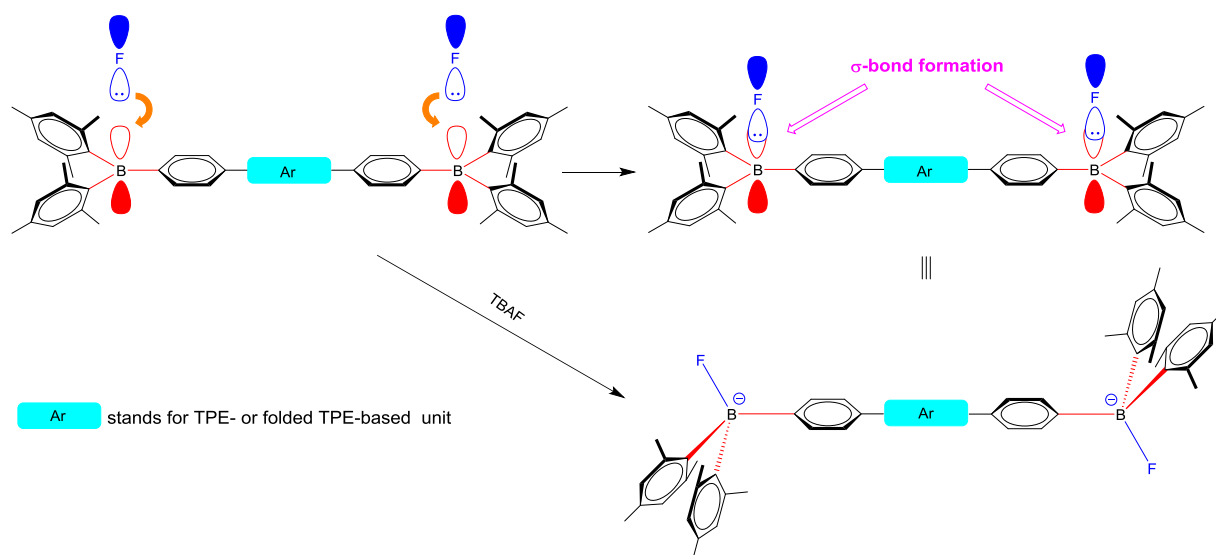


Electronic Supplementary Information (ESI)

General information

Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl under dry nitrogen immediately prior to use. ^1H and ^{13}C NMR spectra were measured on a Bruker AV 500 spectrometer in appropriate deuterated solution at room temperature. High resolution mass spectra (HRMS) were recorded on a GCT premier CAB048 mass spectrometer operating in MALDI-TOF mode. UV-vis absorption spectra were measured on a Shimadzu UV-2600 spectrophotometer. Photoluminescence spectra were recorded on a Horiba Fluoromax-4 spectrofluorometer. Fluorescence quantum yields were measured using a Hamamatsu absolute PL quantum yield spectrometer C11347 Quantaaurus-QY. The fluorescence lifetimes were determined by the compact fluorescence lifetime spectrometer C11367 of Hamamatsu. The molecular structures were optimized by B3LYP/6-31G (d, p), and molecular orbitals were calculated at the B3LYP/6-31+G (d,p) level. All the calculations were performed using Gaussian 09 package.



Scheme S1. Schematic representation of the complexing of tricoordinate organoboranes with Lewis base (e.g. TBAF).

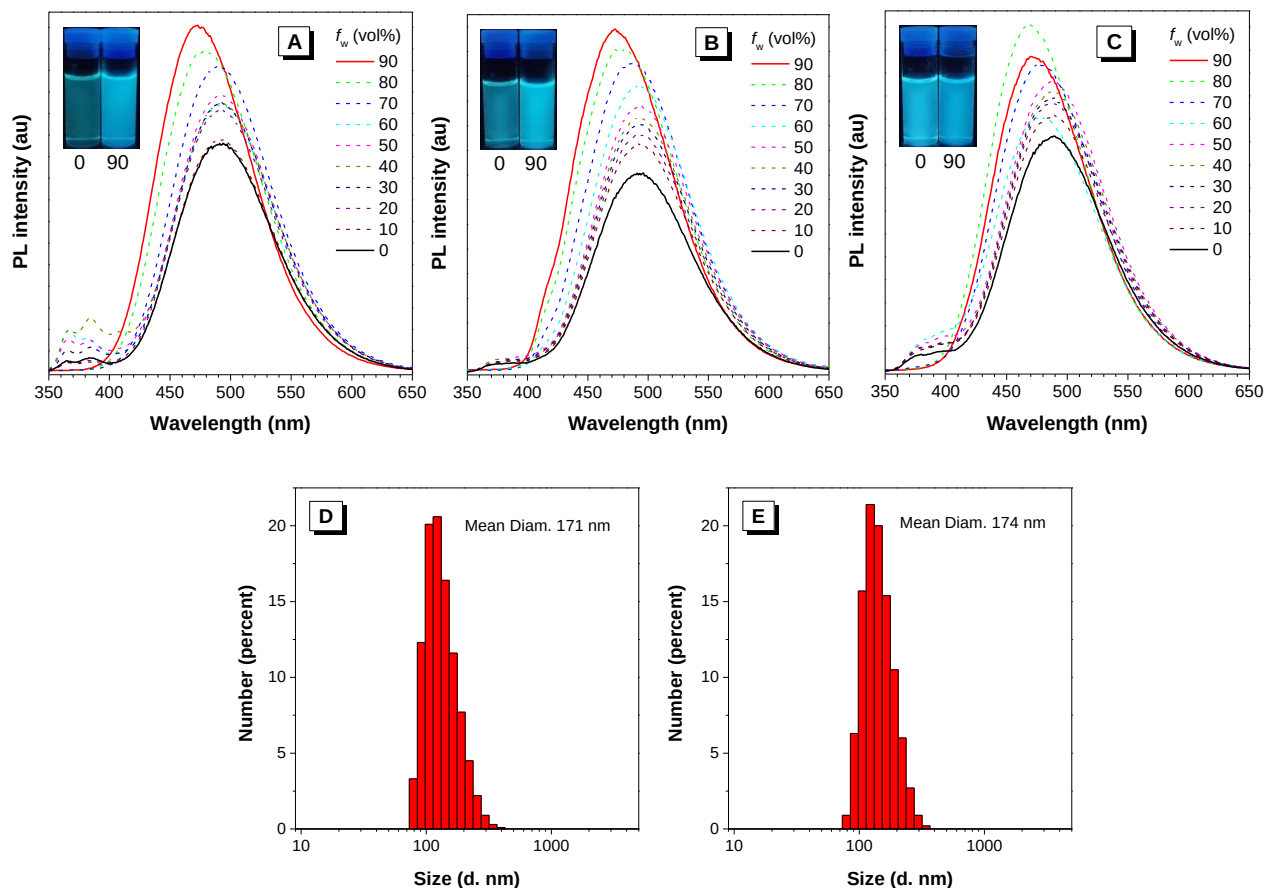


Fig. S1 PL spectra of (A) *f*-TPEF, (B) *f*-TPEC, and (C) *f*-TPEI in THF/water mixtures with different water fractions (f_w), $\lambda_{\text{ex}} = 330 \text{ nm}$. Inset: Photos of them in THF/water mixtures ($f_w = 0$ and 90%), taken under the illumination of a UV lamp (365 nm). Nanoparticle size distributions of (D) *f*-TPEB and (E) *l*-TPEB in THF/water mixtures ($f_w = 90\%$) at a concentration of $10 \mu\text{M}$.

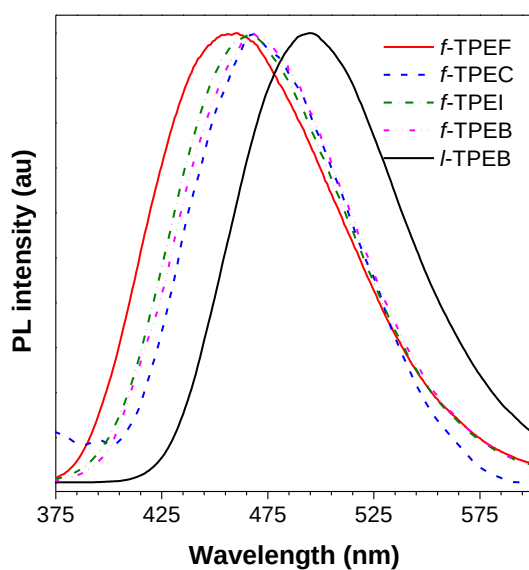


Fig. S2 PL spectra of *f*-TPEF, *f*-TPEC, *f*-TPEI, *f*-TPEB and *l*-TPEB in the film state, $\lambda_{\text{ex}} = 320 \text{ nm}$.

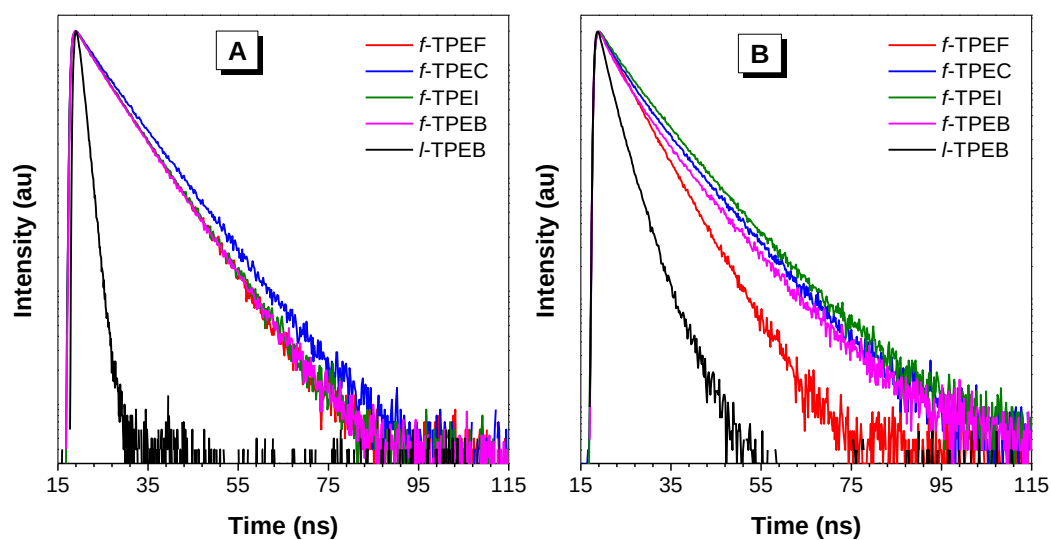


Fig. S3 Fluorescence decay curves of folded TPE derivatives and *l*-TPEB (A) in THF (10 μM) and (B) in film state.

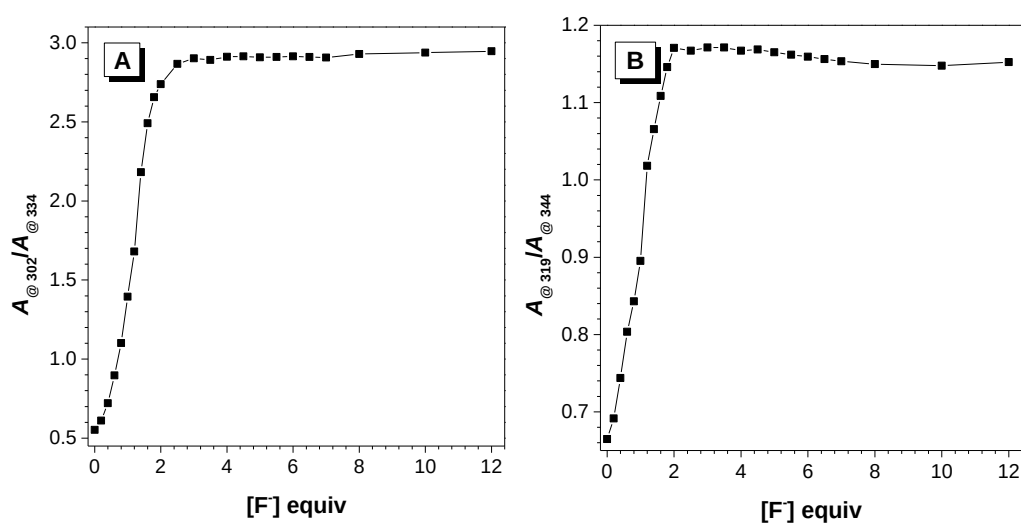


Fig. S4 Relative absorbance changes of (A) *f*-TPEB monitored at 302 and 334 nm; (B) *l*-TPEB monitored at 319 and 344 nm when adding F^- .

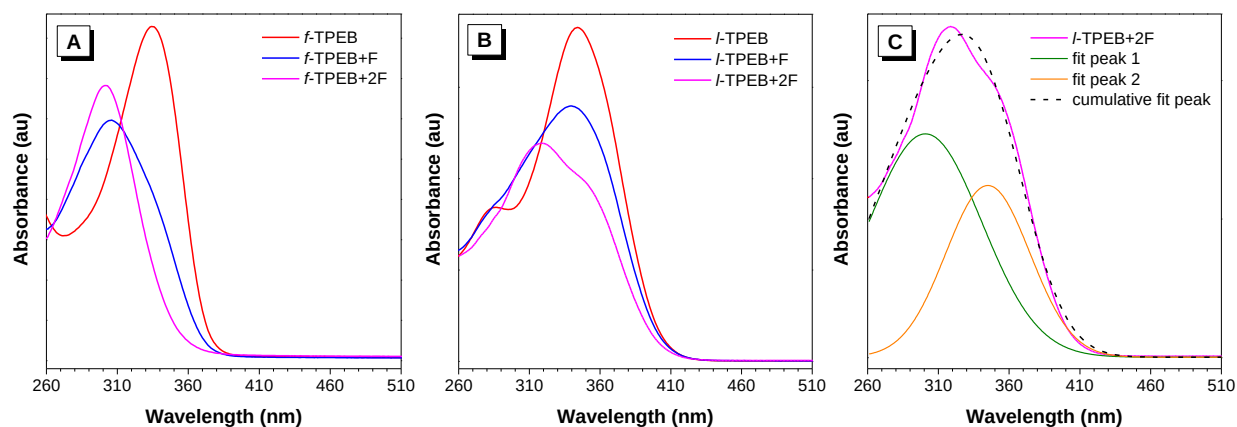


Fig. S5 (A) UV spectra of *f*-TPEB, *f*-TPEB+F and *f*-TPEB+2F; (B) UV spectra of *l*-TPEB, *l*-TPEB+F and *l*-TPEB+2F; (C) Gaussian fitting UV spectra of *l*-TPEB+2F in THF.

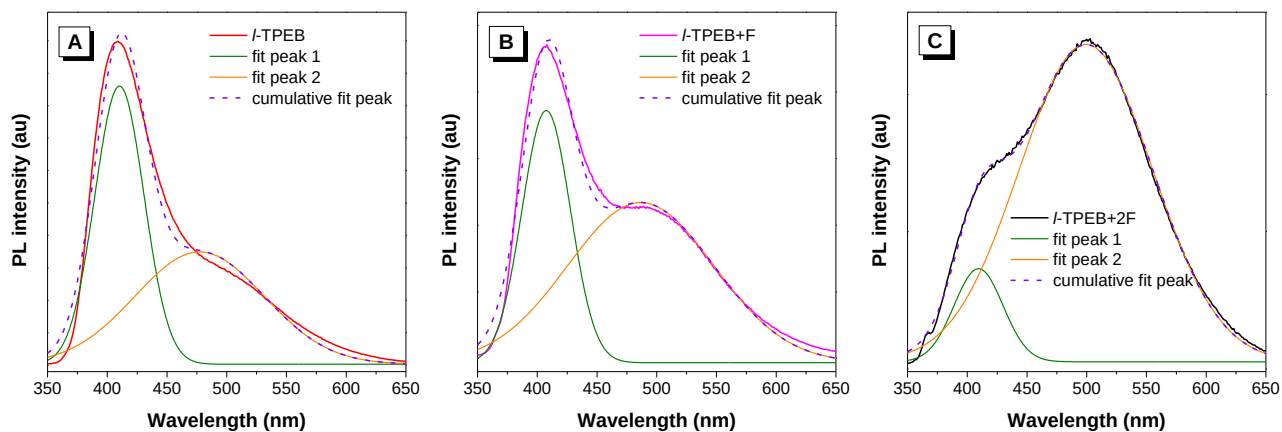


Fig. S6 Gaussian fitting PL spectra of (A) *l*-TPEB, (B) *l*-TPEB+F and (C) *l*-TPEB+2F in THF.

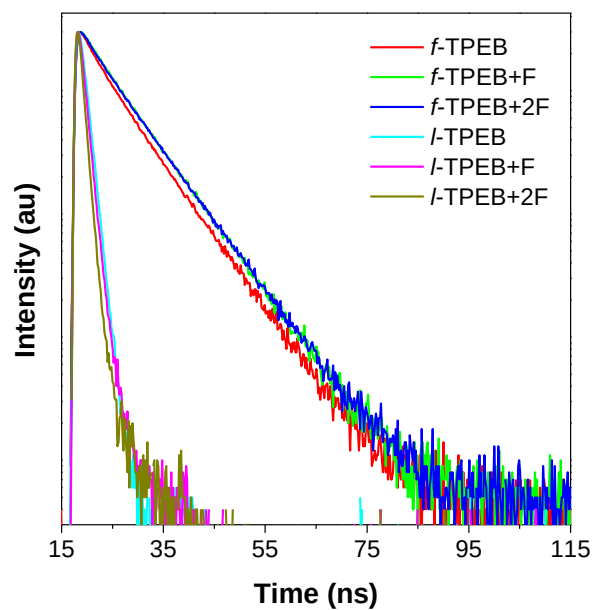


Fig. S7 Fluorescence decay curves of *f*-TPEB, *f*-TPEB+F, *f*-TPEB+2F; and *l*-TPEB, *l*-TPEB+F, *l*-TPEB+2F in THF.

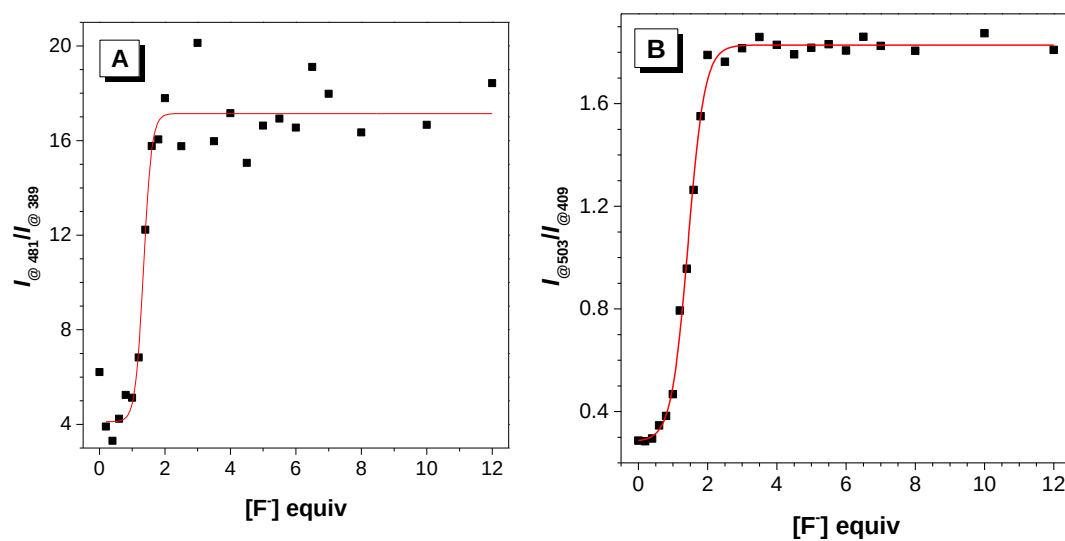


Fig. S8 Relative PL intensity changes while titrating by TBAF in THF (10 μ M), (A) *f*-TPEB monitored at 481 nm and 389 nm; (B) *l*-TPEB monitored at 503 nm and 409 nm; $\lambda_{\text{ex}} = 330$ nm.

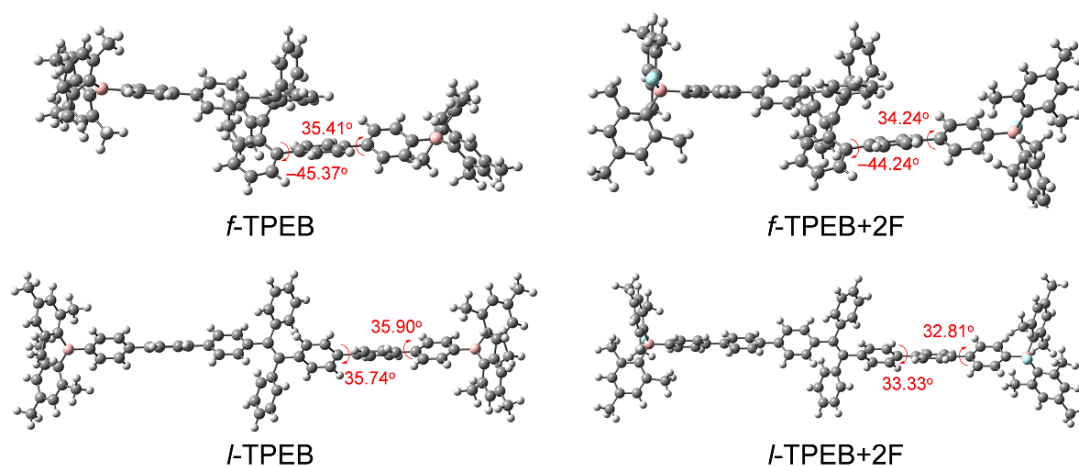


Fig. S9 Optimized molecular structures of *f*-TPEB and *l*-TPEB, as well as their corresponding binding adducts with two fluoride anions, calculated at the B3LYP/6-31G (d, p) level, selected twisted angles are also indicated.

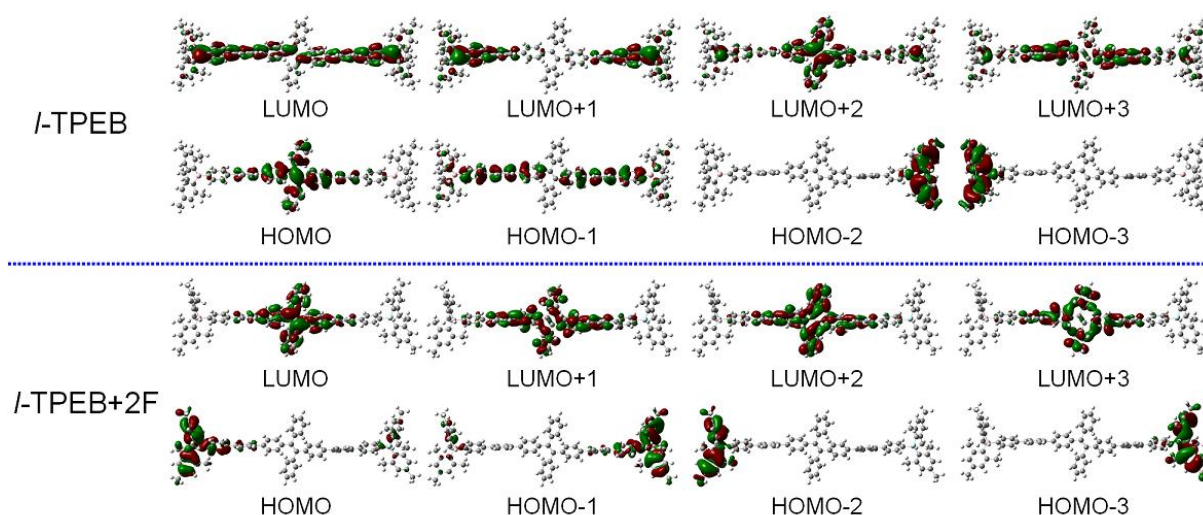


Fig. S10 Selected molecular orbital distributions of *l*-TPEB and *l*-TPEB+2F (from HOMO-3 to LUMO+3), calculated at the B3LYP/6-31+G (d, p) level.

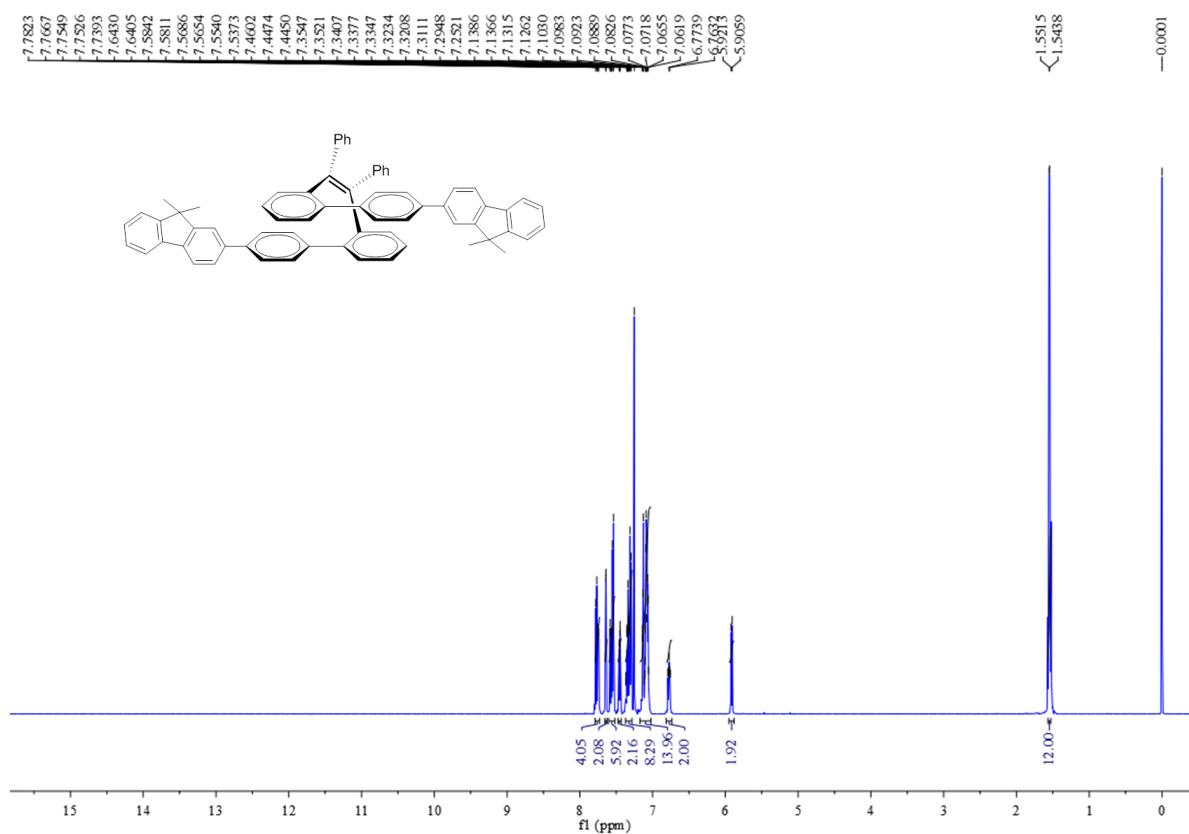


Fig. S11 ¹H NMR spectrum of *f*-TPEF (500 MHz, CDCl₃).

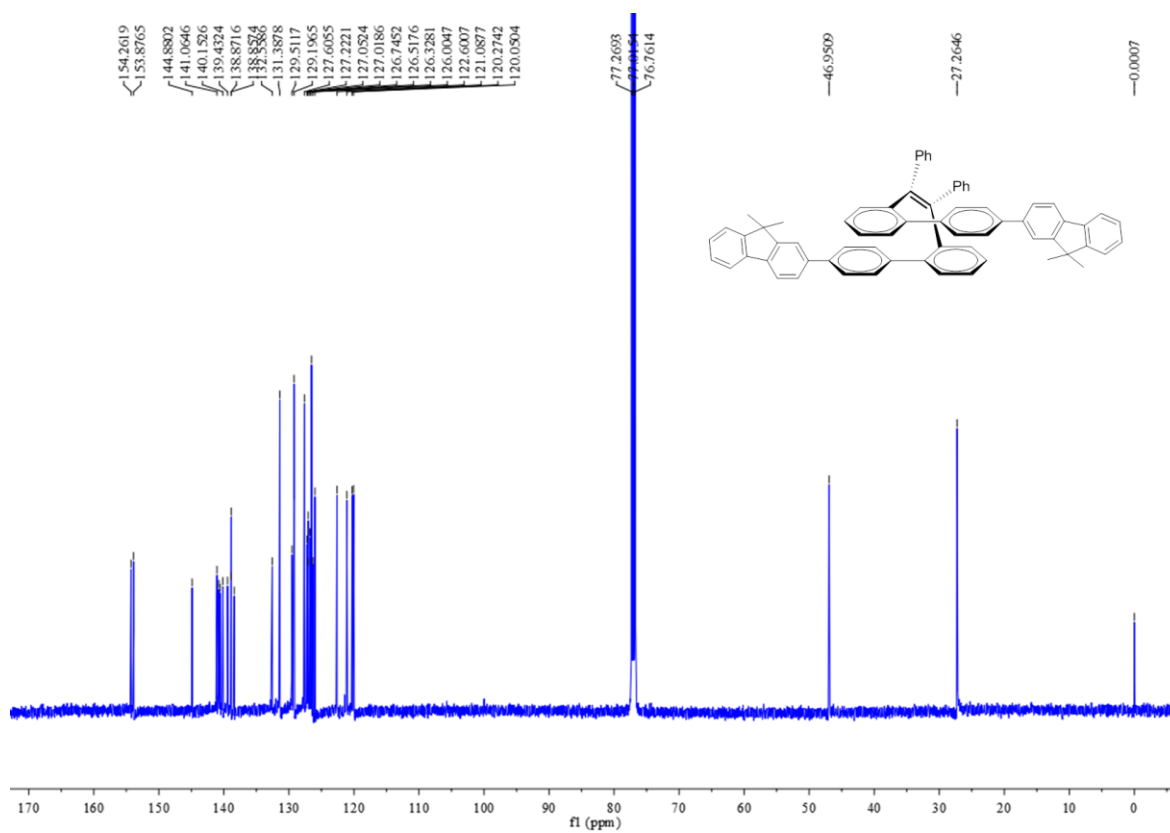


Fig. S12 ¹³C NMR spectrum of *f*-TPEF (125 MHz, CDCl₃).

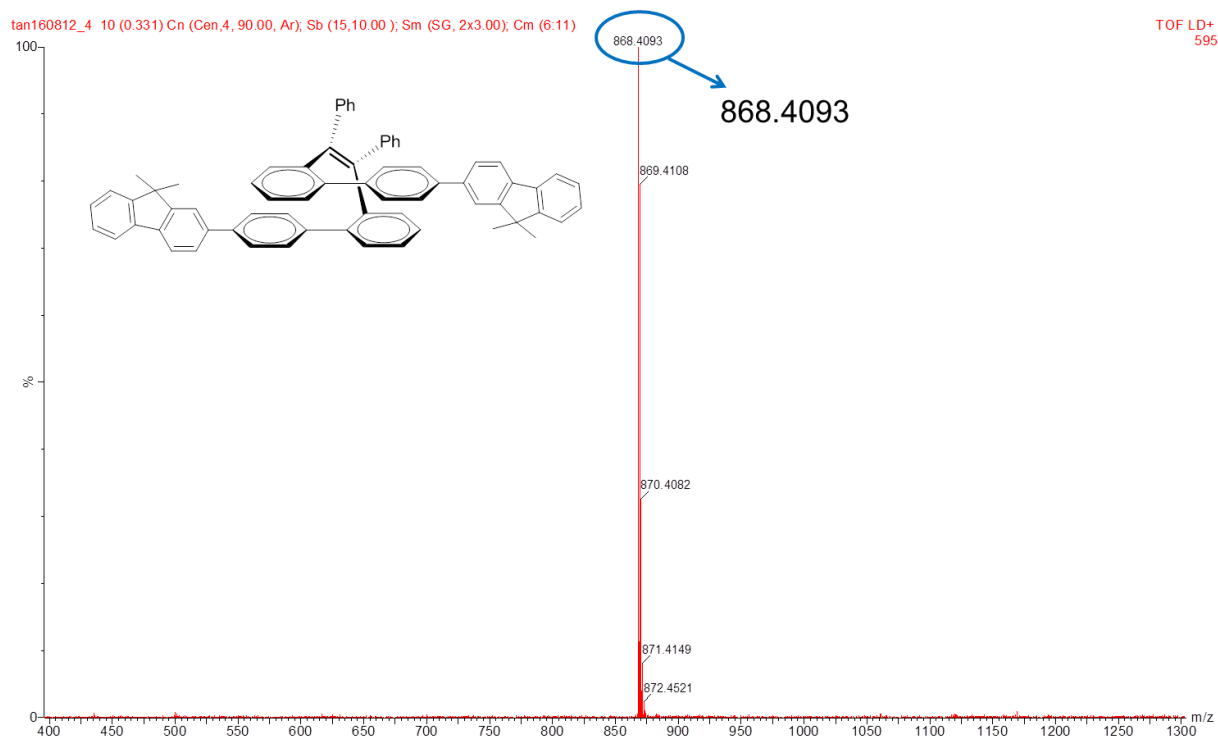


Fig. S13 HRMS spectrum of *f*-TPEF.

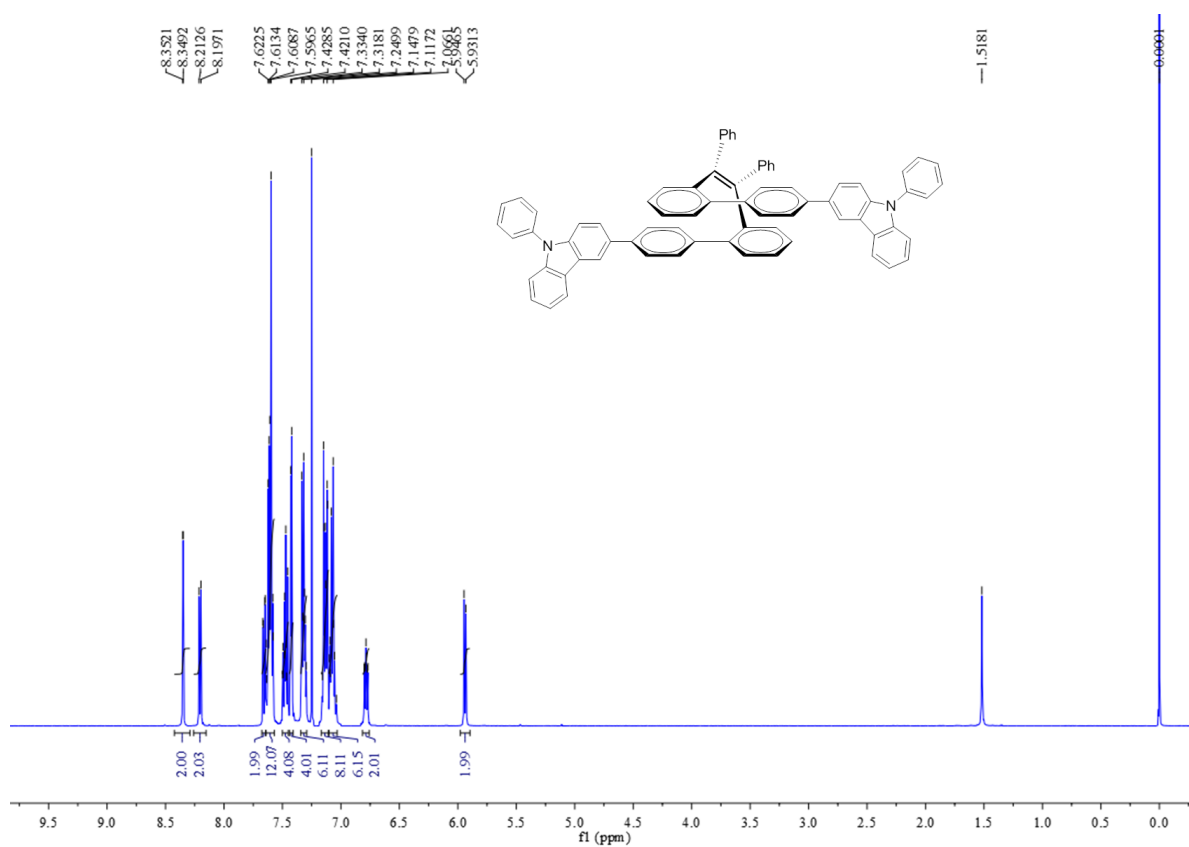


Fig. S14 ^1H NMR spectrum of *f*-TPEC (500 MHz, CDCl_3).

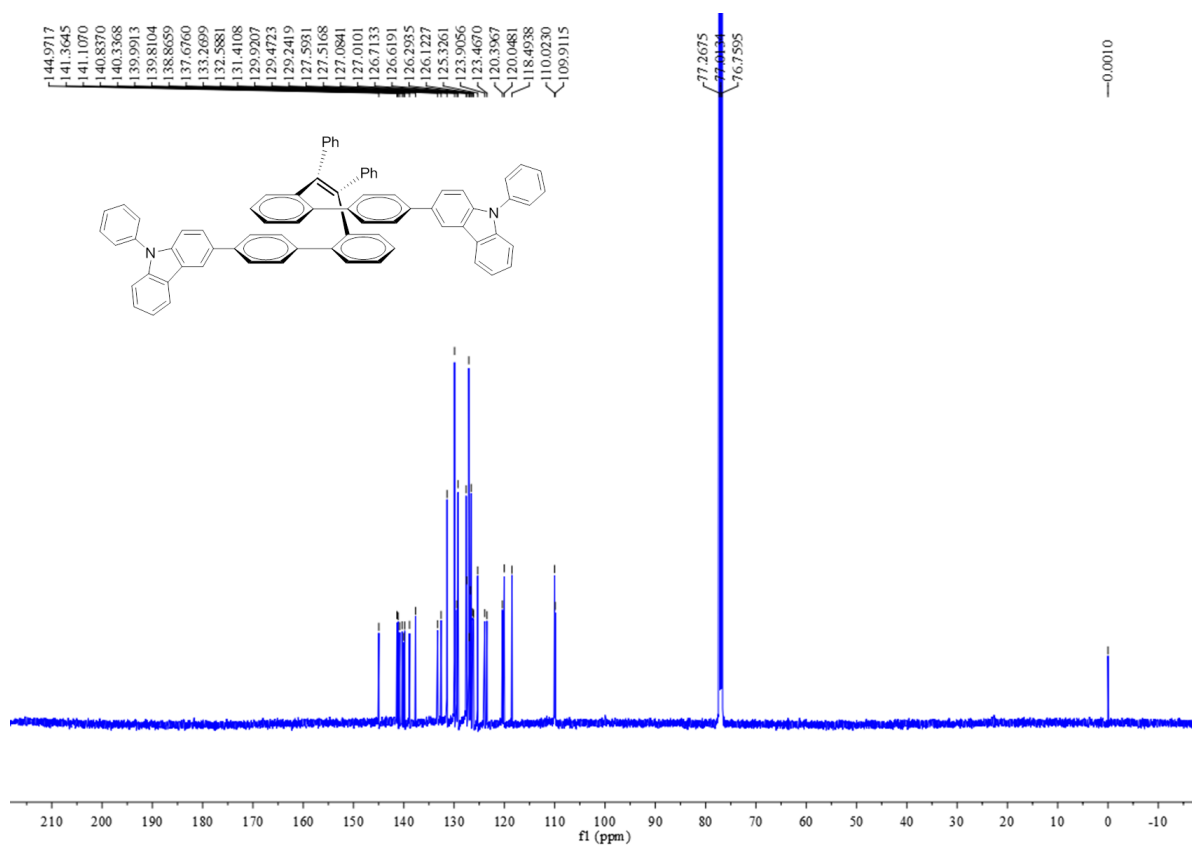


Fig. S15 ¹³C NMR spectrum of *f*-TPEC (125 MHz, CDCl₃).

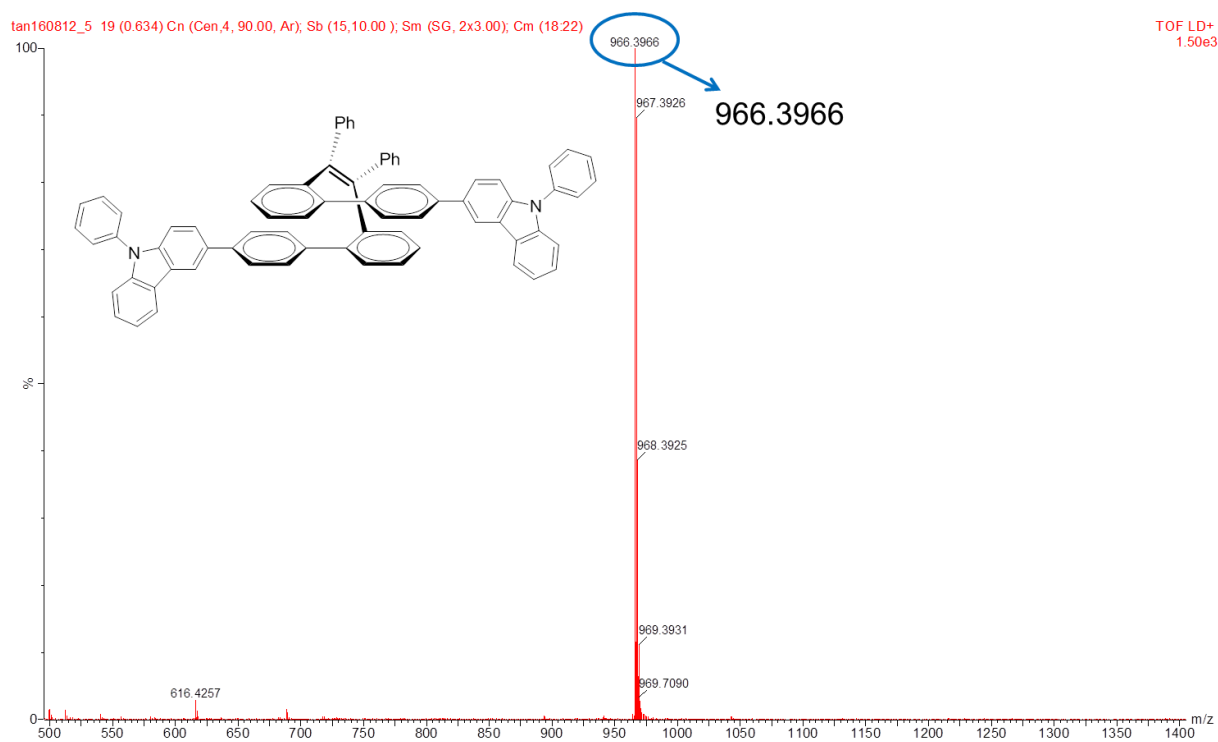


Fig. S16 HRMS spectrum of *f*-TPEC.

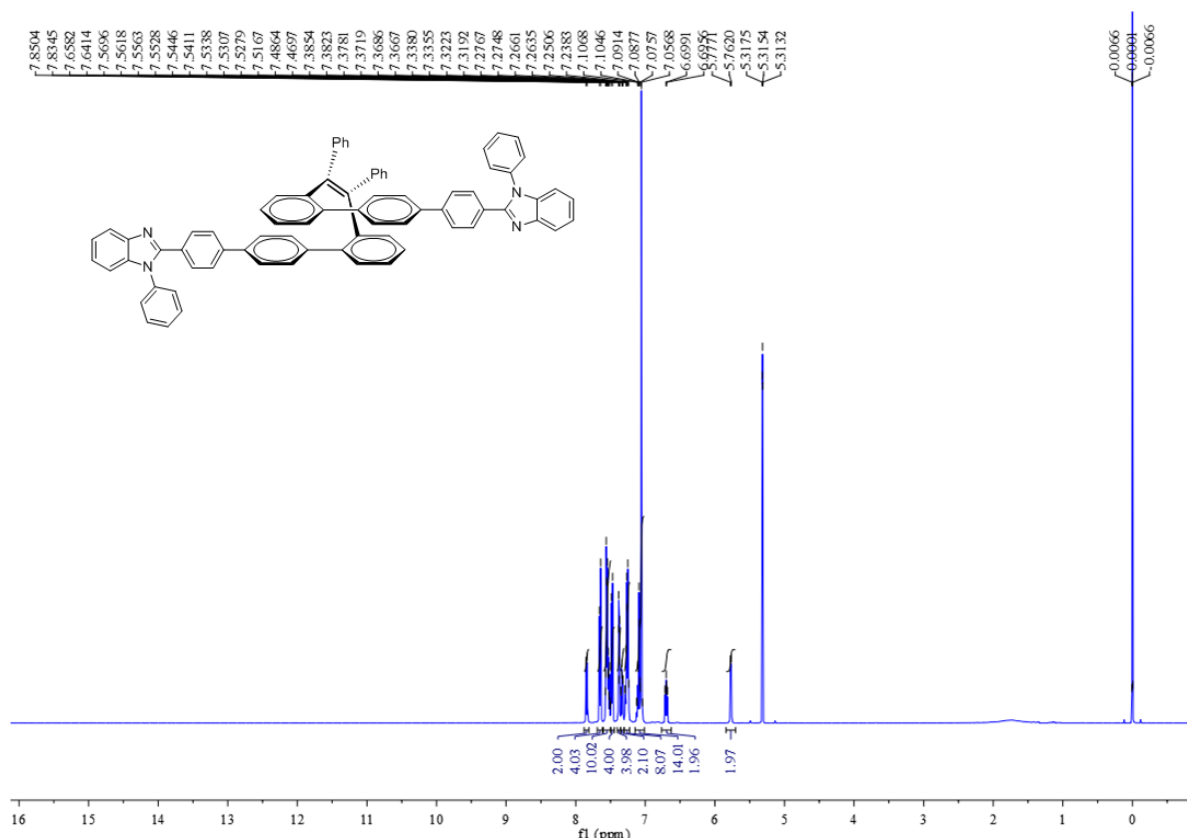


Fig. S17 ¹H NMR spectrum of *f*-TPEI (500 MHz, CD₂Cl₂).

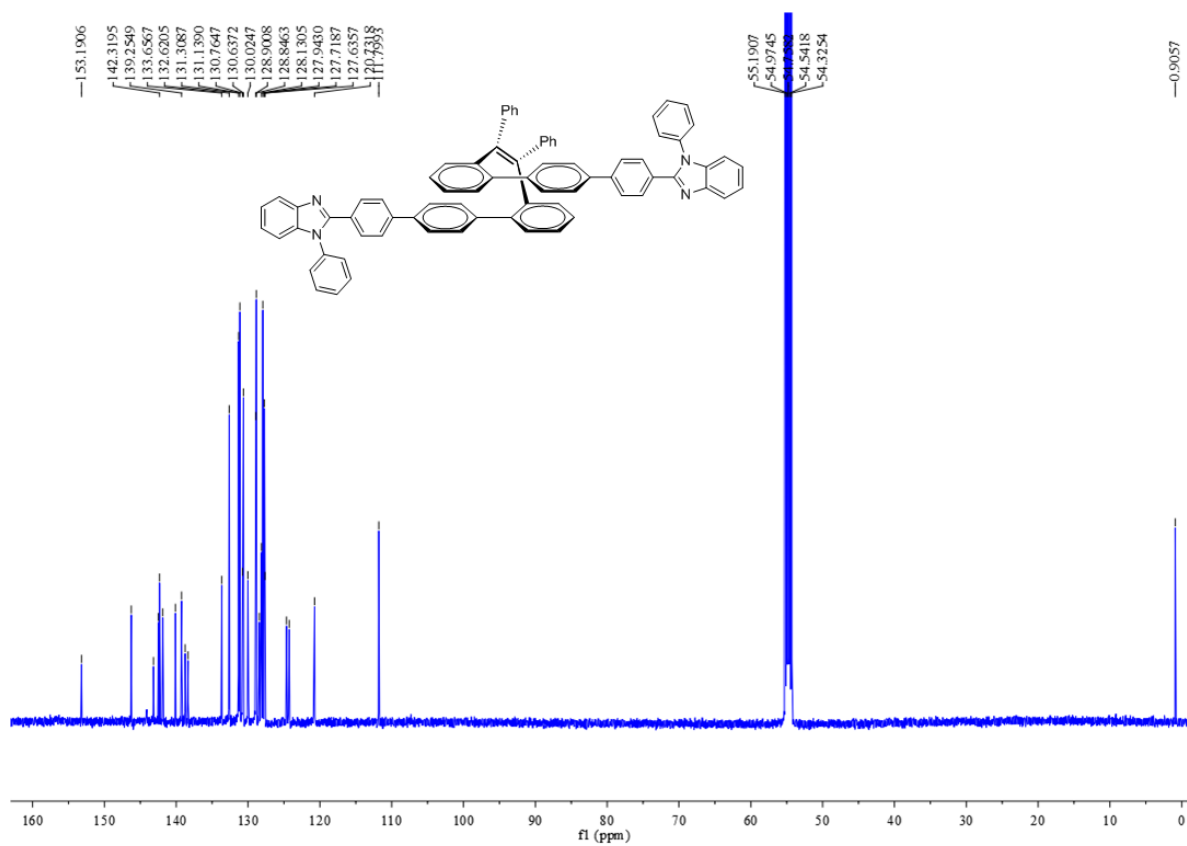


Fig. S18 ¹³C NMR spectrum of *f*-TPEI (125 MHz, CD₂Cl₂).

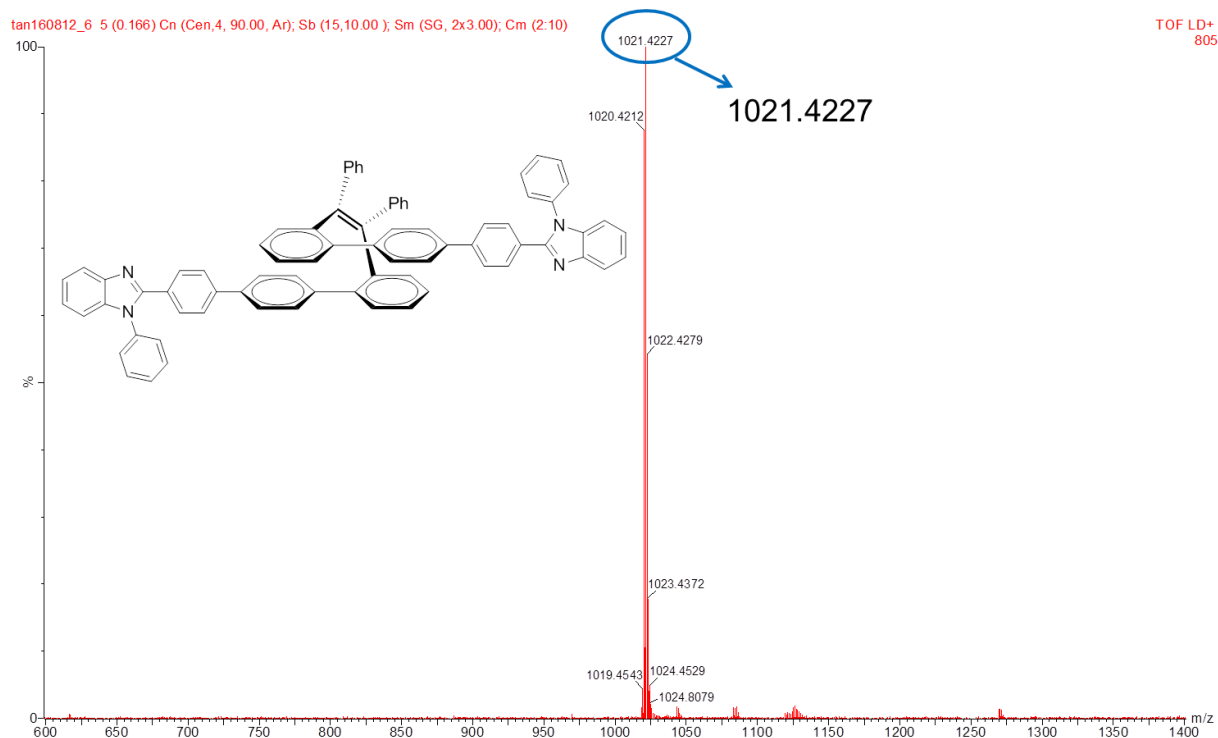


Fig. S19 HRMS spectrum of *f*-TPEI.

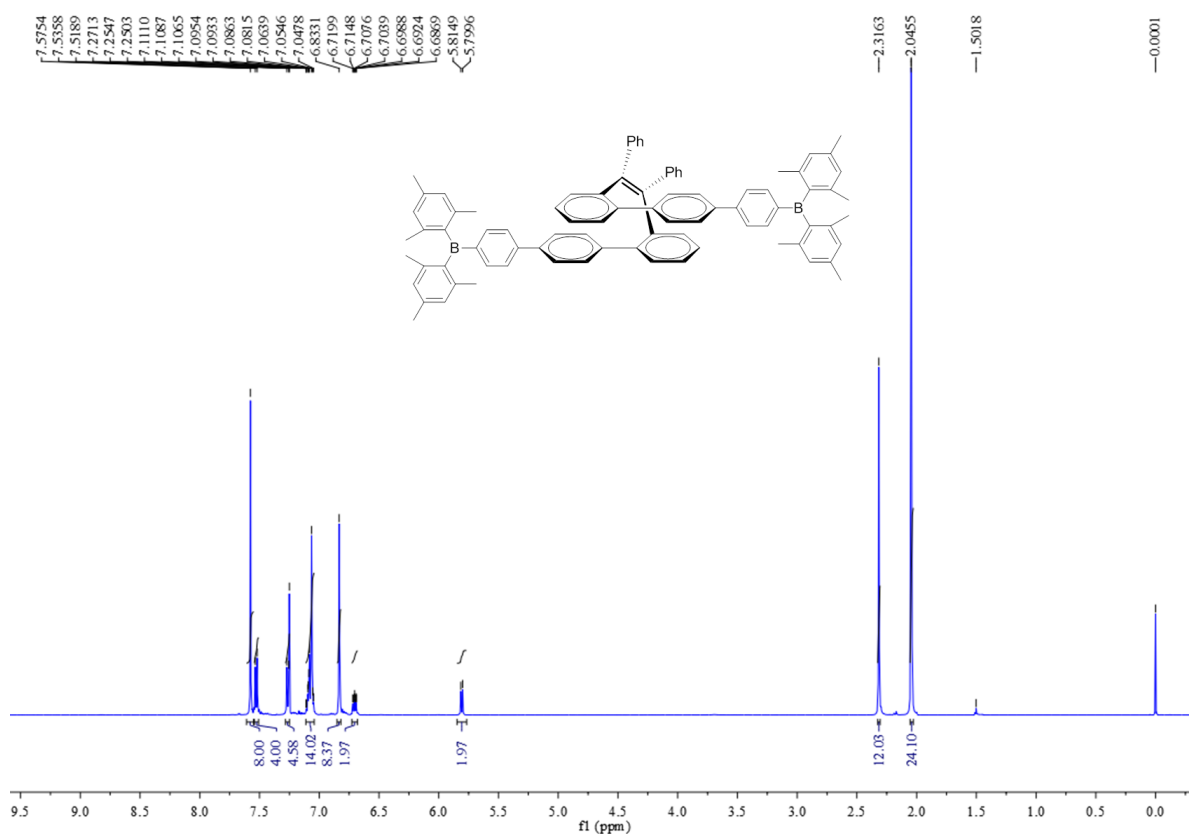


Fig. S20 ^1H NMR spectrum of *f*-TPEB (500 MHz, CDCl_3).

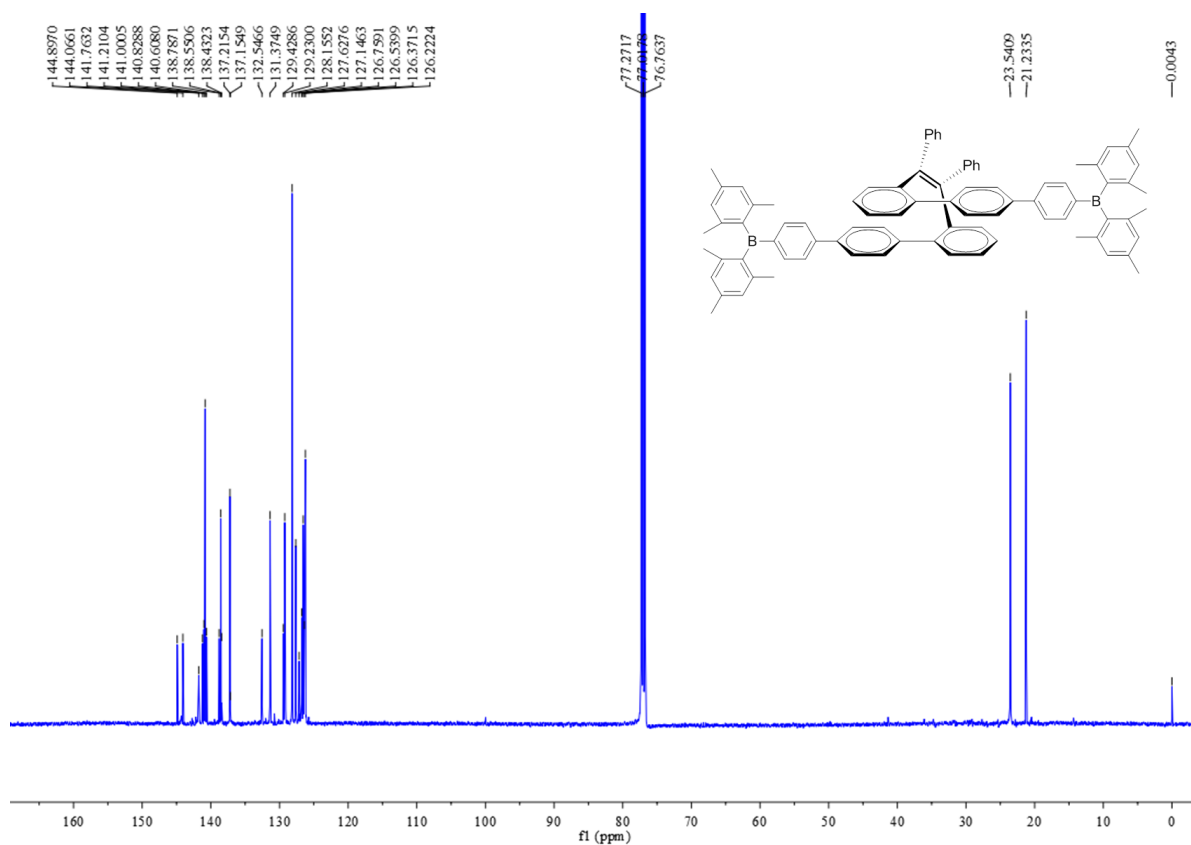


Fig. S21 ^{13}C NMR spectrum of *f*-TPEB (125 MHz, CDCl_3).

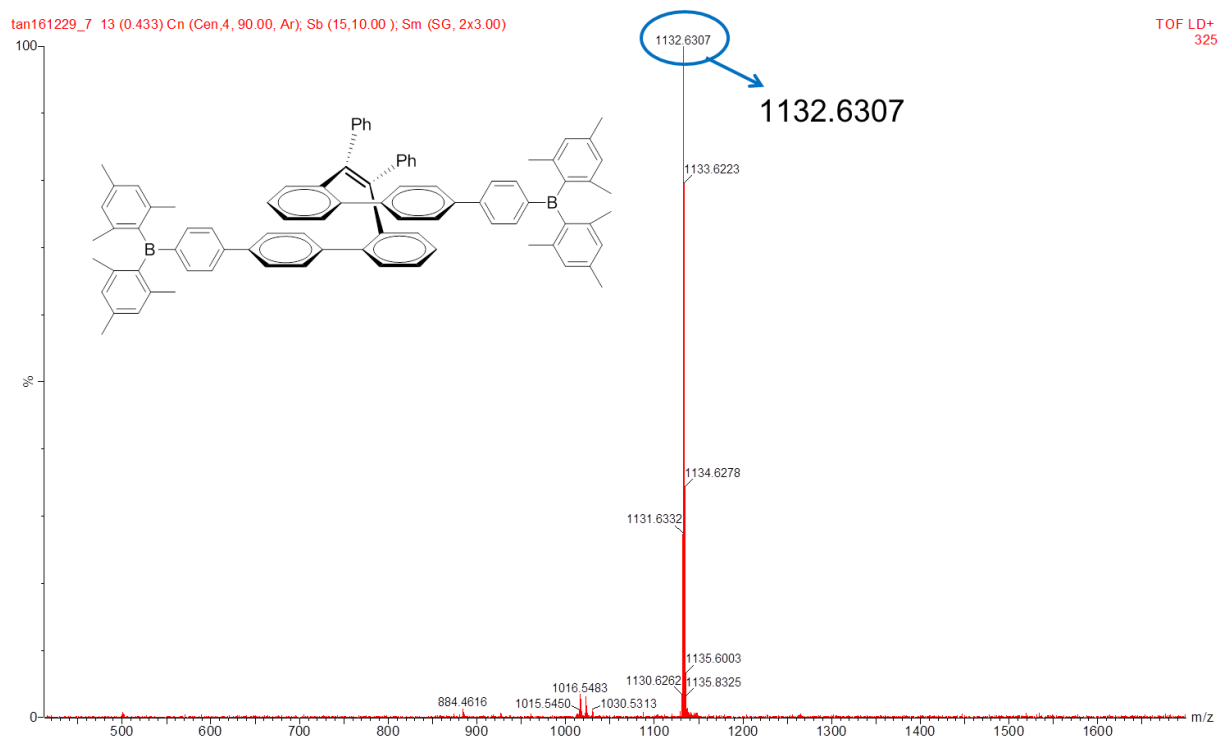


Fig. S22 HRMS spectrum of *f*-TPEB.

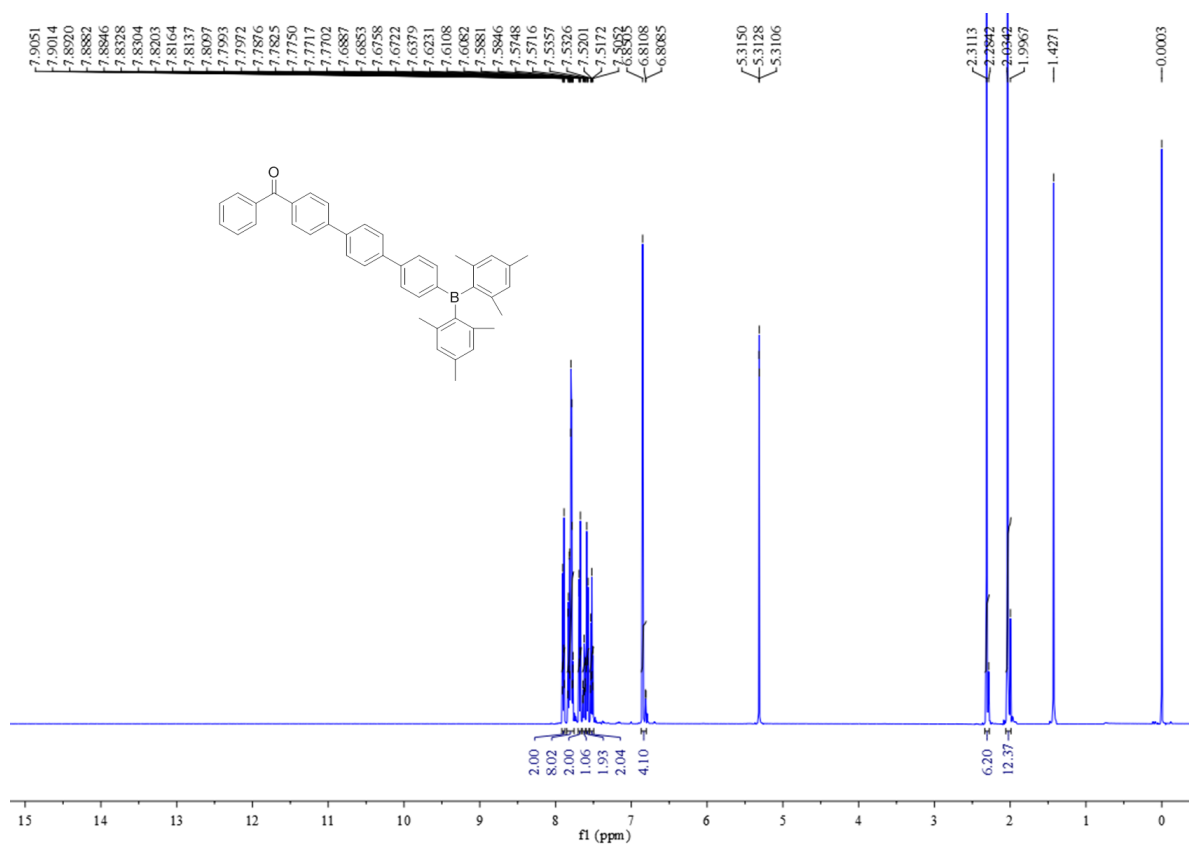


Fig. S23 ¹H NMR spectrum of compound **14** (500 MHz, CD₂Cl₂).

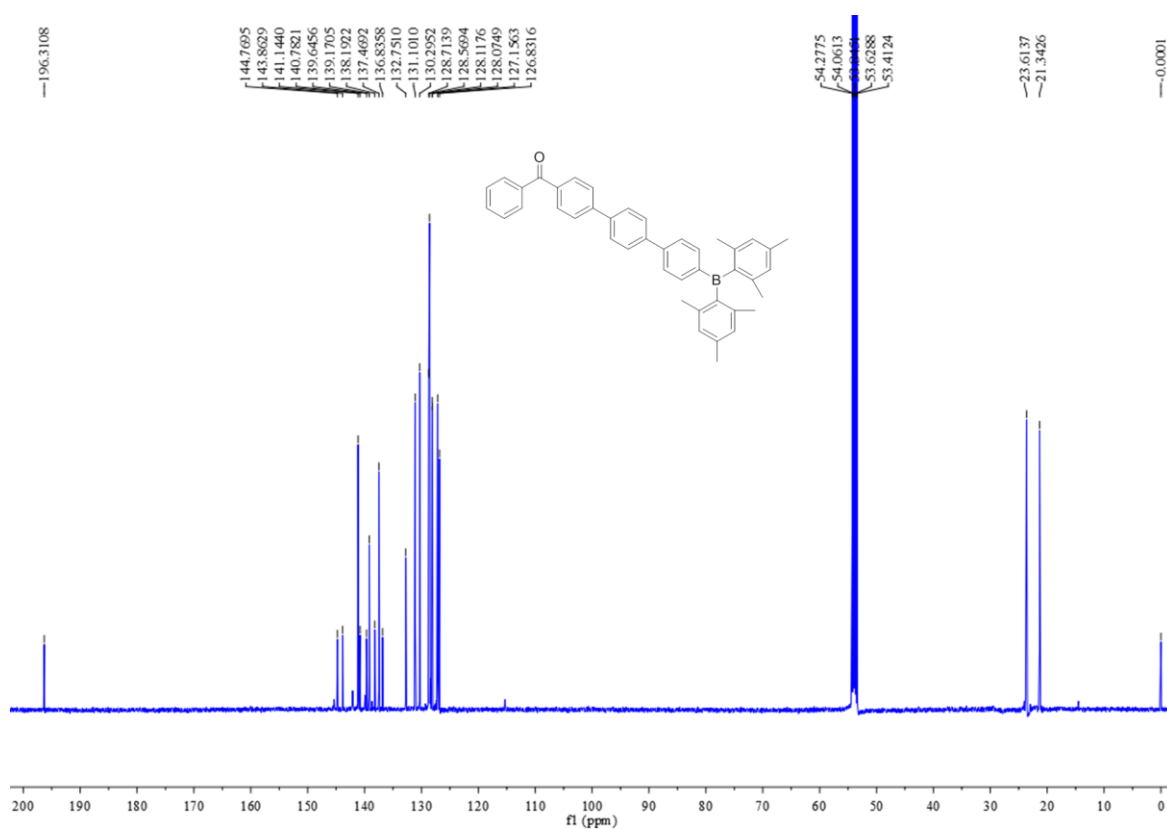
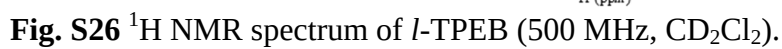


Fig. S24 ¹³C NMR spectrum of compound **14** (125 MHz, CD₂Cl₂).



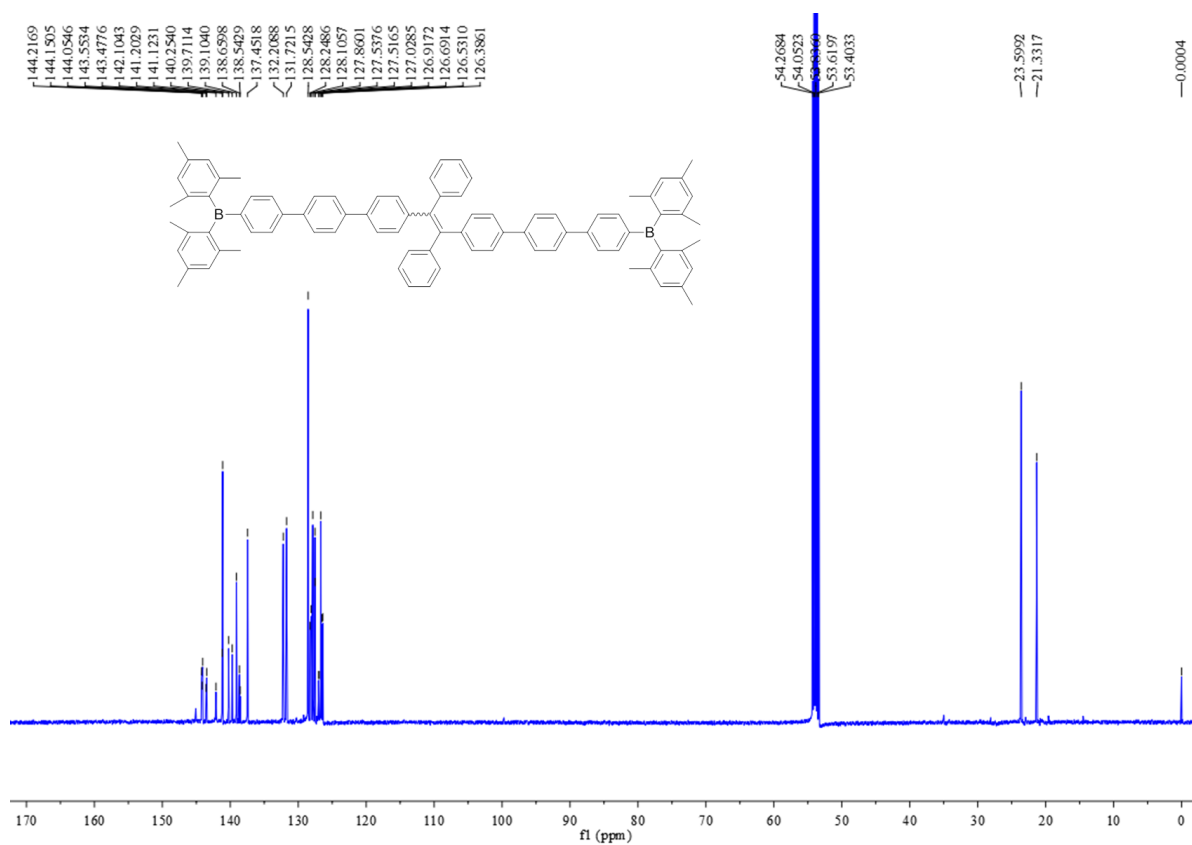


Fig. S27 ^{13}C NMR spectrum of *l*-TPEB (125 MHz, CD_2Cl_2).

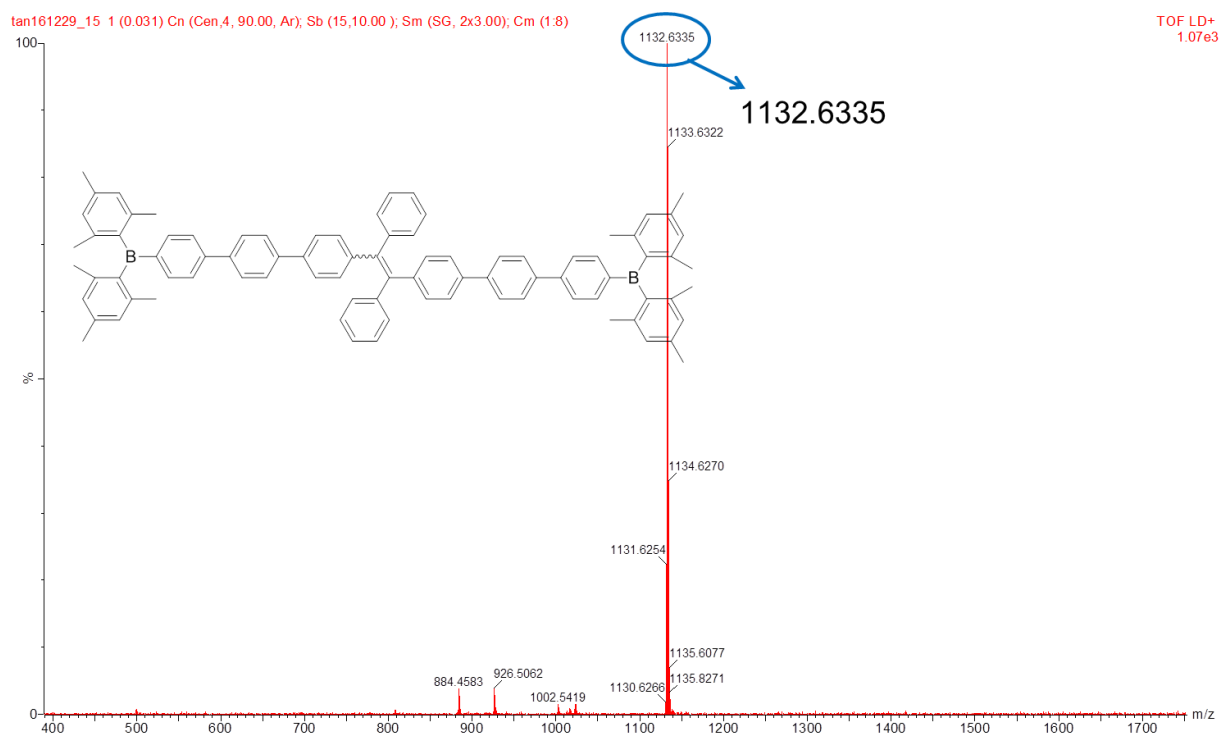


Fig. S28 HRMS spectrum of *l*-TPEB.