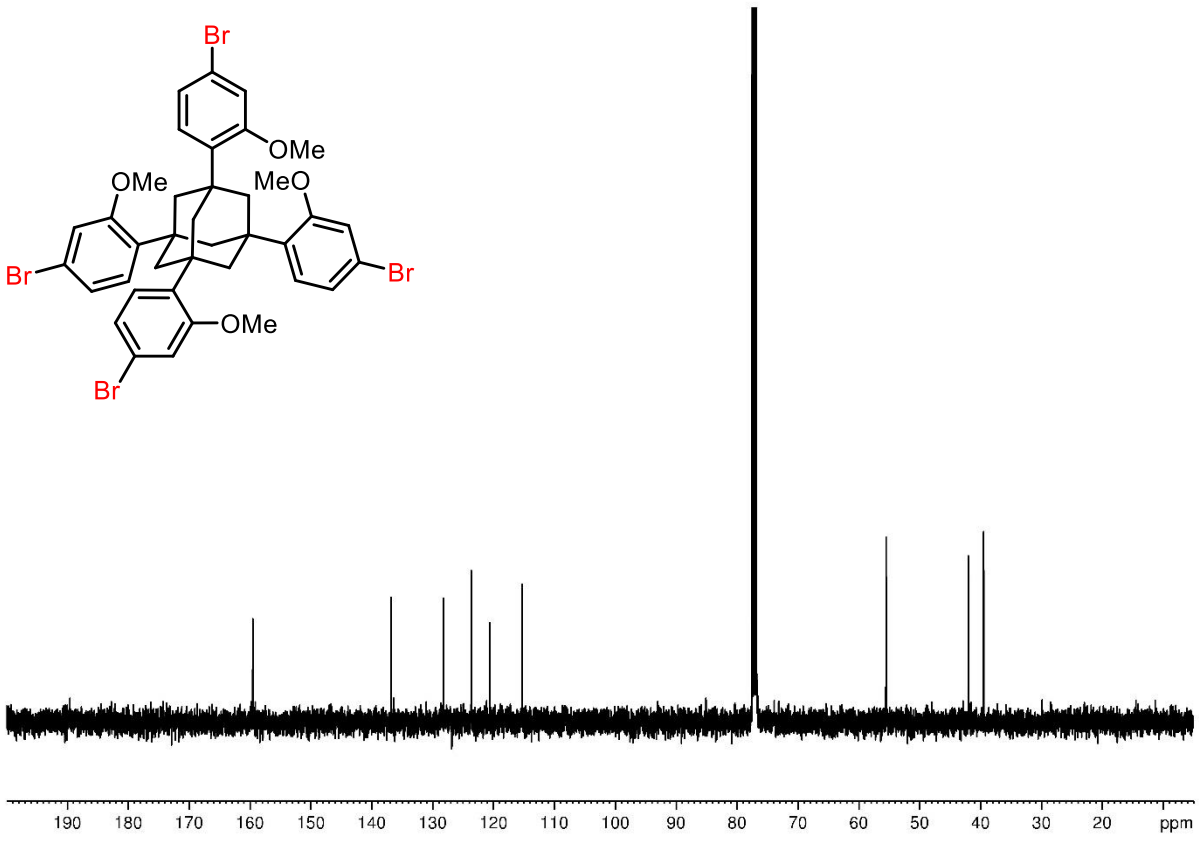

Figure S16. ^{13}C-NMR spectrum of iTBro ($CDCl_3$, 101 MHz).

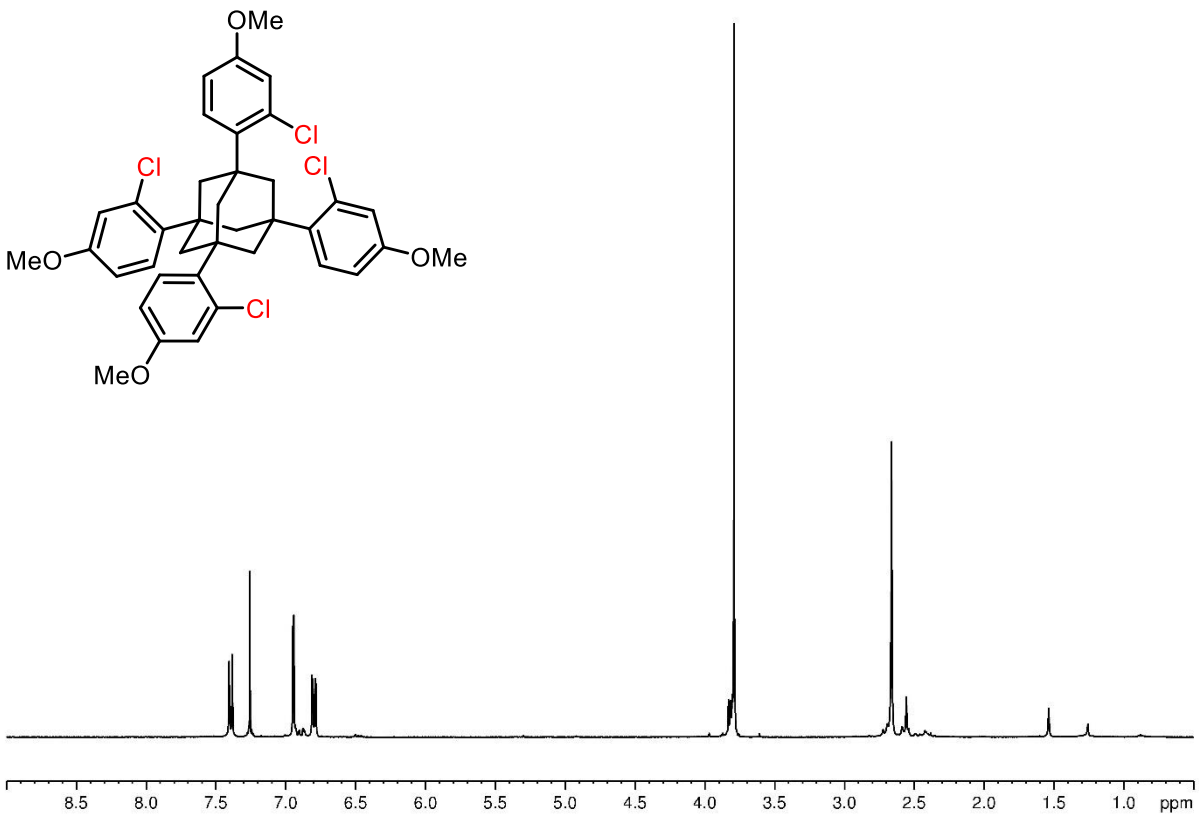

Figure S17. ^{1}H-NMR spectrum of TClM ($CDCl_3$, 400 MHz).

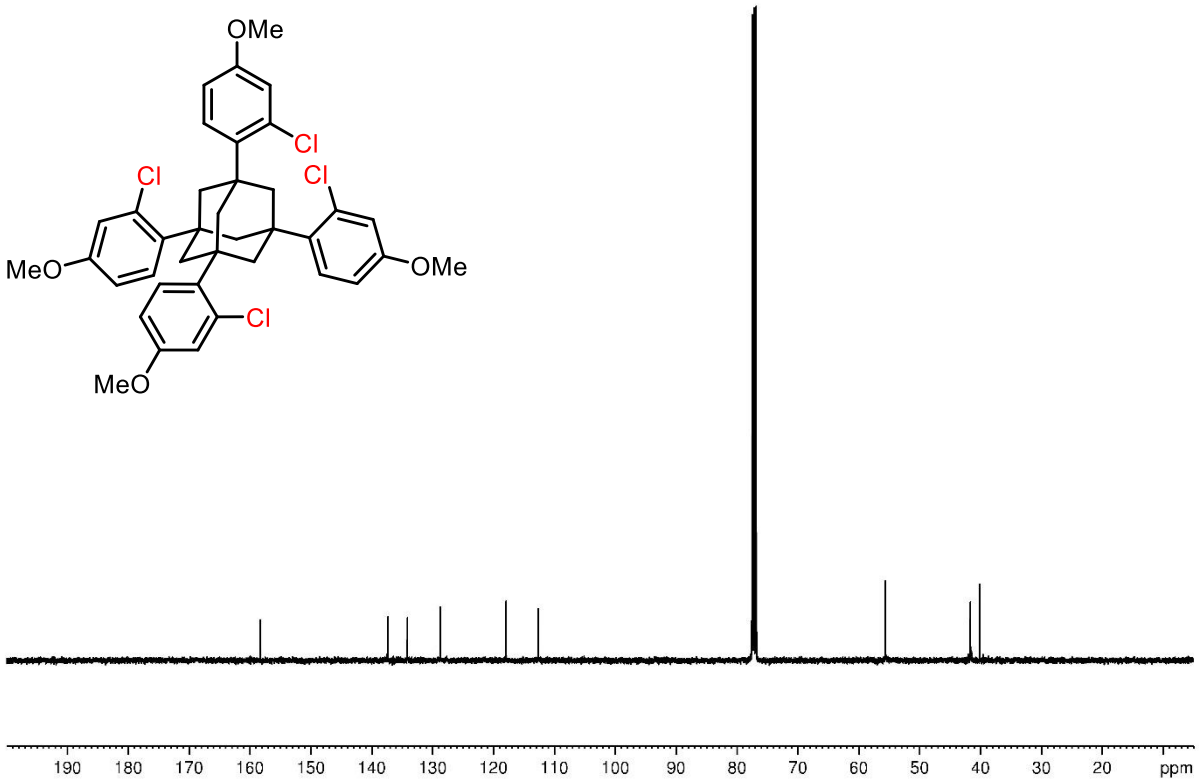

Figure S18. ^{13}C-NMR spectrum of Compound TClM ($CDCl_3$, 101 MHz).

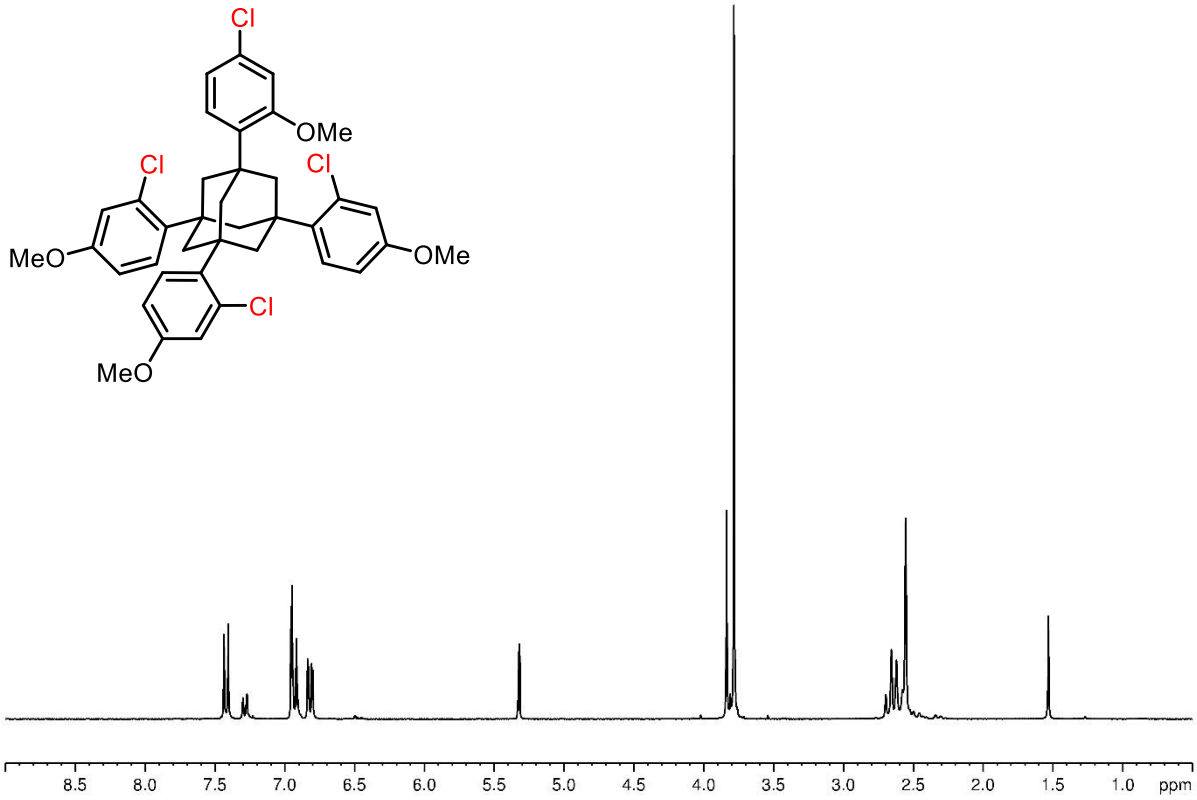

Figure S19. ^{1}H-NMR spectrum of compound **16** (CD_2Cl_2, 300 MHz).

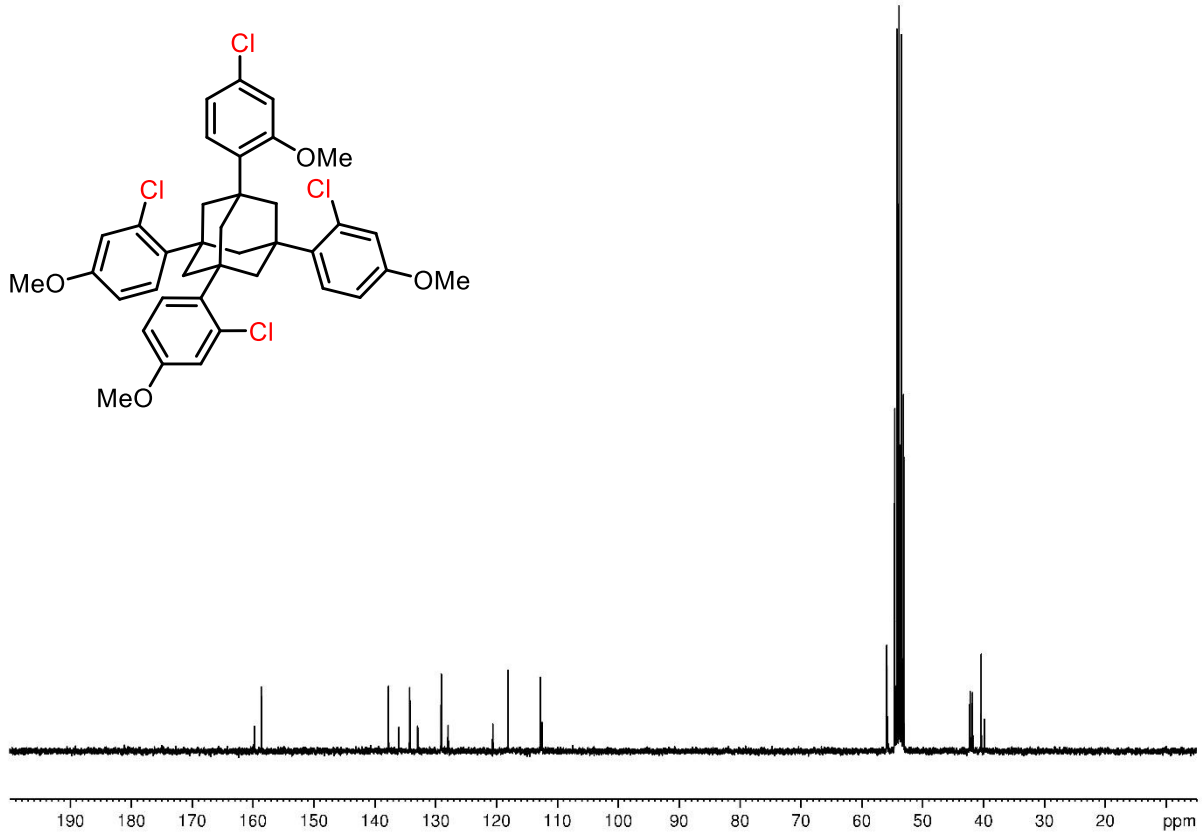

Figure S20. ^{13}C-NMR spectrum of compound **16** (CD_2Cl_2, 75 MHz).

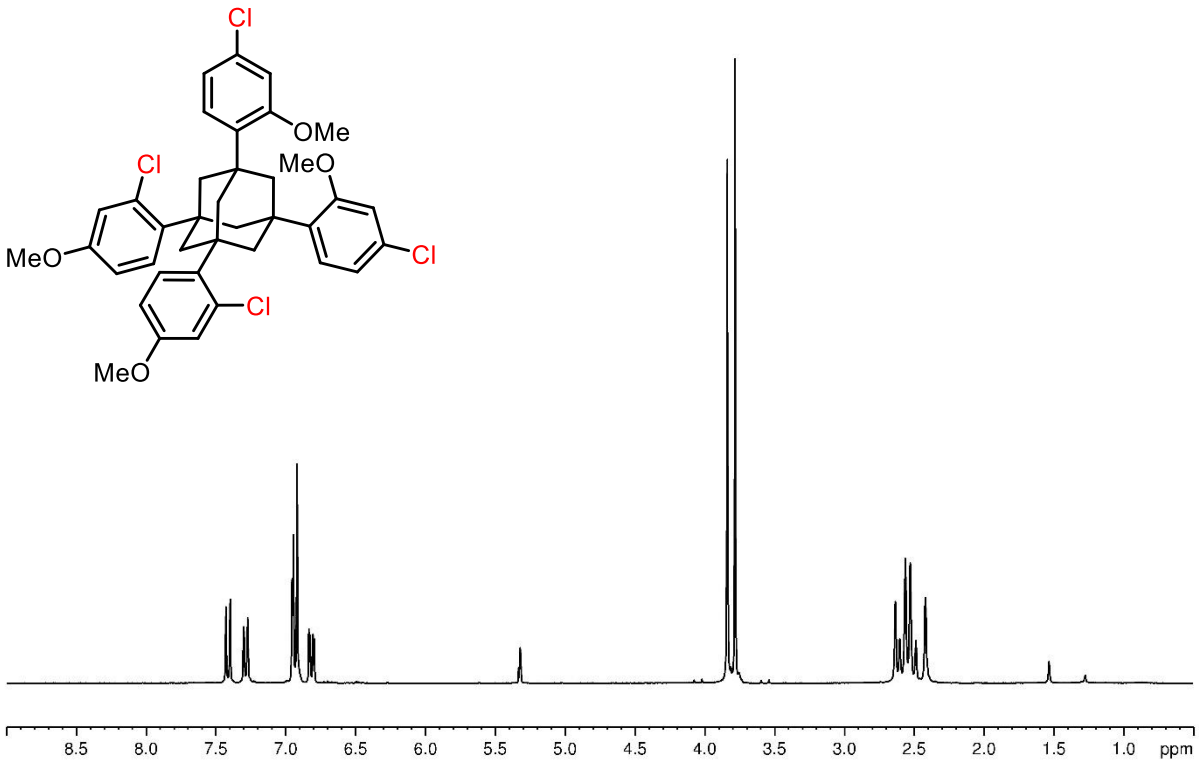

Figure S21. ^{1}H-NMR spectrum of compound **17** (CD_2Cl_2, 300 MHz).

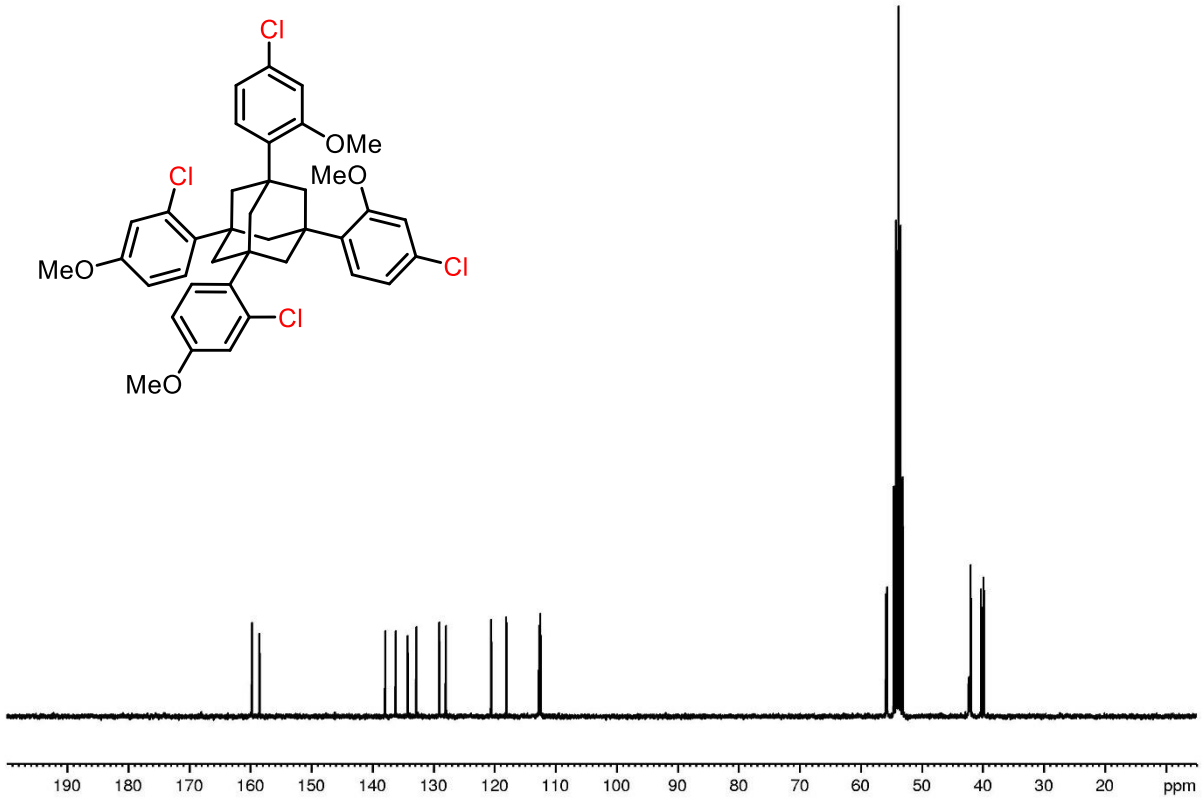

Figure S22. ^{13}C-NMR spectrum of compound **17** ($CDCl_3$, 75 MHz).

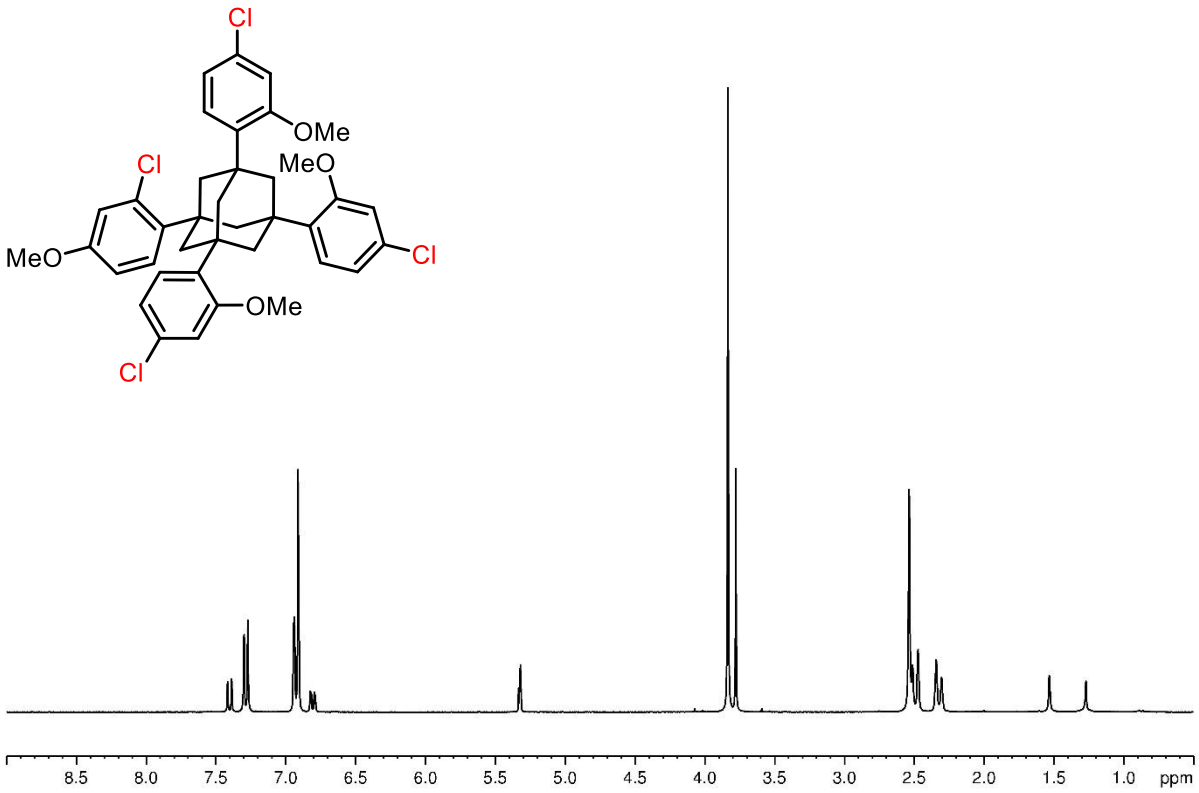

Figure S23. ^{1}H-NMR spectrum of compound **18** (CD_2Cl_2, 300 MHz).

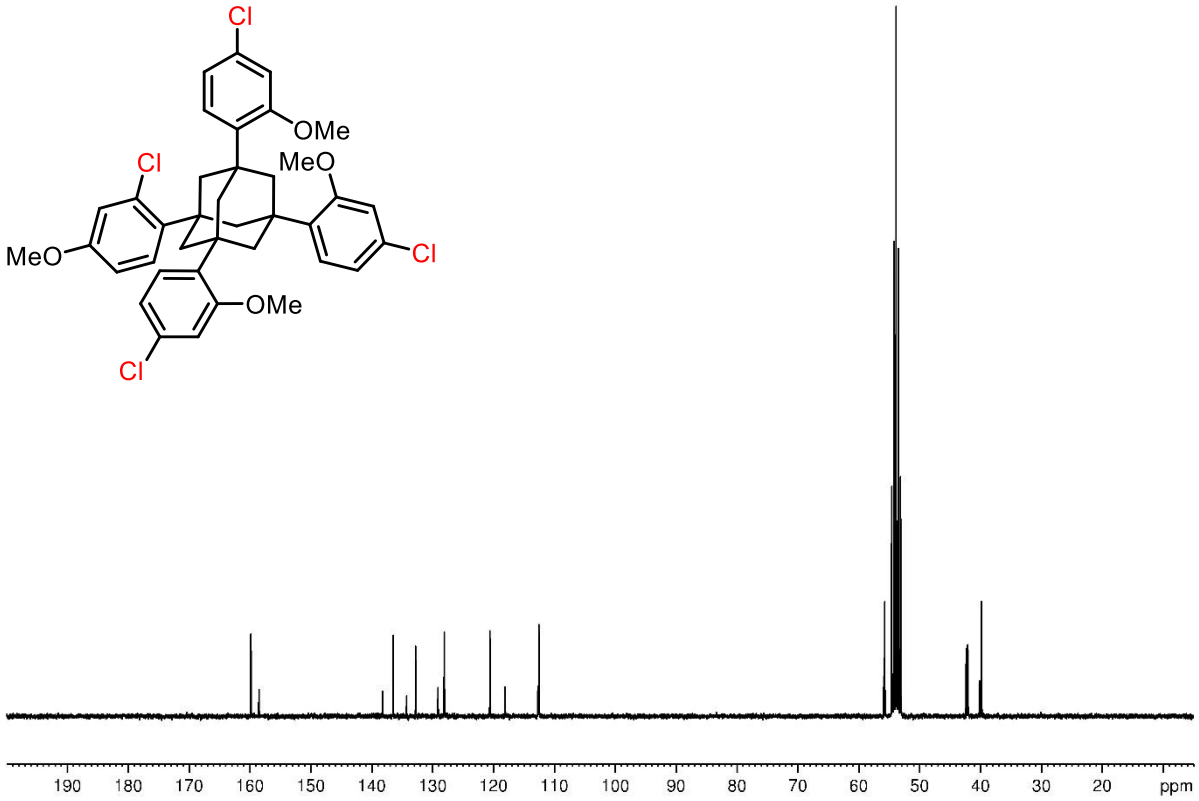

Figure S24. ^{13}C-NMR spectrum of compound **18** (CD_2Cl_2, 75 MHz).

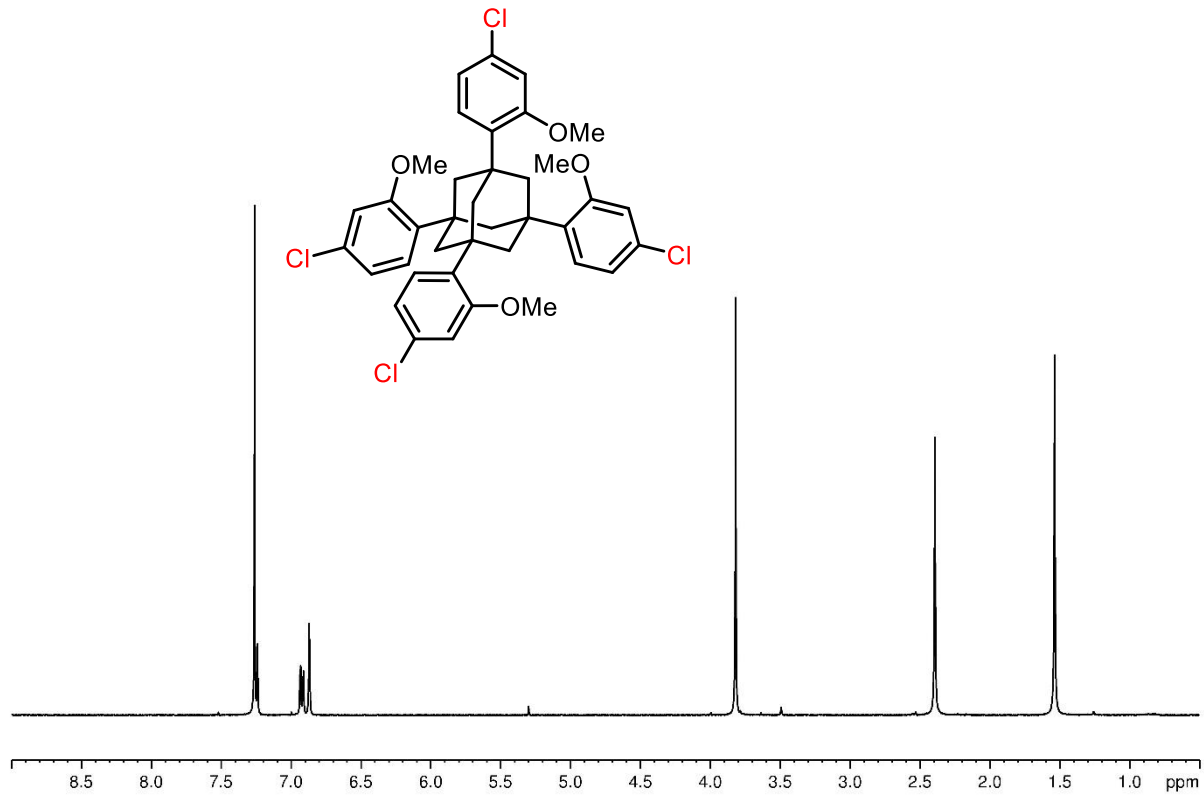

Figure S25. ^{1}H-NMR spectrum of iTClM ($CDCl_3$, 400 MHz).

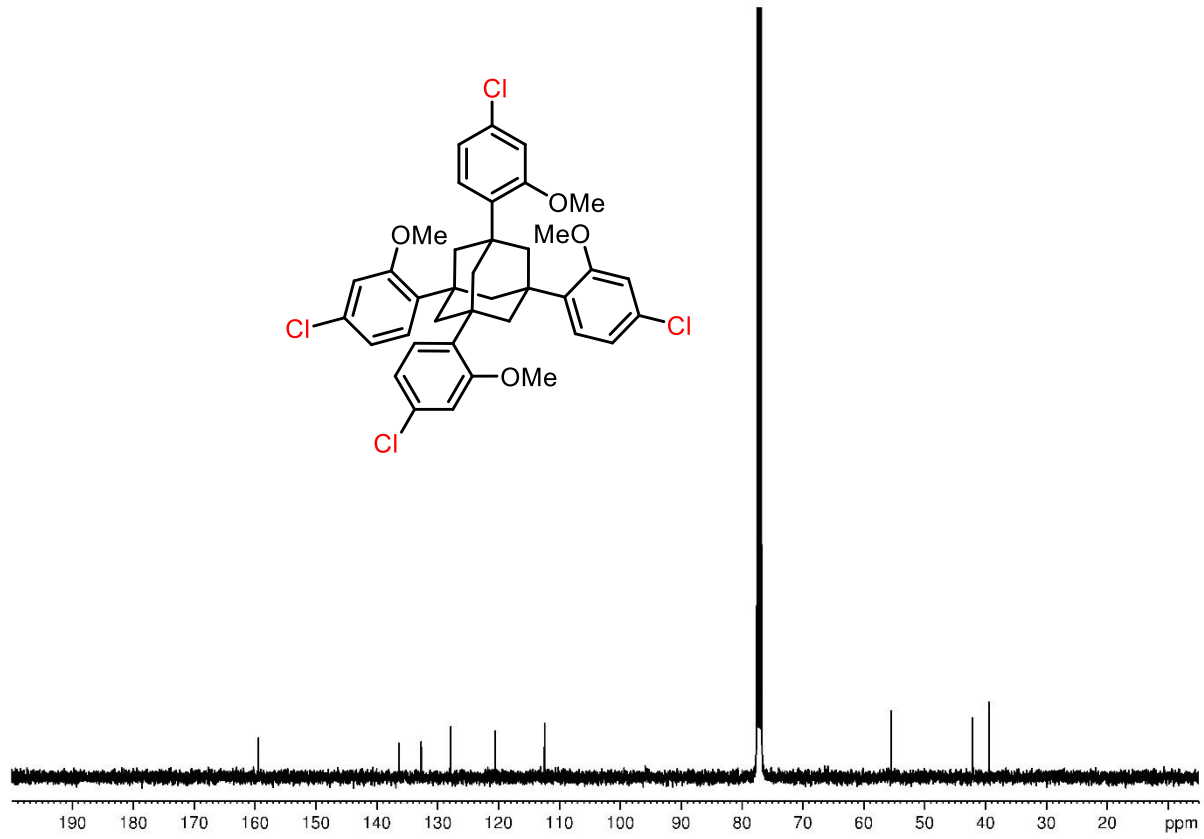

Figure S26. ^{13}C-NMR spectrum of iTClM ($CDCl_3$, 101 MHz).

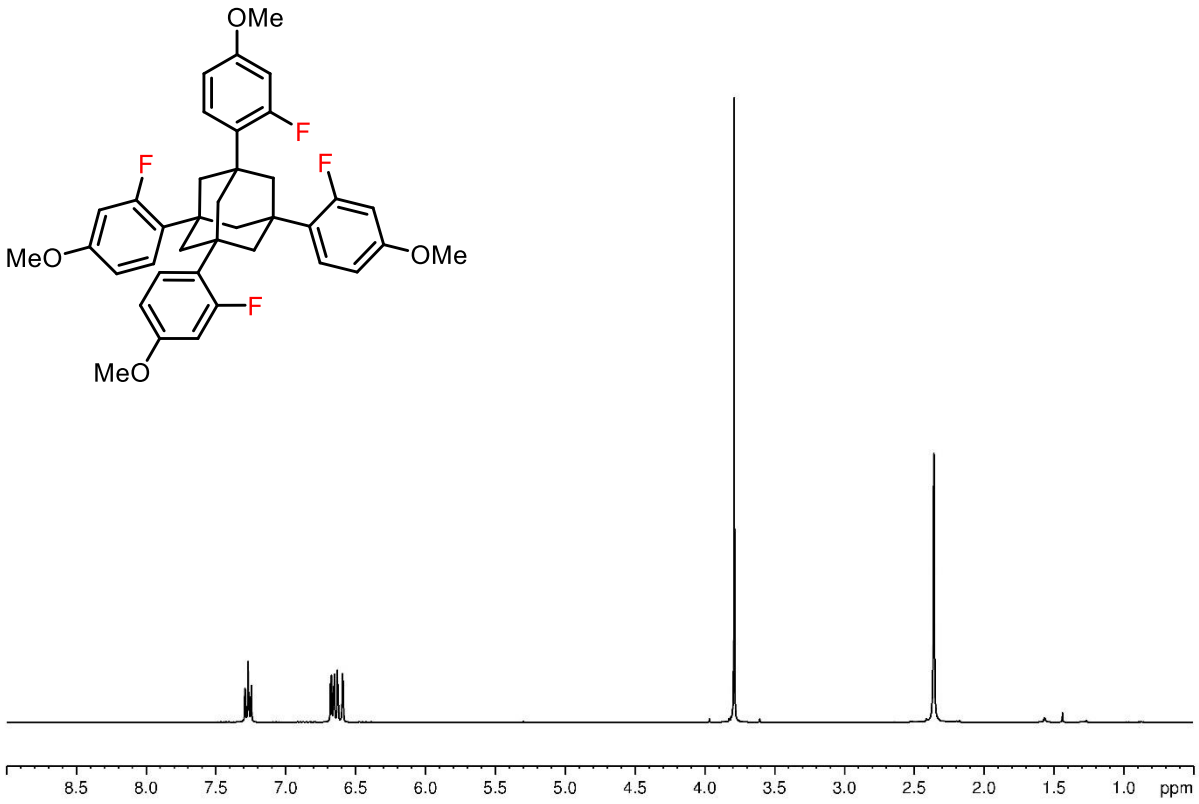

Figure S27. ^{1}H-NMR spectrum of TFM ($CDCl_3$, 400 MHz).

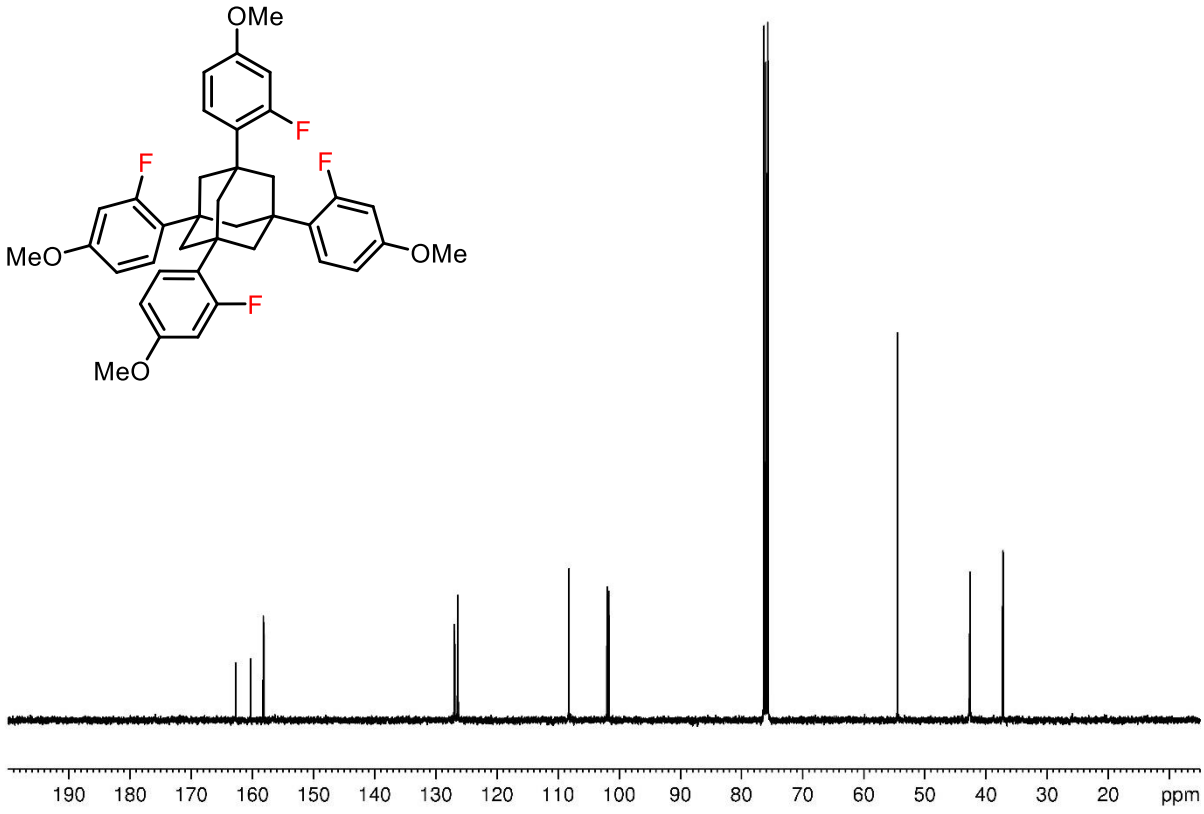

Figure S28. ^{13}C-NMR spectrum of TFM ($CDCl_3$, 101 MHz).

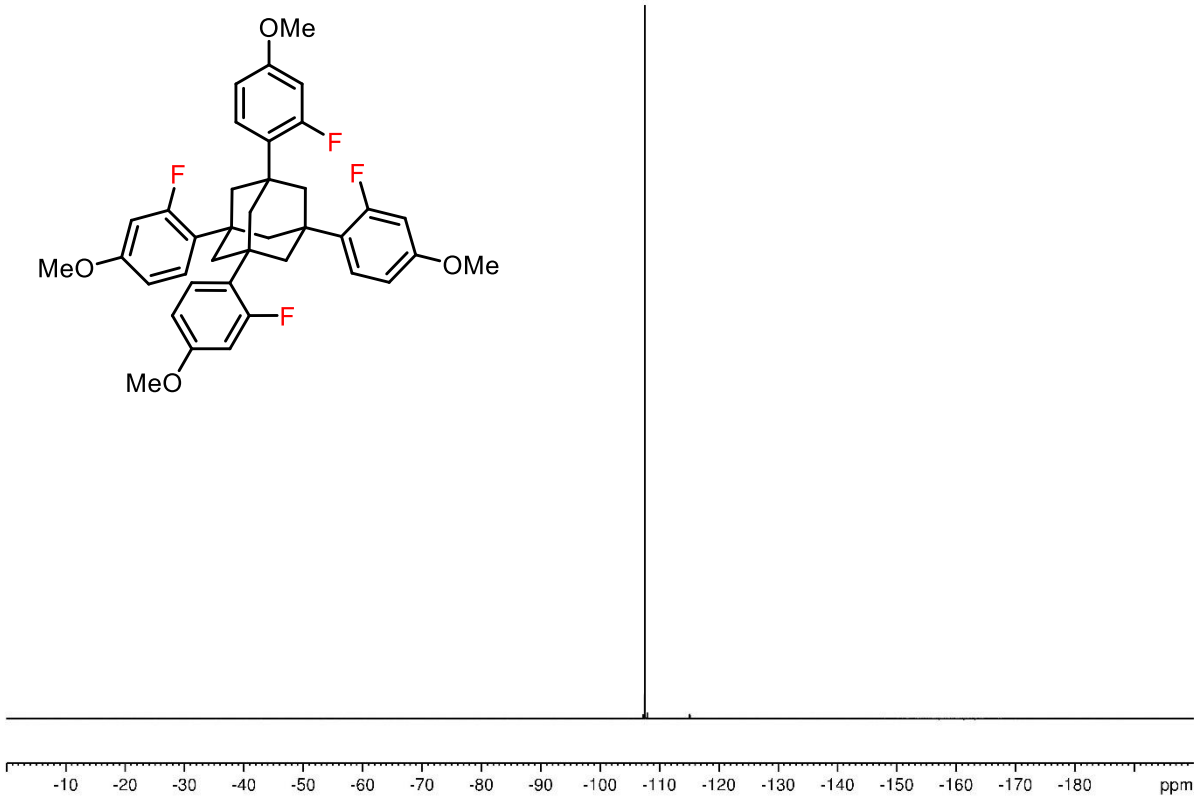

Figure S29. ^{19}F-NMR spectrum of Compound TFM ($CDCl_3$, 376 MHz).

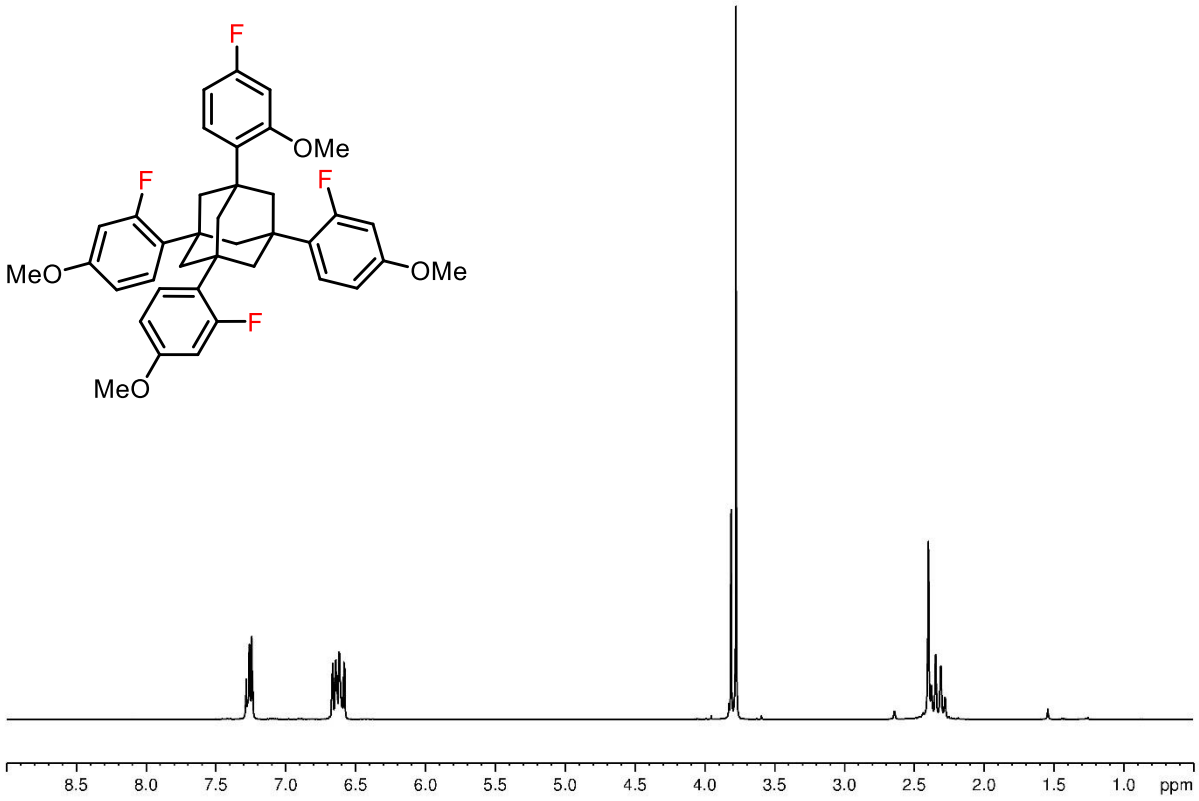

Figure S30. ^{1}H-NMR spectrum of compound **22** ($CDCl_3$, 400 MHz).

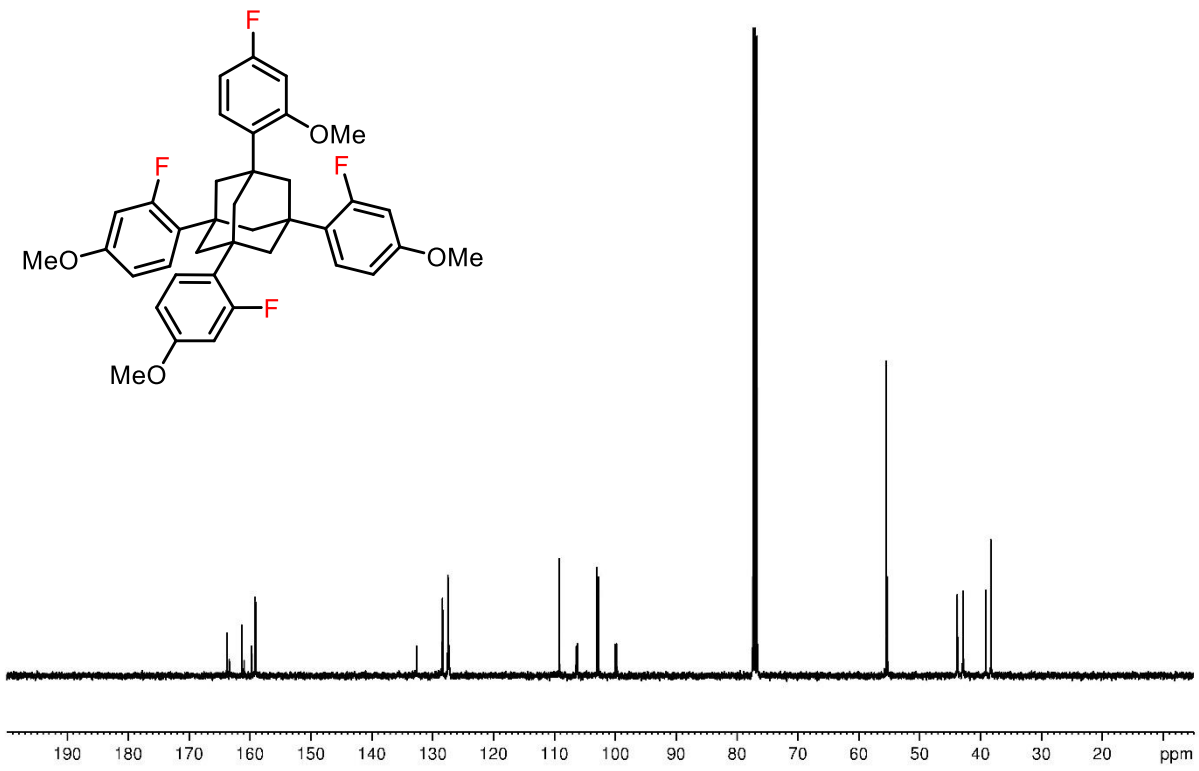

Figure S31. ^{13}C-NMR spectrum of compound **22** ($CDCl_3$, 101 MHz).

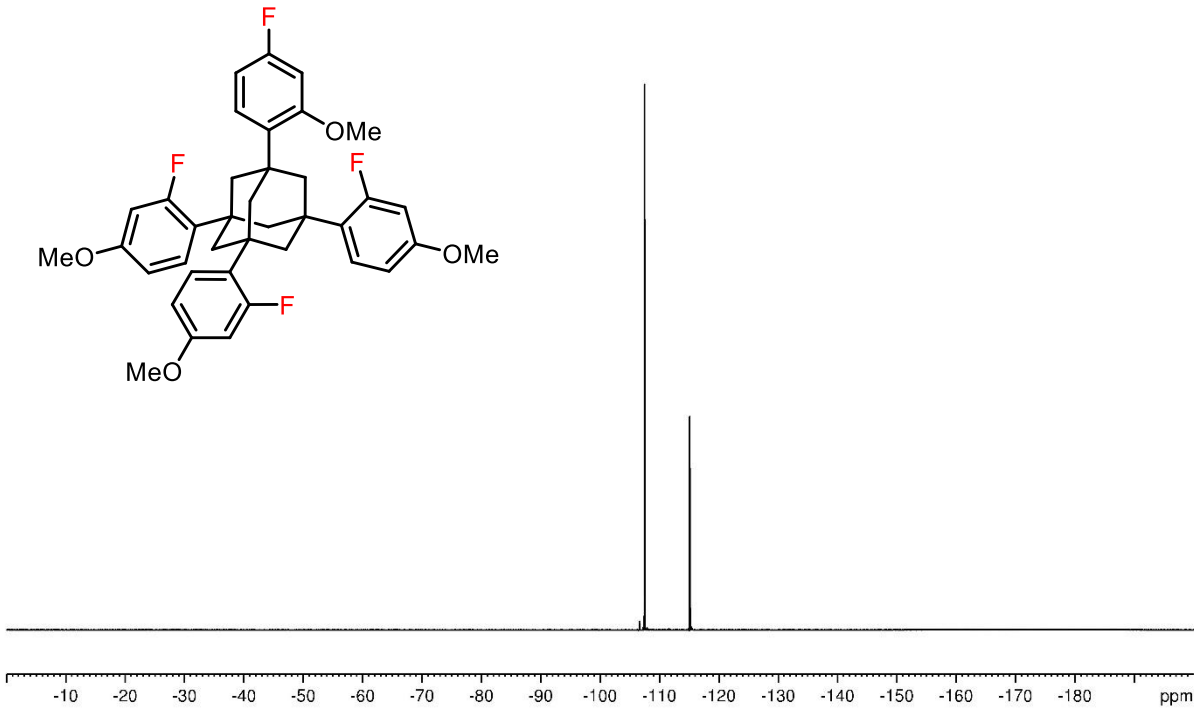

Figure S32. ^{19}F-NMR spectrum of compound **22** ($CDCl_3$, 376 MHz).

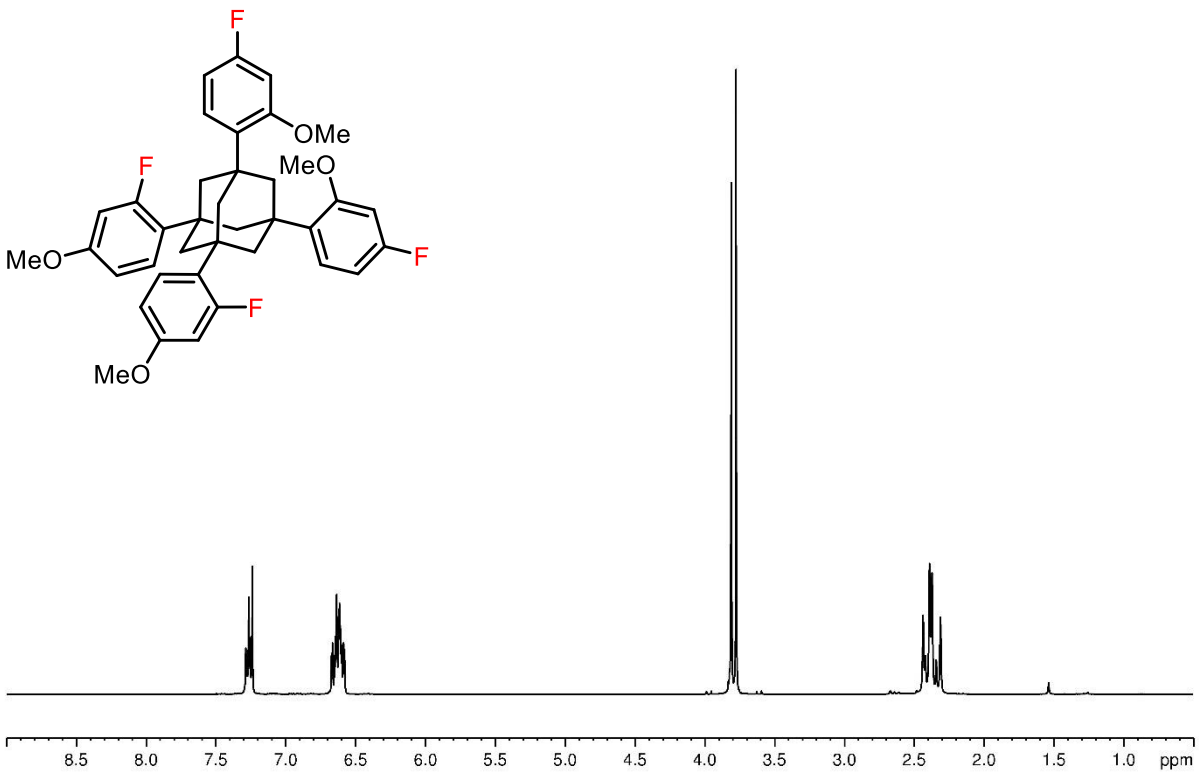

Figure S33. ^{1}H-NMR spectrum of compound **23** ($CDCl_3$, 400 MHz).

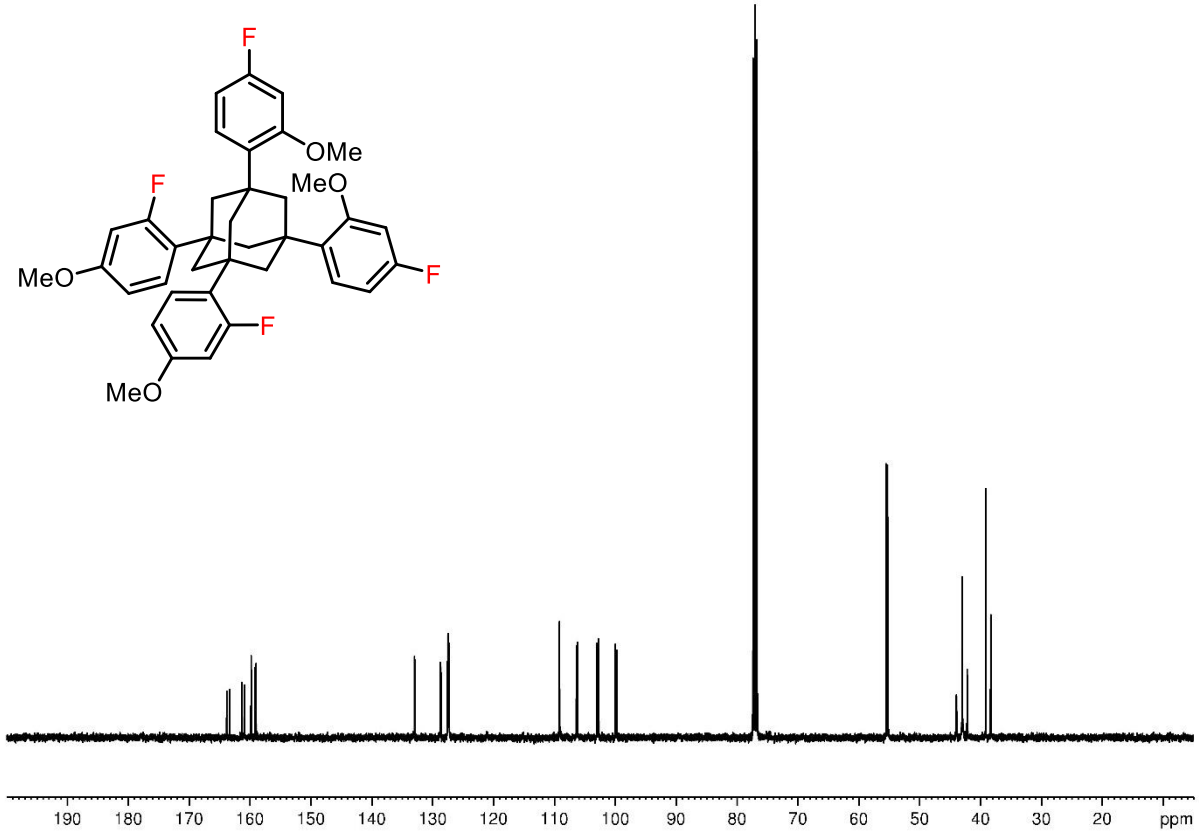

Figure S34. ^{13}C-NMR spectrum of compound **23** ($CDCl_3$, 101 MHz).

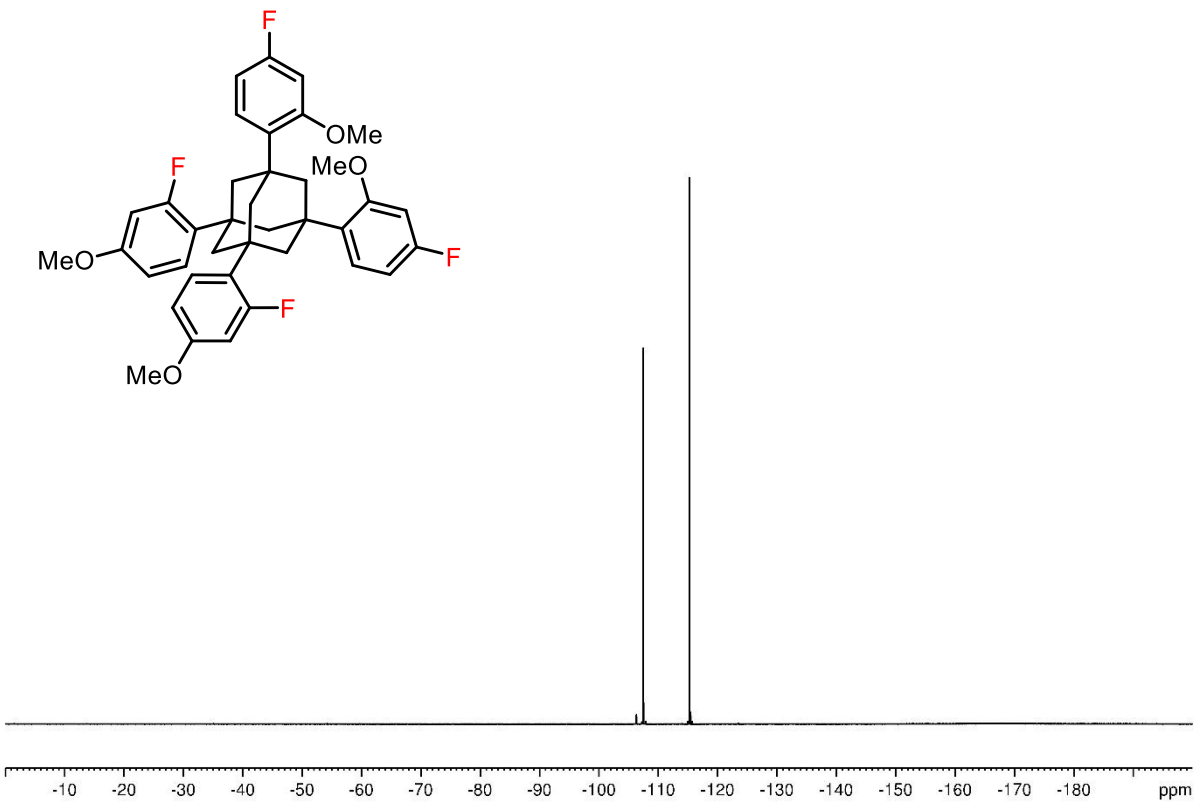

Figure S35. ^{19}F-NMR spectrum of compound **23** ($CDCl_3$, 376 MHz).

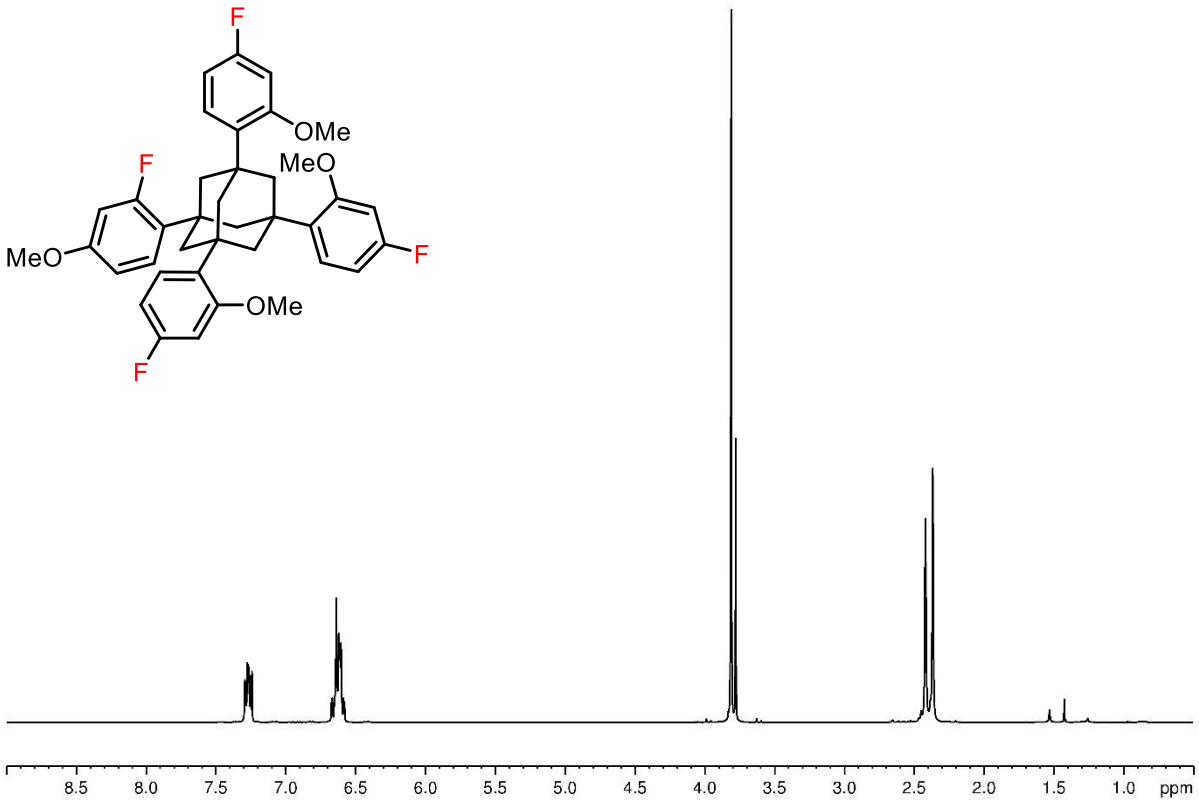

Figure S36. ^{1}H-NMR spectrum of compound **23** ($CDCl_3$, 400 MHz).

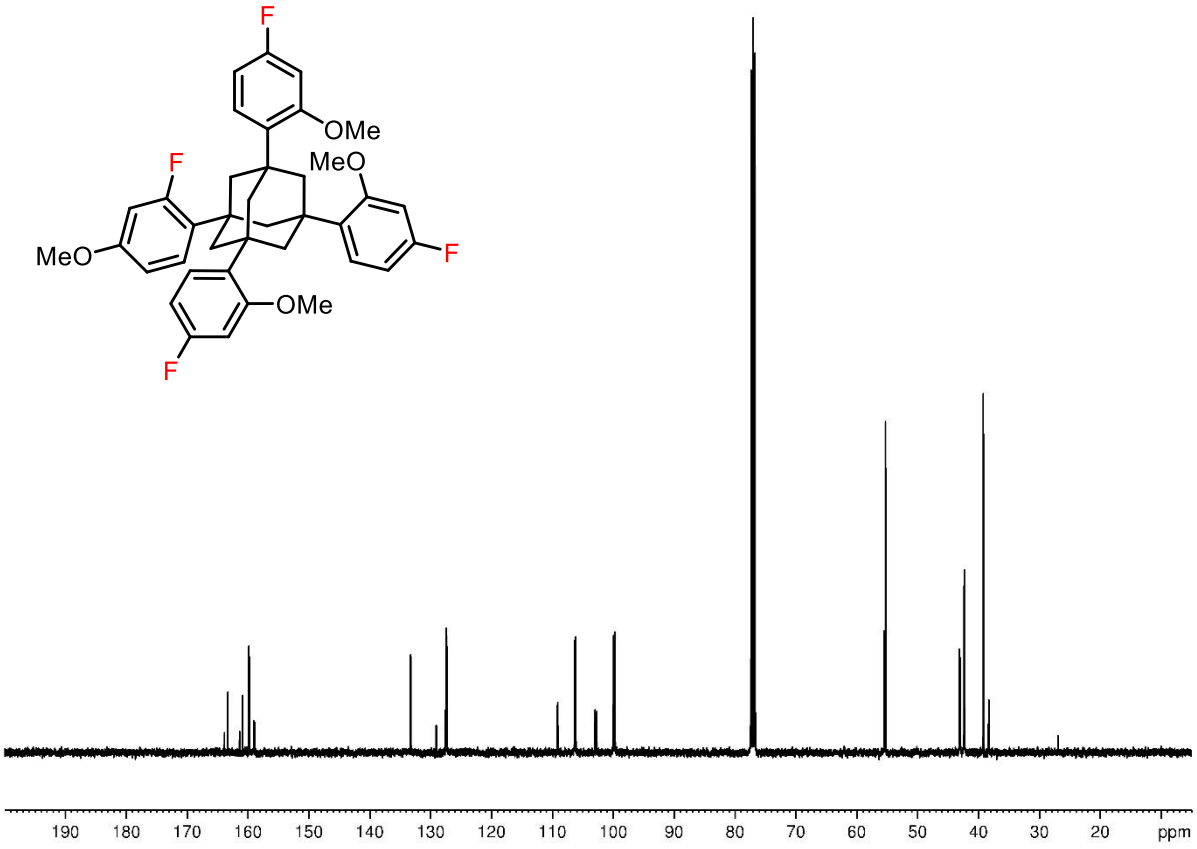

Figure S37. ^{13}C-NMR spectrum of compound **23** ($CDCl_3$, 101 MHz).

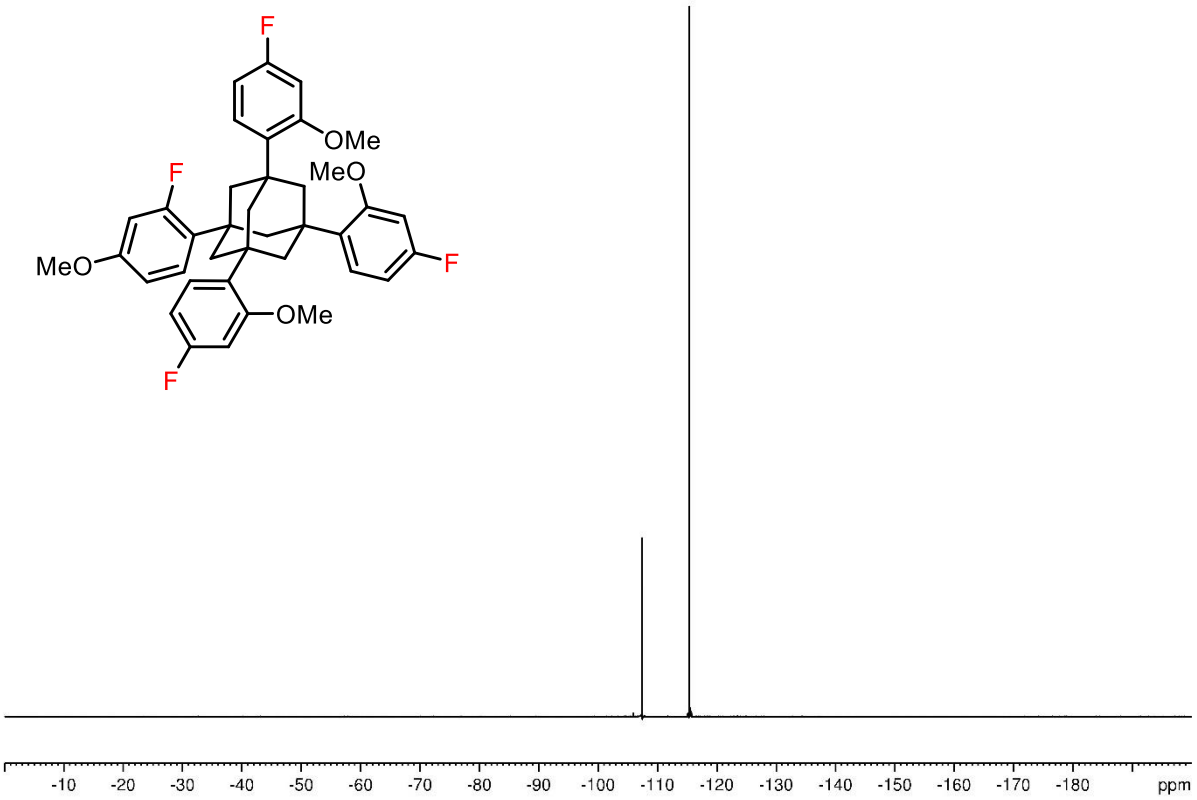

Figure S38. ^{19}F-NMR spectrum of compound **23** ($CDCl_3$, 376 MHz).

2. X-ray Crystal Structures

Strcutural details for solvate-free form of iTBro
CCDC deposition number 2251141

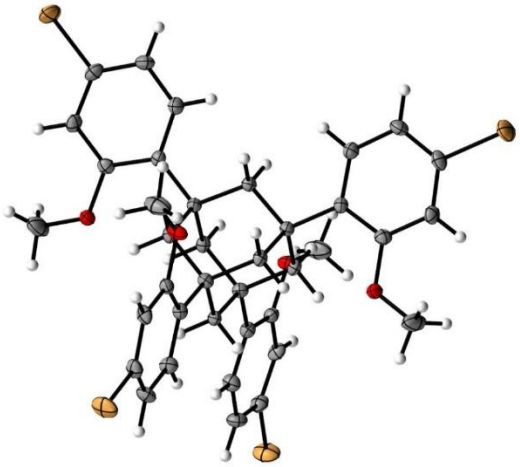

Figure S39. One molecule of iTBro in the crystal structure of without inclusion.

Empirical formula	$C_{38}H_{36}Br_4O_4$	
Formula weight	876.31	
Temperature	140(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Tetragonal, I4(1)/a	
Unit cell dimensions	a = 19.4378(10) Å	alpha = 90 °
	b =19.4378(10) Å	beta = 90 °
	c = 9.1102(6) Å	gamma = 90 °
Volume	3442.1(4) Å ^3	
Z, Calculated density	4, 1.691 Mg/m^3	
Absorption coefficient	4.717 mm^-1	
F(000)	1744	
Crystal size	0.240 x 0.081 x 0.048 mm	
Theta range for data collection	2.095 to 26.454 °	
Limiting indices	-24<=h<=24, -24<=k<=24, -11<=l<=11	
Reflections collected / unique	24082 / 1777 [R(int) = 0.0651]	
Completeness to theta = 25.242	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8645 and 0.4337	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1777 / 0 / 106	
Goodness-of-fit on F^2	1.007	
Final R indices [I>2sigma(I)]	R1 = 0.0314, wR2 = 0.0634	
R indices (all data)	R1 = 0.0557, wR2 = 0.0674	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.377 and -0.271 e. Å^-3	

Structural details of iTBro with encapsulated dichloromethane
CCDC deposition number 2251142

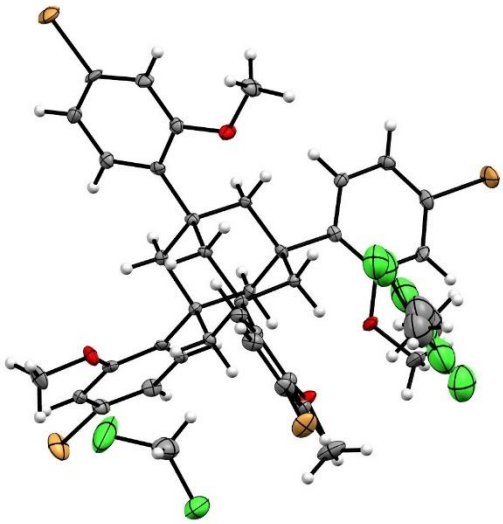

Figure S40. Structure of iTBro and two molecules of encapsulated dichloromethane

Empirical formula	$C_{39.50}H_{39}Br_4Cl_3O_4$	
Chemical formula moiety	$C_{38}H_{36}Br_4O_4$, 1.5 CH_2Cl_2	
Formula weight	1003.69	
Temperature	140(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 12.5470(9) Å	alpha = 80.520(4) °
	b = 12.5556(10) Å	beta = 77.892(4) °
	c = 13.9319(12) Å	gamma = 66.382(3) °
Volume	1958.2(3) Å ^3	
Z, Calculated density	2, 1.702 Mg/m^3	
Absorption coefficient	4.355 mm^-1	
F(000)	998	
Crystal size	0.310 x 0.242 x 0.150 mm	
Theta range for data collection	1.777 to 26.440 °	
Limiting indices	-15<=h<=15, -14<=k<=15, -17<=l<=17	
Reflections collected / unique	32061 / 8011 [R(int) = 0.0436]	
Completeness to theta = 25.242	99.9 %	
Absorption correction	Numerical	
Max. and min. transmission	0.6239 and 0.4113	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8011 / 102 / 501	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0413, wR2 = 0.0773	
R indices (all data)	R1 = 0.0701, wR2 = 0.0856	
Extinction coefficient	0.0024(2)	
Largest diff. peak and hole	2.051 and -1.961 e. Å ^-3	