molecules

MDPI

*Supplementary Materials*

# Reaction of Aldoximes with Sodium Chloride and Oxone under Ball-Milling Conditions

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## 1. $^{1}H$ and $^{13}C$ NMR spectra of 2 and 3a

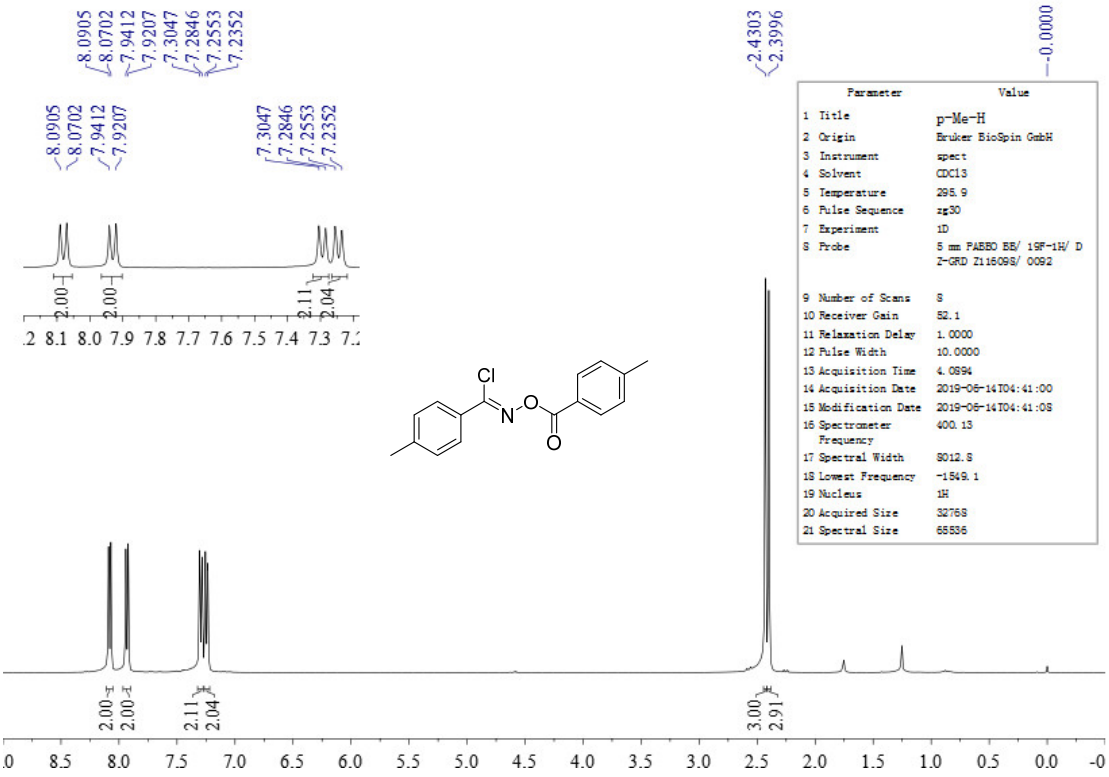


**Figure S1.** $^{1}H$ NMR (400 MHz, $CDCl_3$) spectrum of compound 2a.

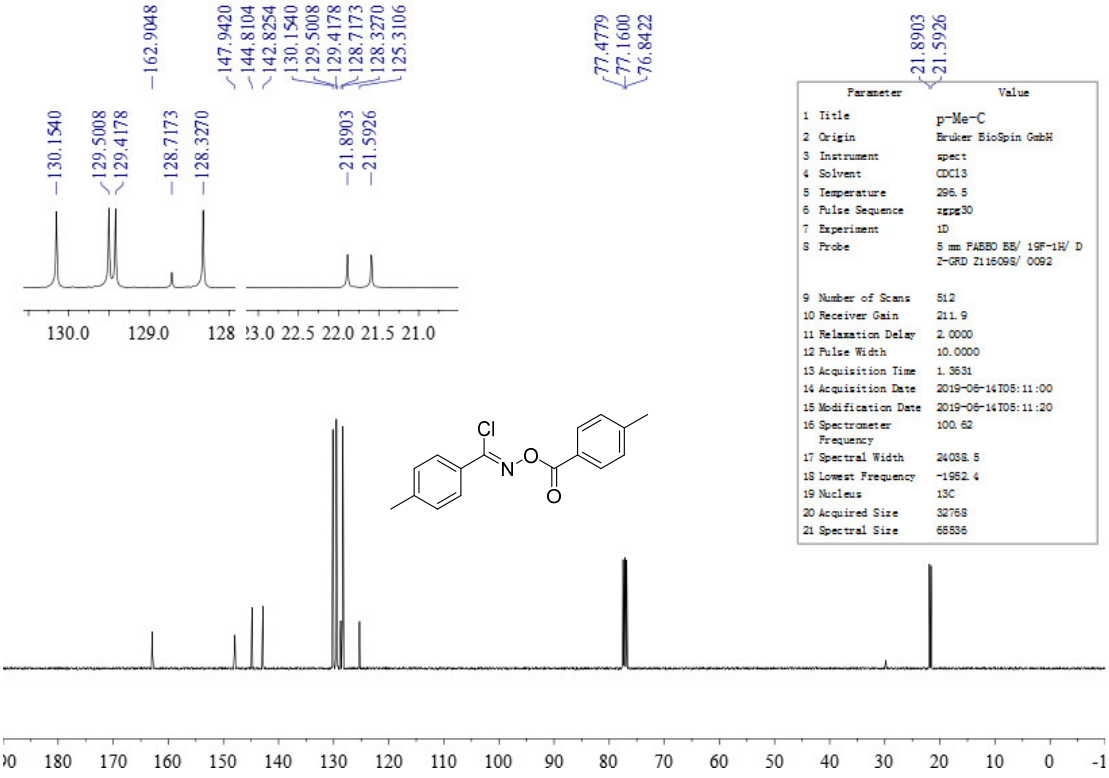


**Figure S2.** $^{13}C$ NMR (101 MHz, $CDCl_3$) spectrum of compound 2a.

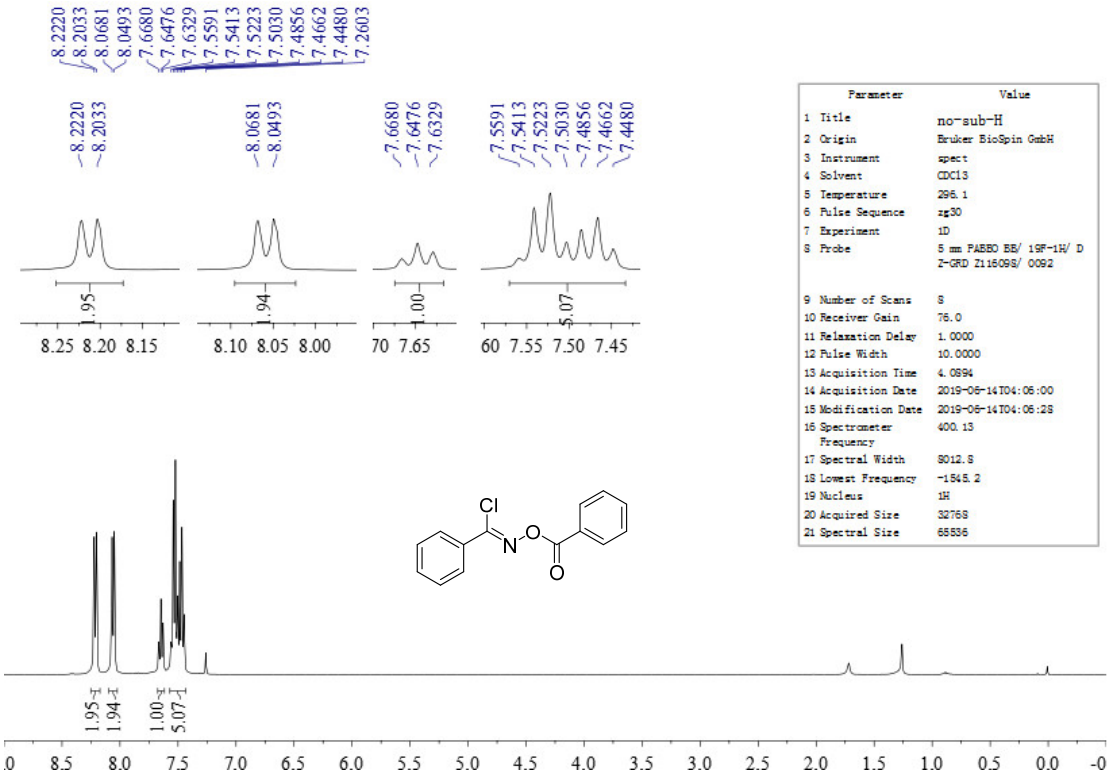


**Figure S3.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2b.

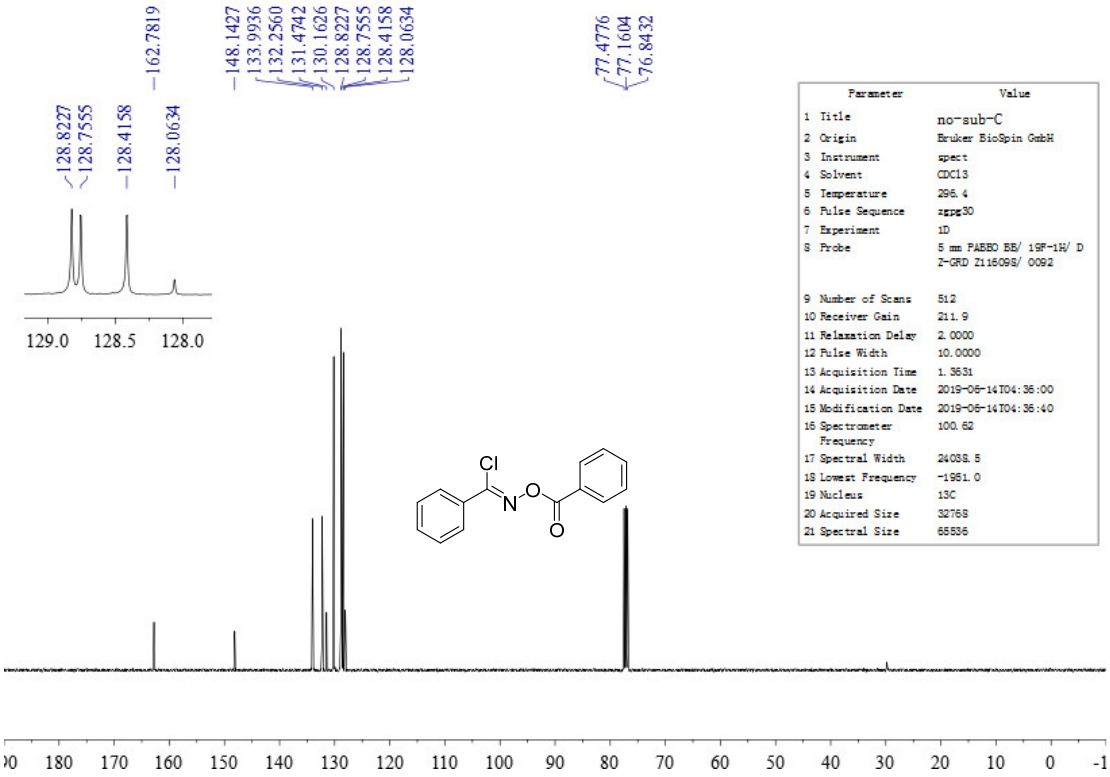


**Figure S4.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2b.

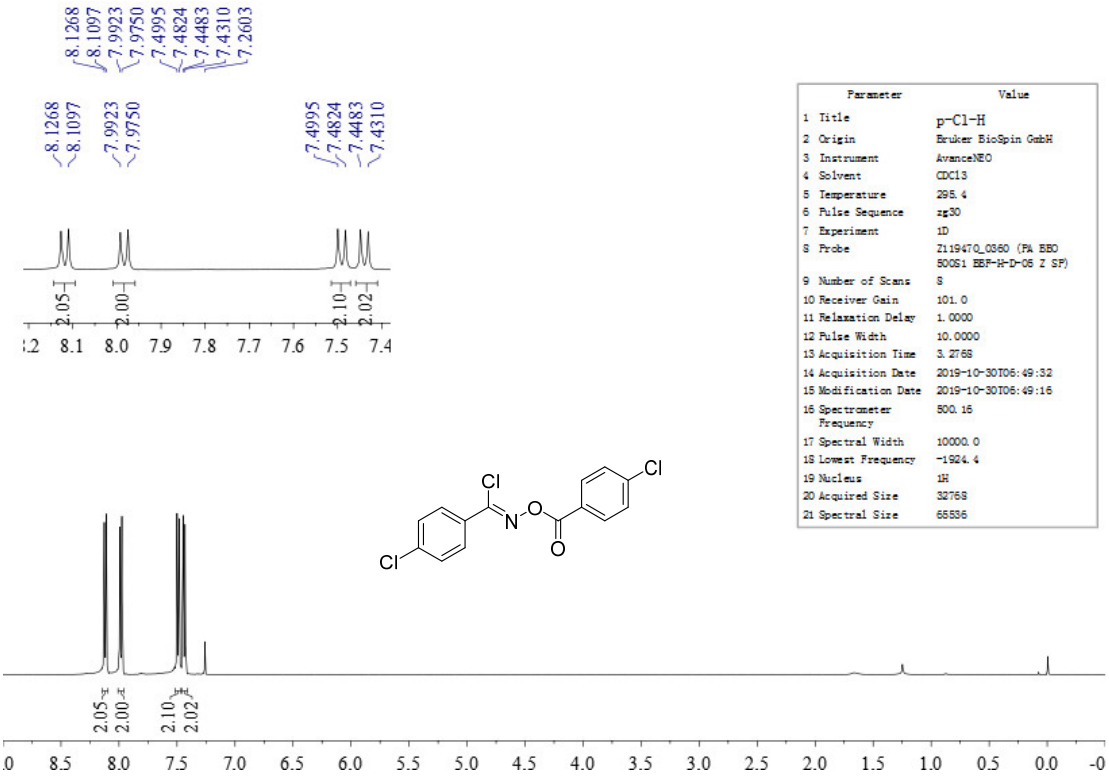


**Figure S5.** $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 2c.

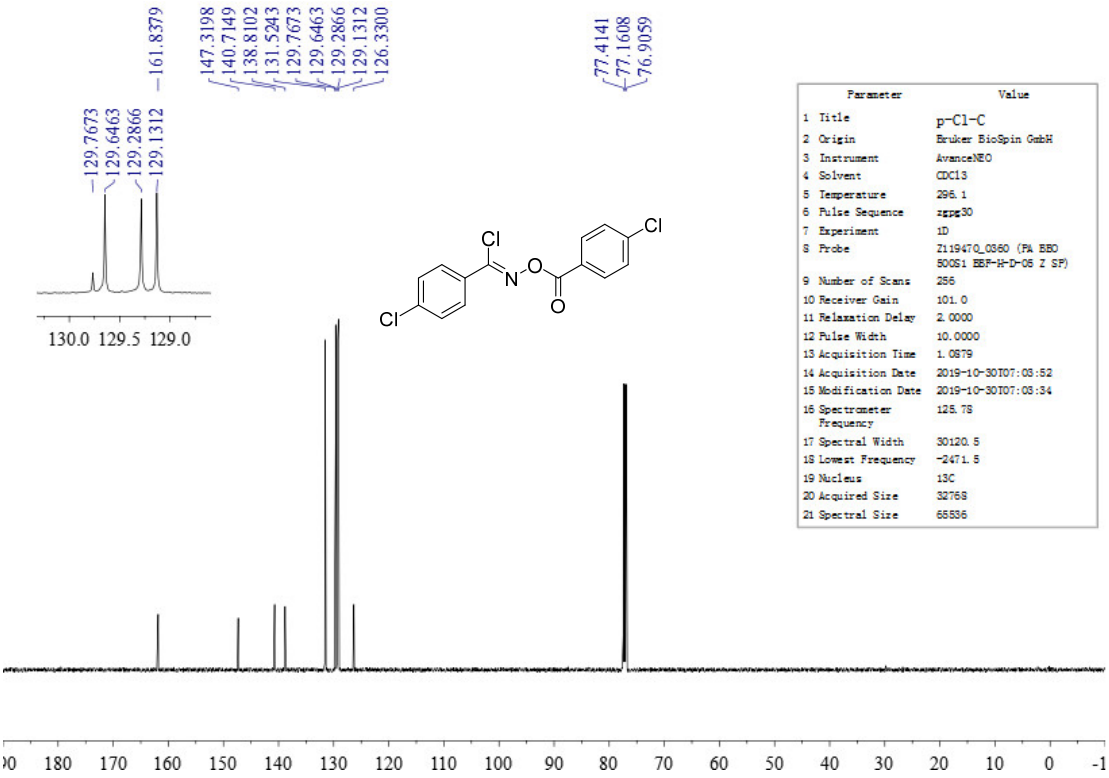


**Figure S6.** $^{13}$C NMR (126 MHz, $CDCl_3$) spectrum of compound 2c.

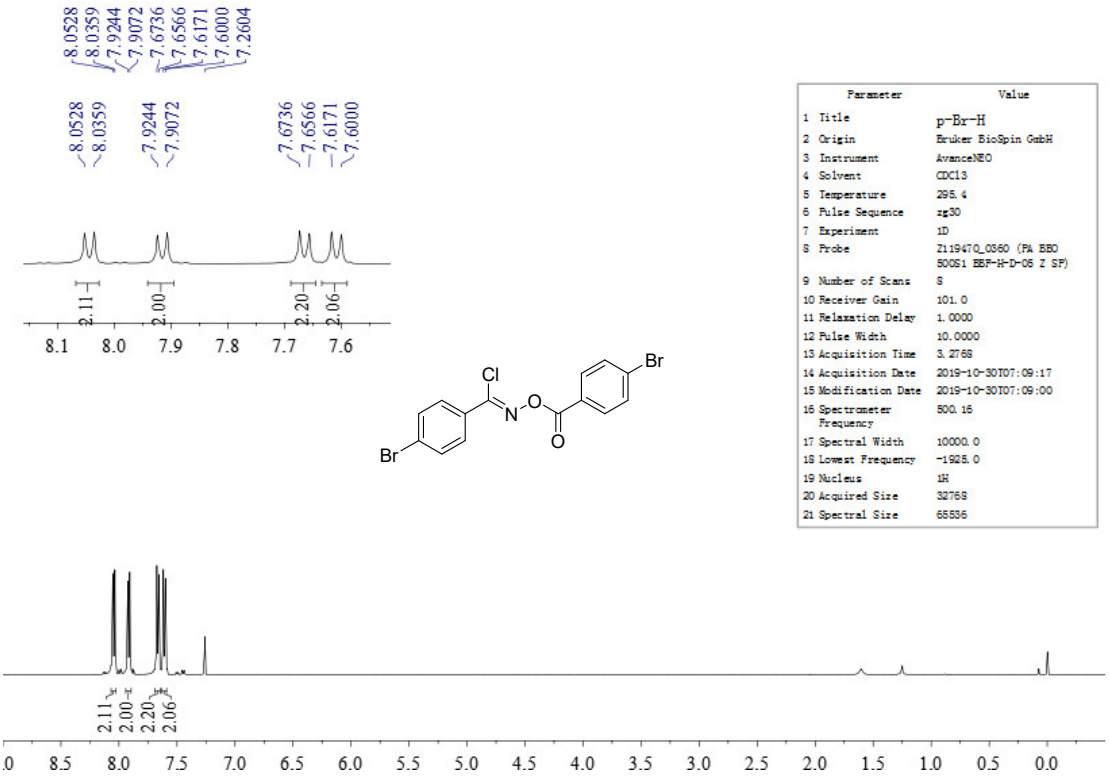


**Figure S7.** $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 2d.

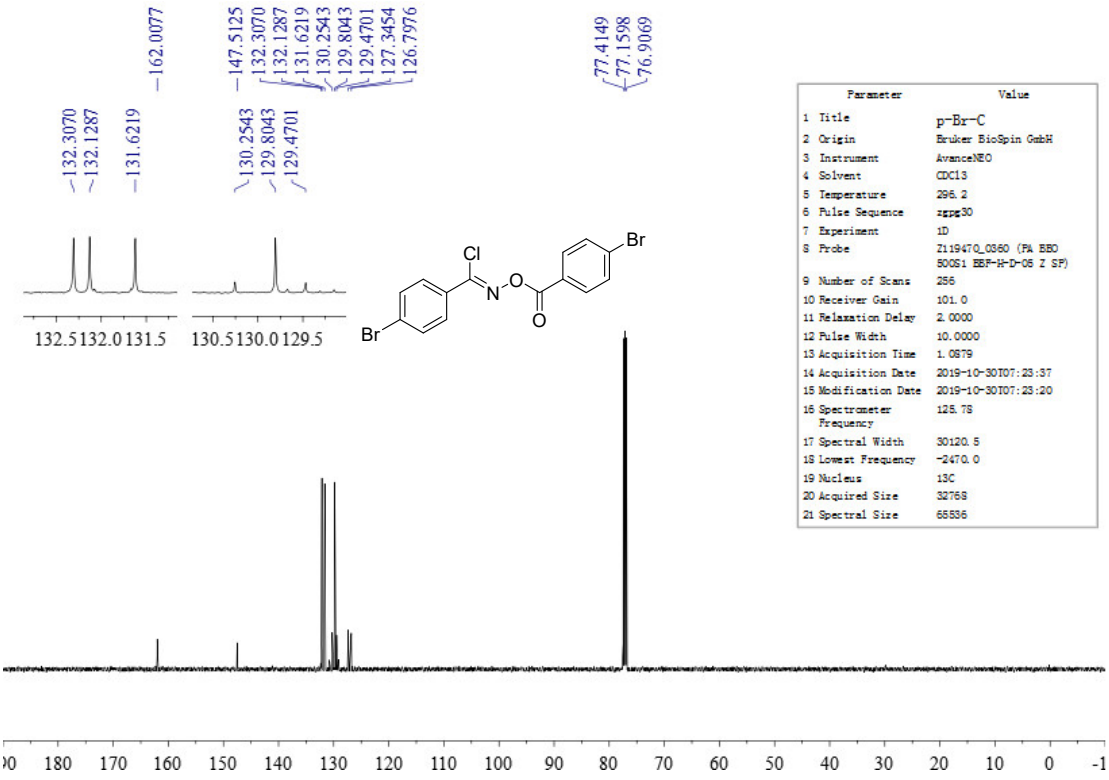


**Figure S8.** $^{13}$C NMR (126 MHz, $CDCl_3$) spectrum of compound 2d.

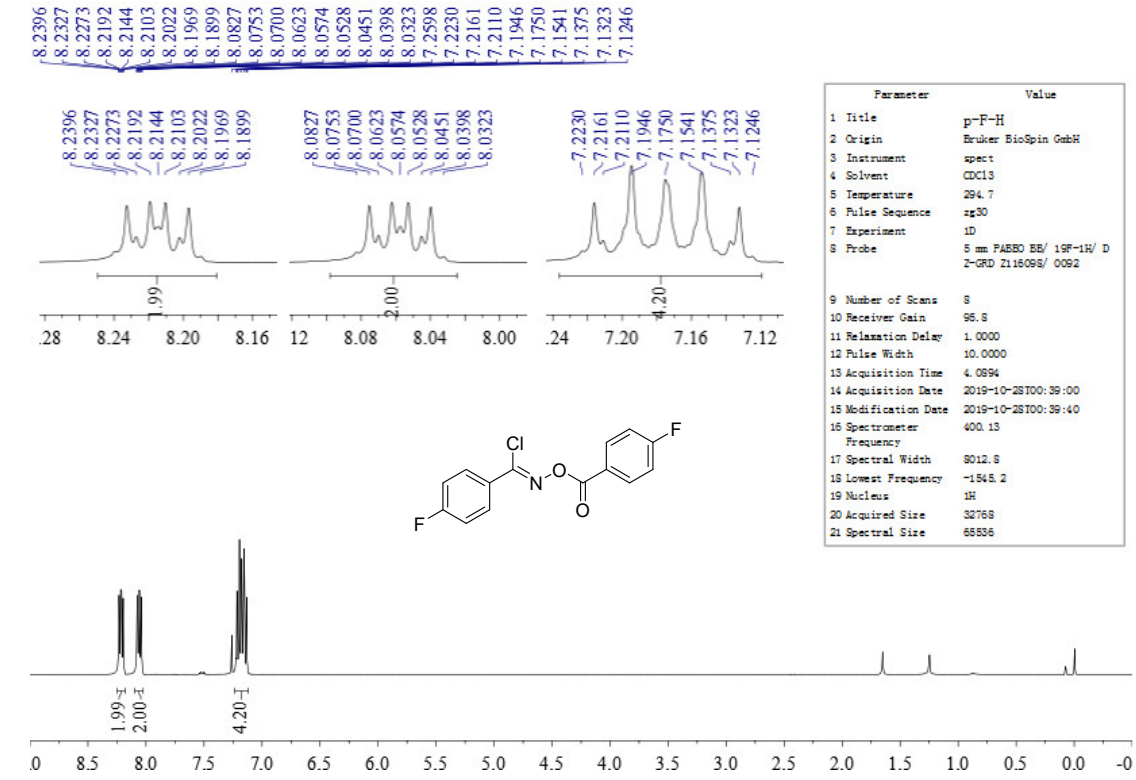


**Figure S9.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2e.

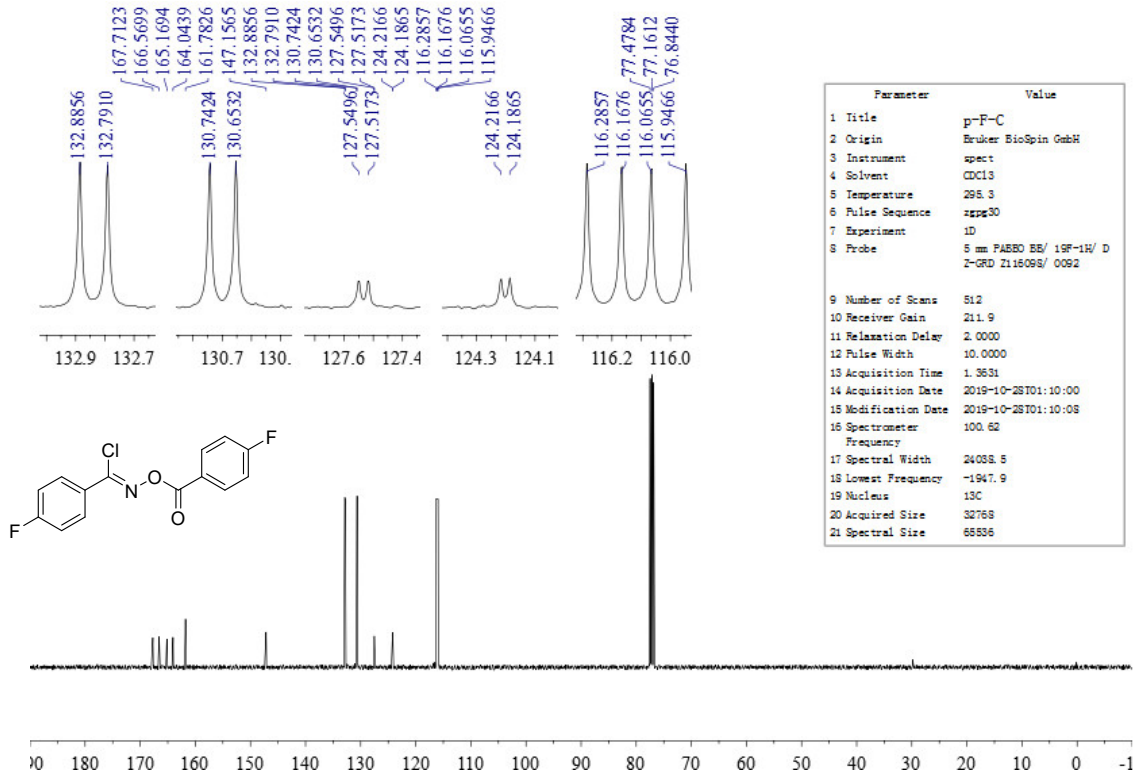


**Figure S10.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2e.

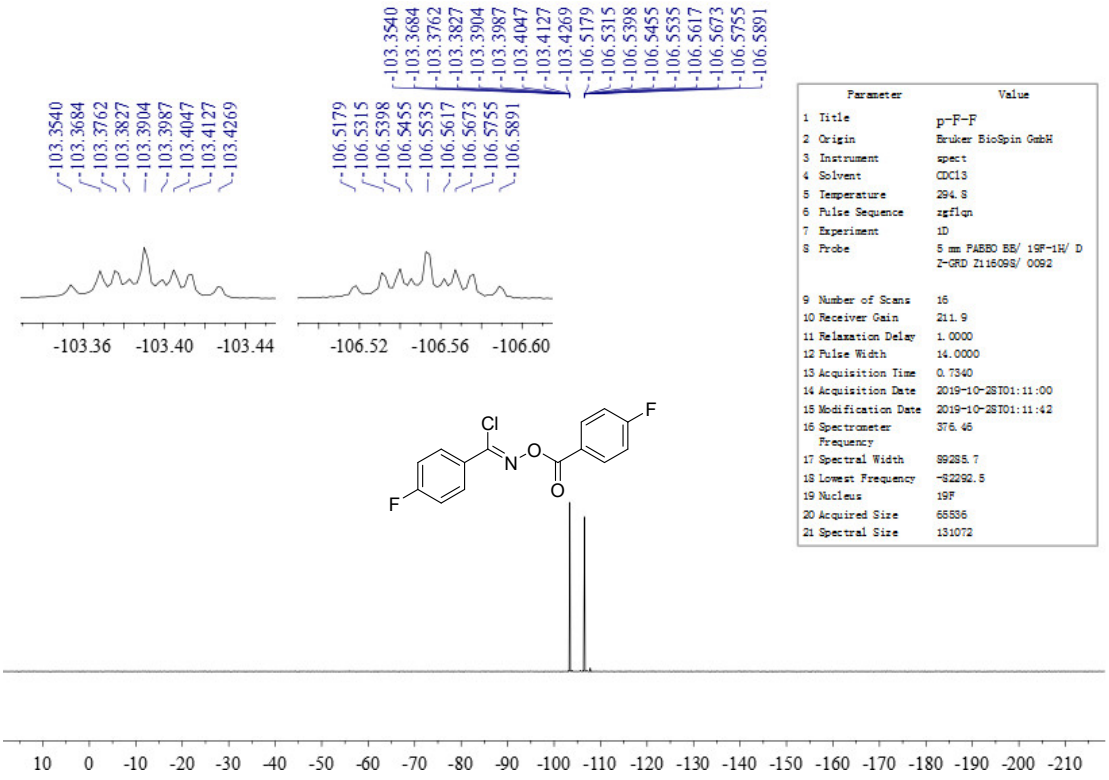


**Figure S11.** $^{19}$F NMR (376 MHz, $CDCl_3$) spectrum of compound 2e.

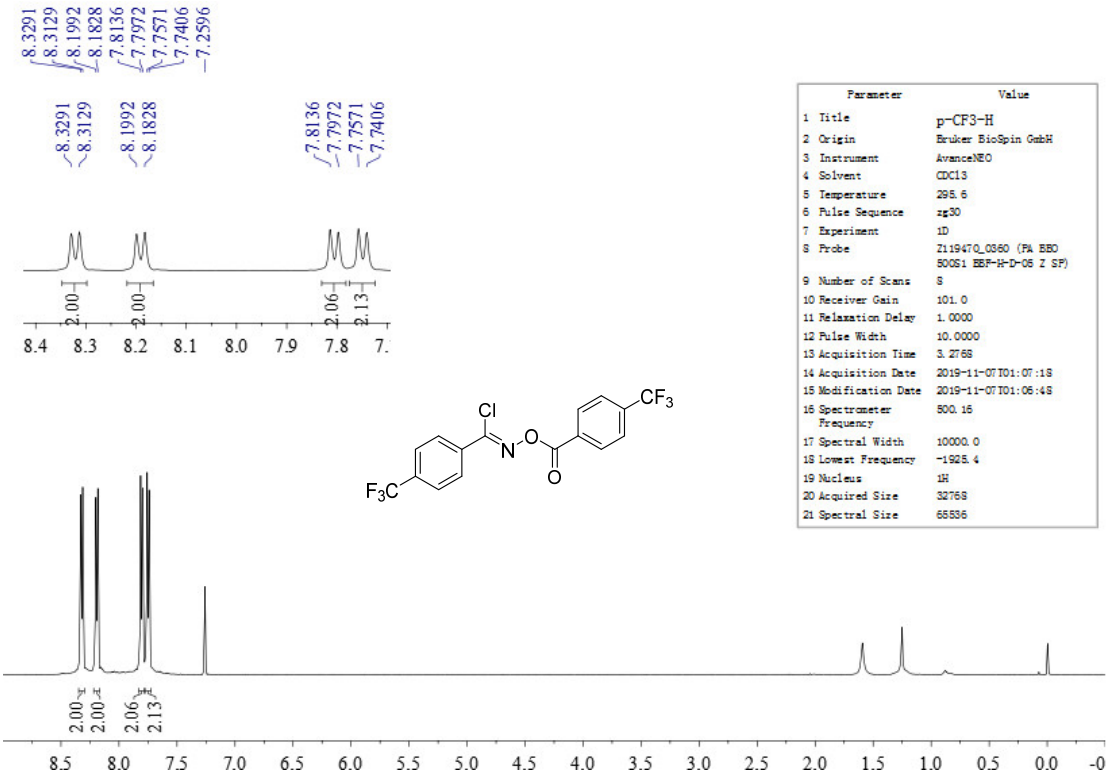


**Figure S12.** $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 2f.

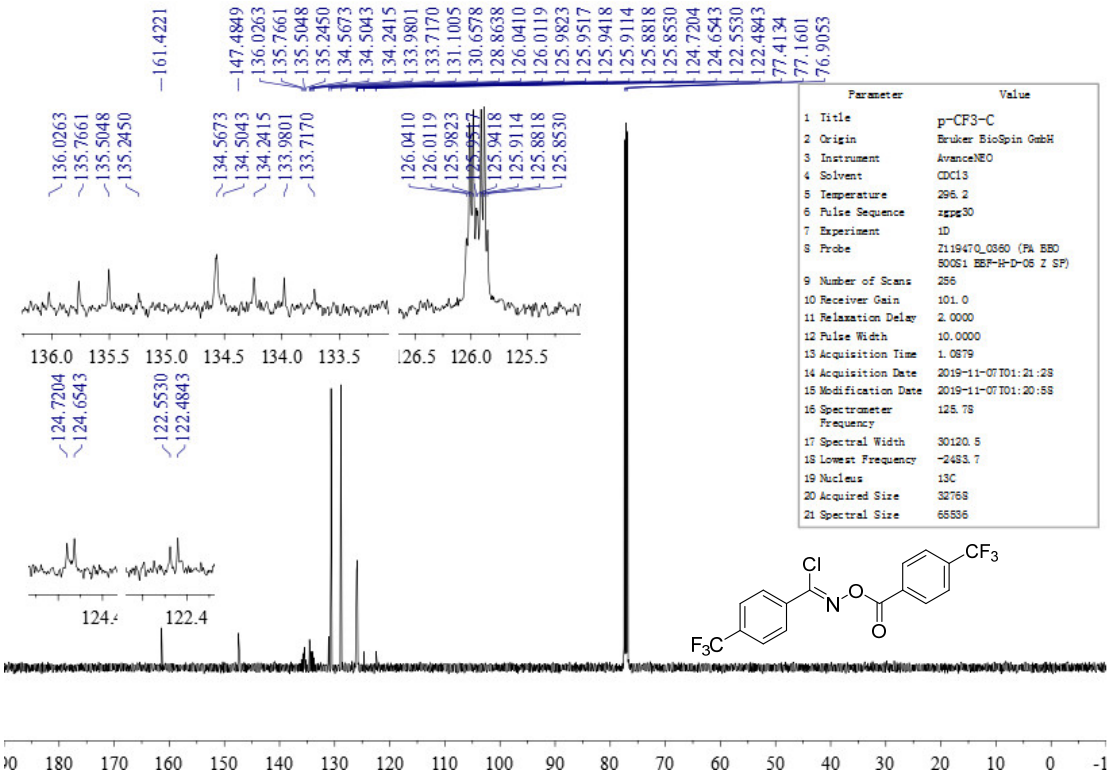


**Figure S13.** $^{13}C$ NMR (126 MHz, $CDCl_3$) spectrum of compound 2f.

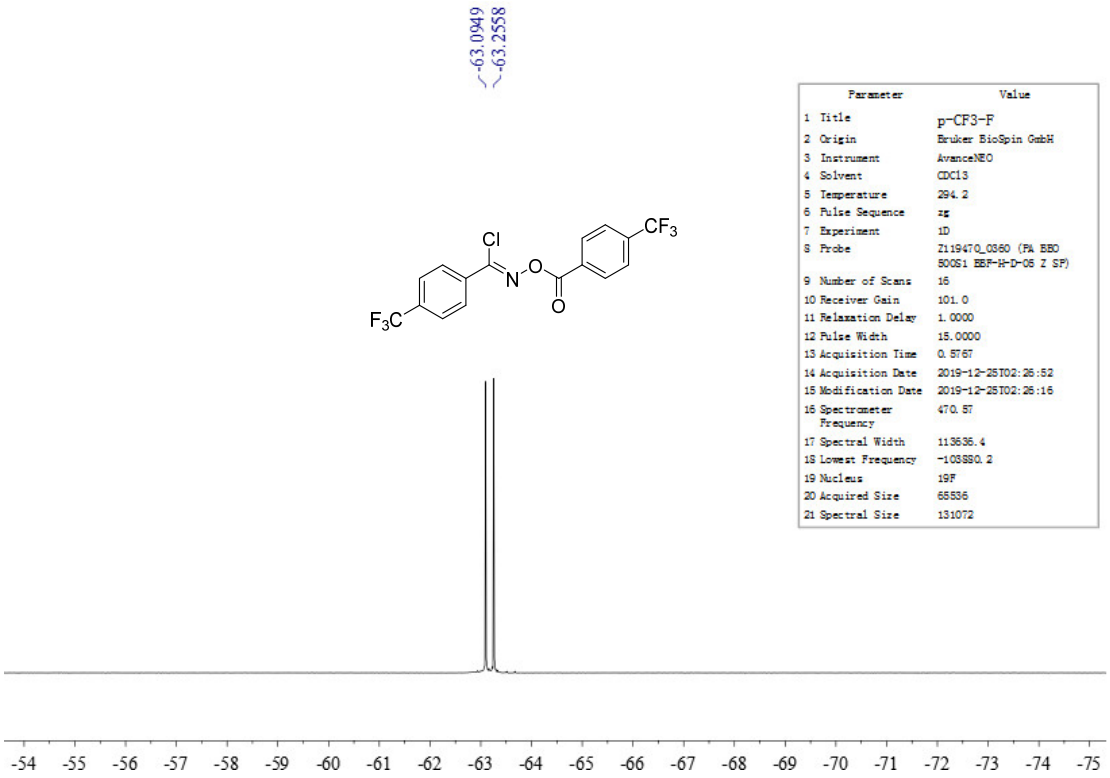


**Figure S14.** $^{19}F$ NMR (471 MHz, $CDCl_3$) spectrum of compound 2f.

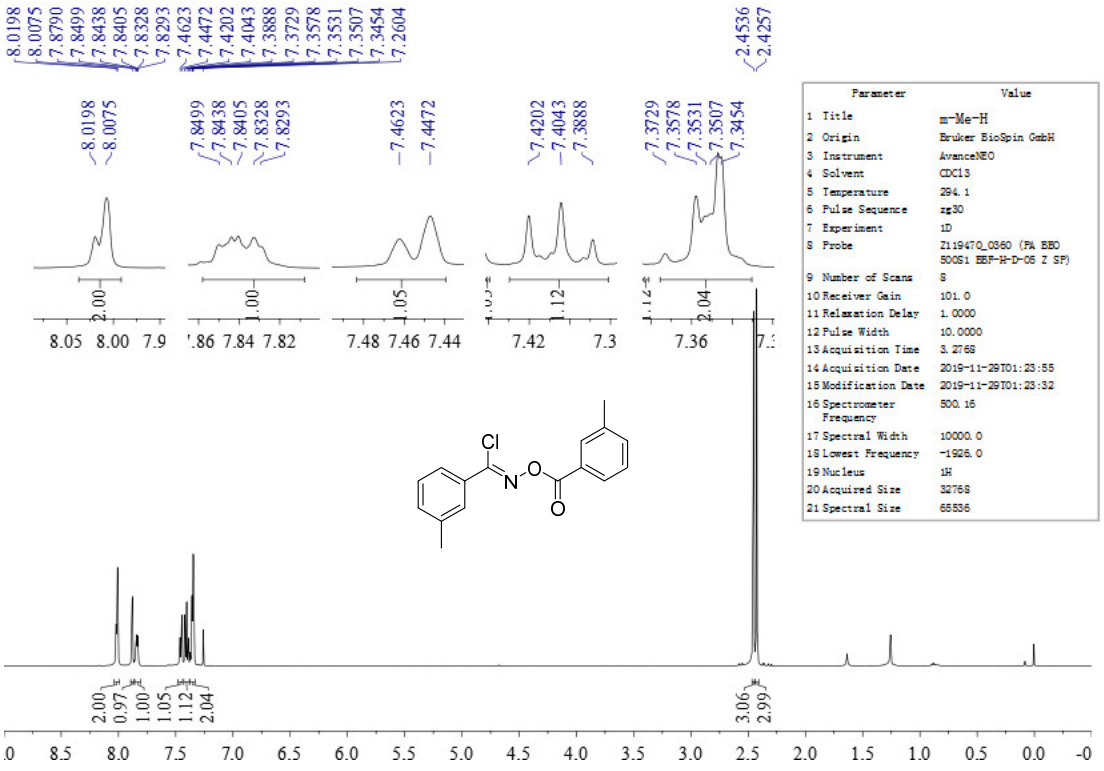


**Figure S15.** $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 2g.

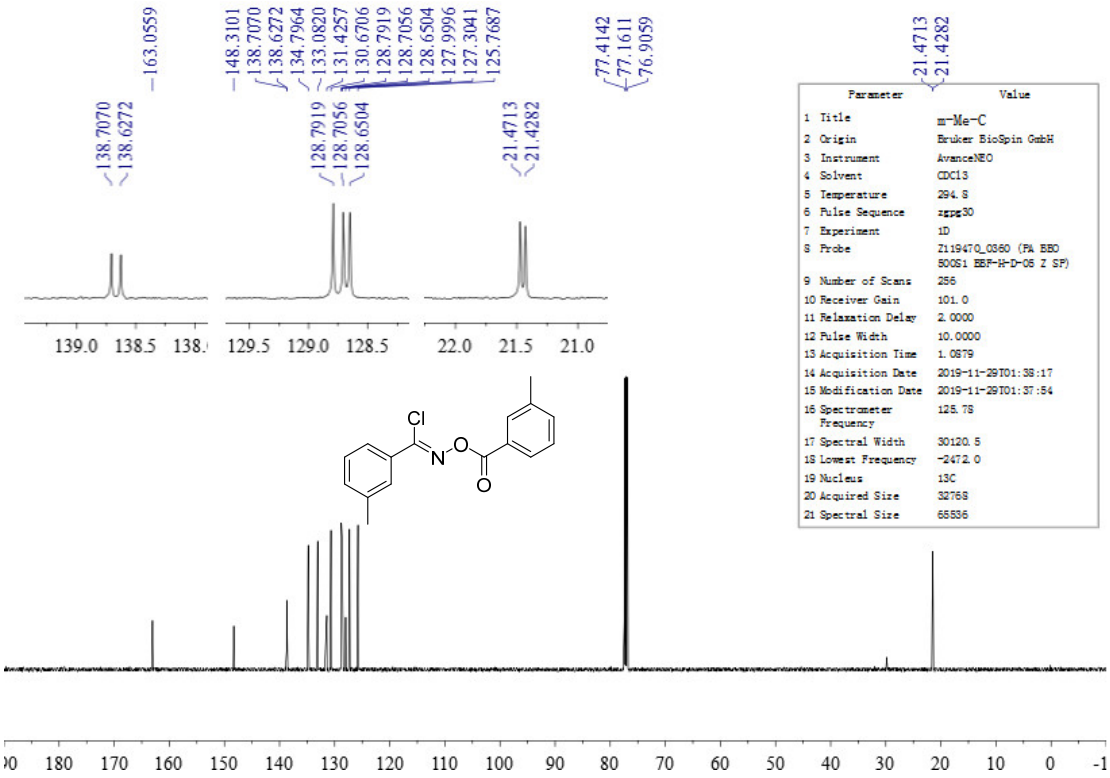


**Figure S16.** $^{13}$C NMR (126 MHz, $CDCl_3$) spectrum of compound 2g.

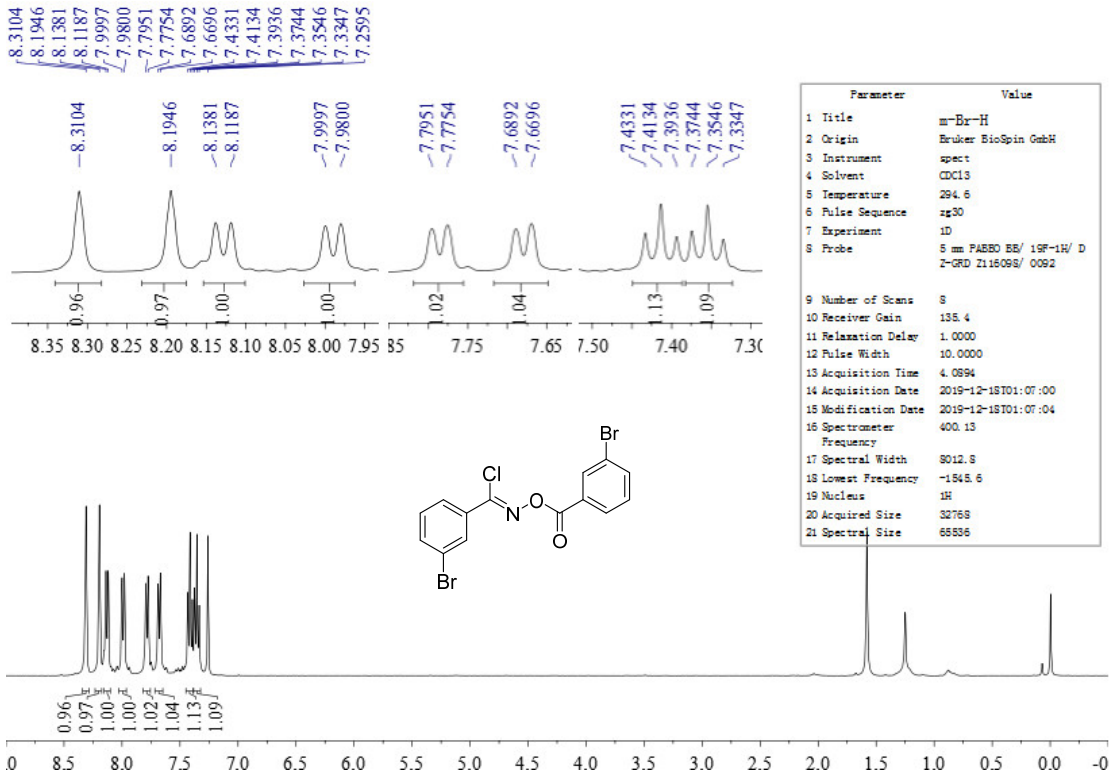


**Figure S17.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2h.

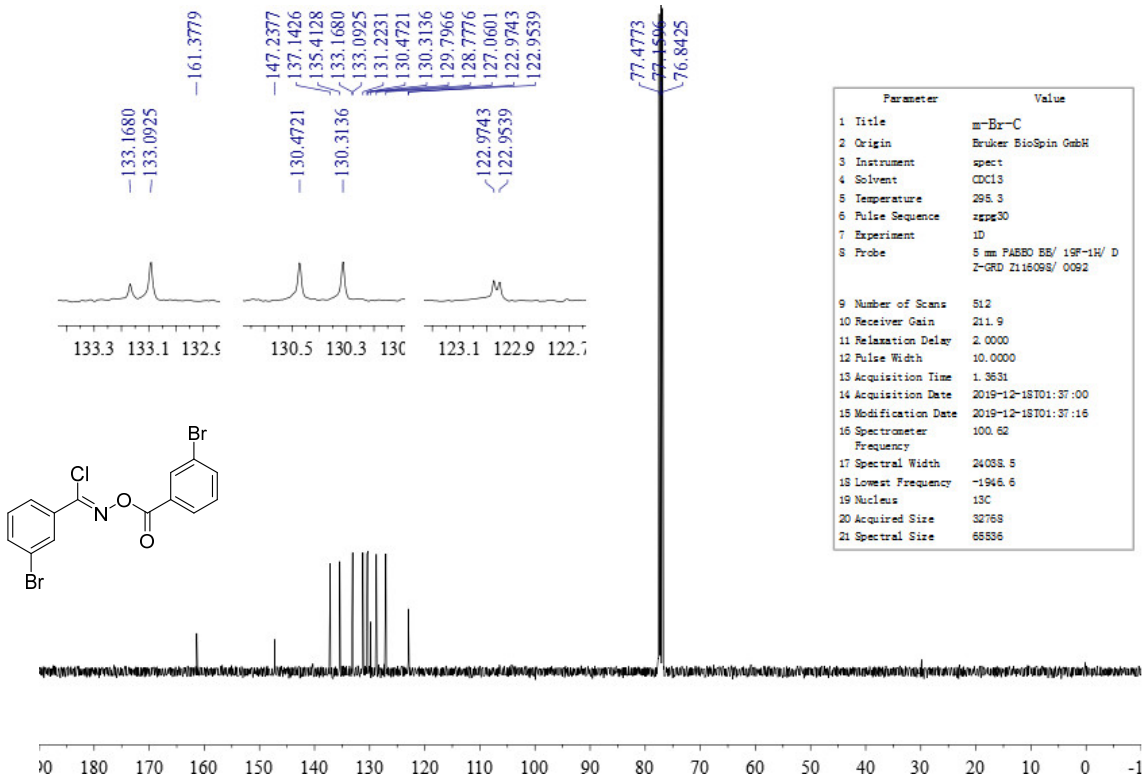


**Figure S18.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2h.

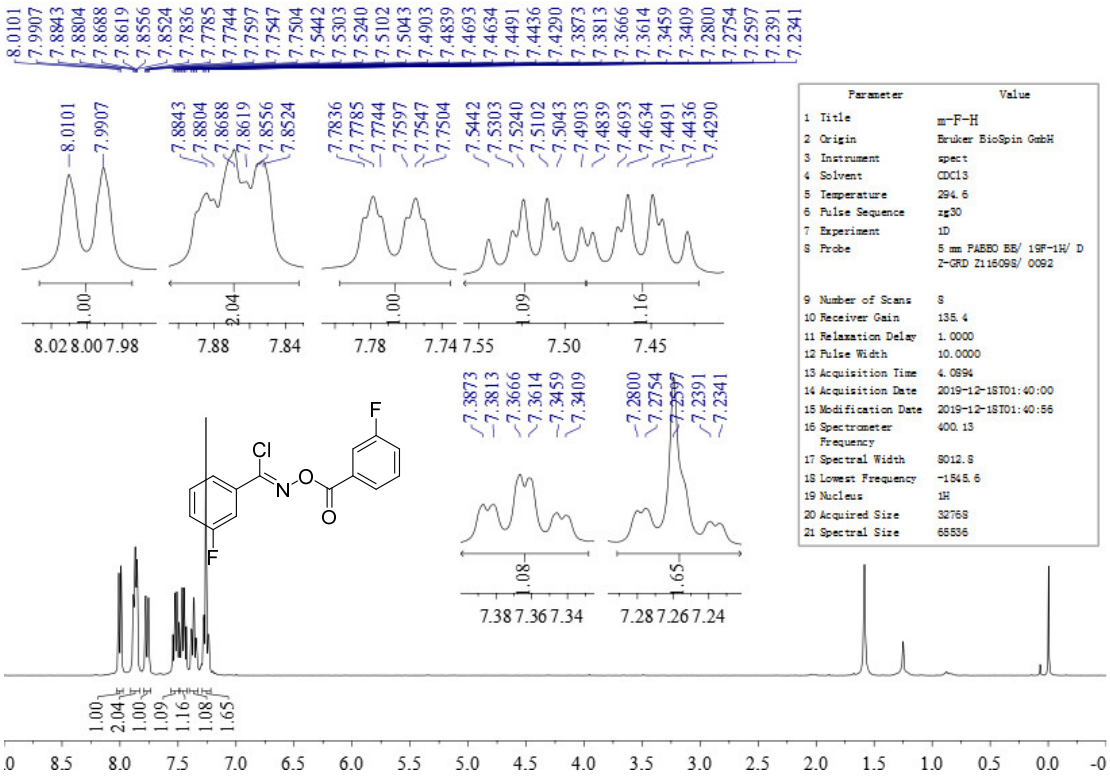


**Figure S19.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2i.

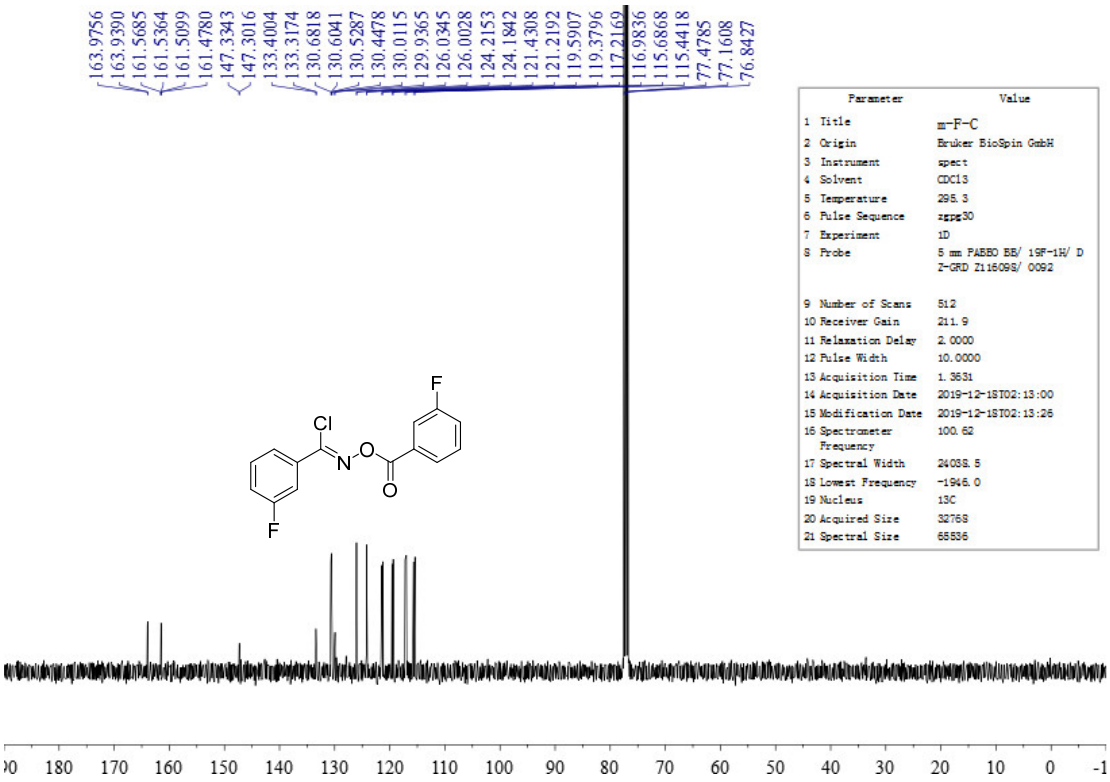


**Figure S20.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2i.

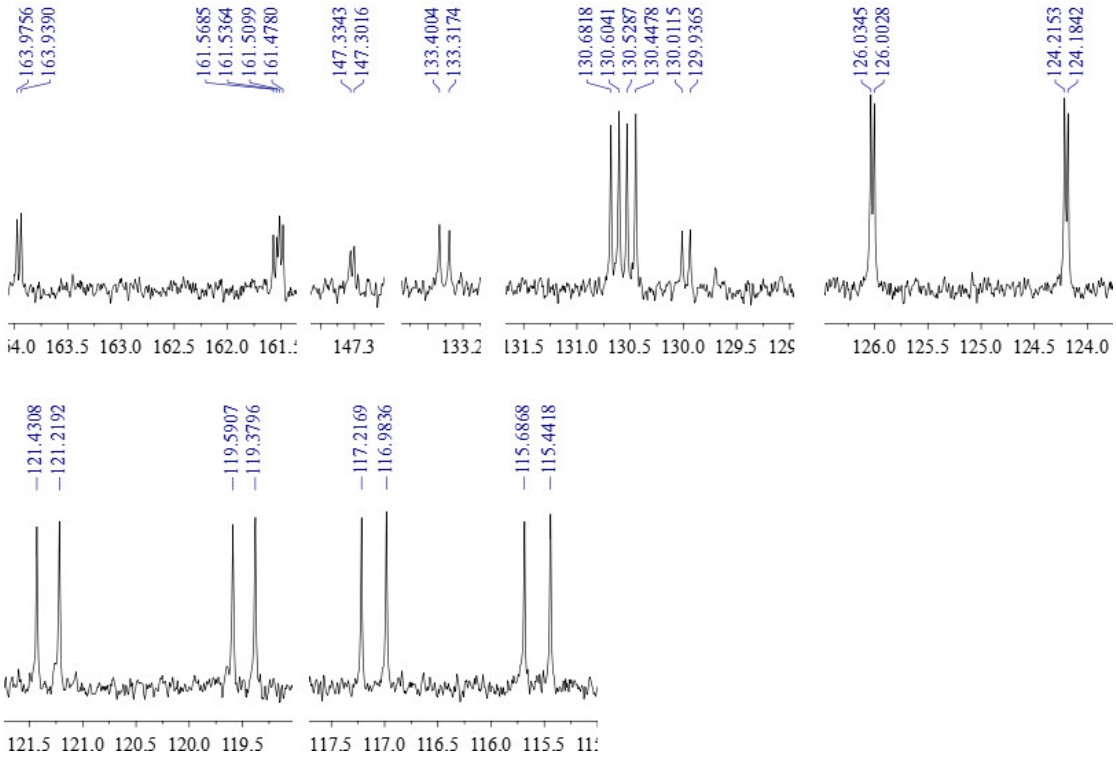


**Figure S21.** Expanded $^{13}C$ NMR (101 MHz, $CDCl_3$) spectrum of compound 2i.

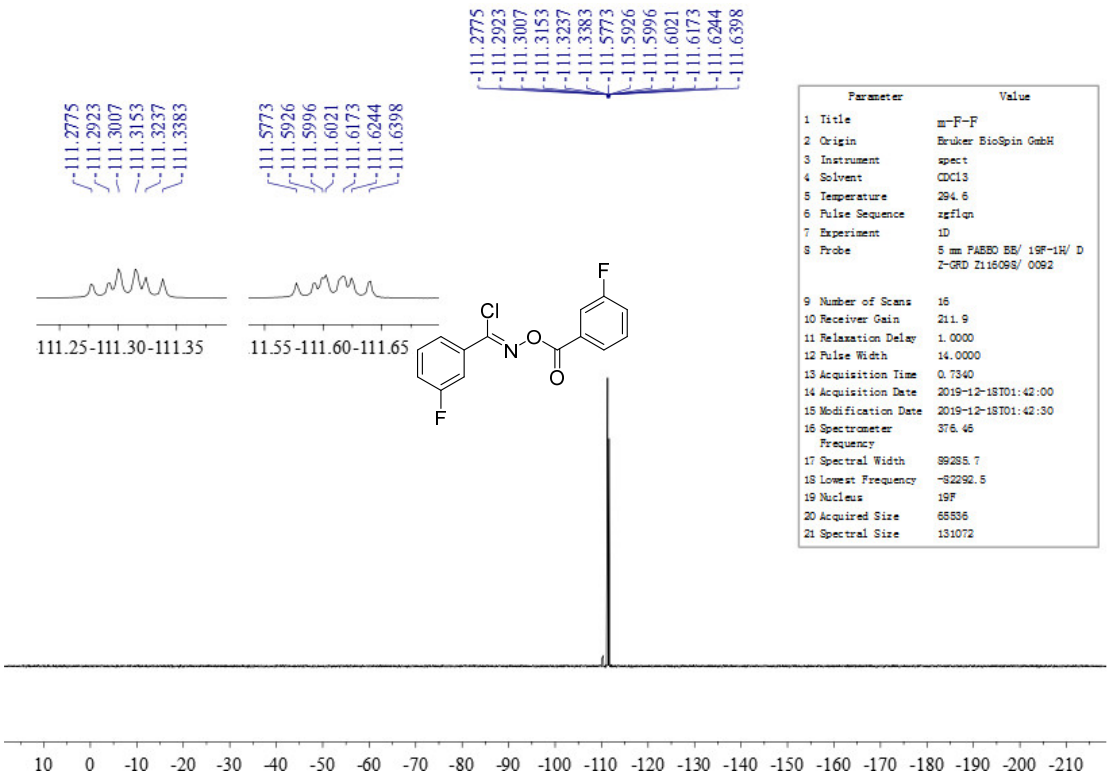


**Figure S22.** $^{19}F$ NMR (376 MHz, $CDCl_3$) spectrum of compound 2i.

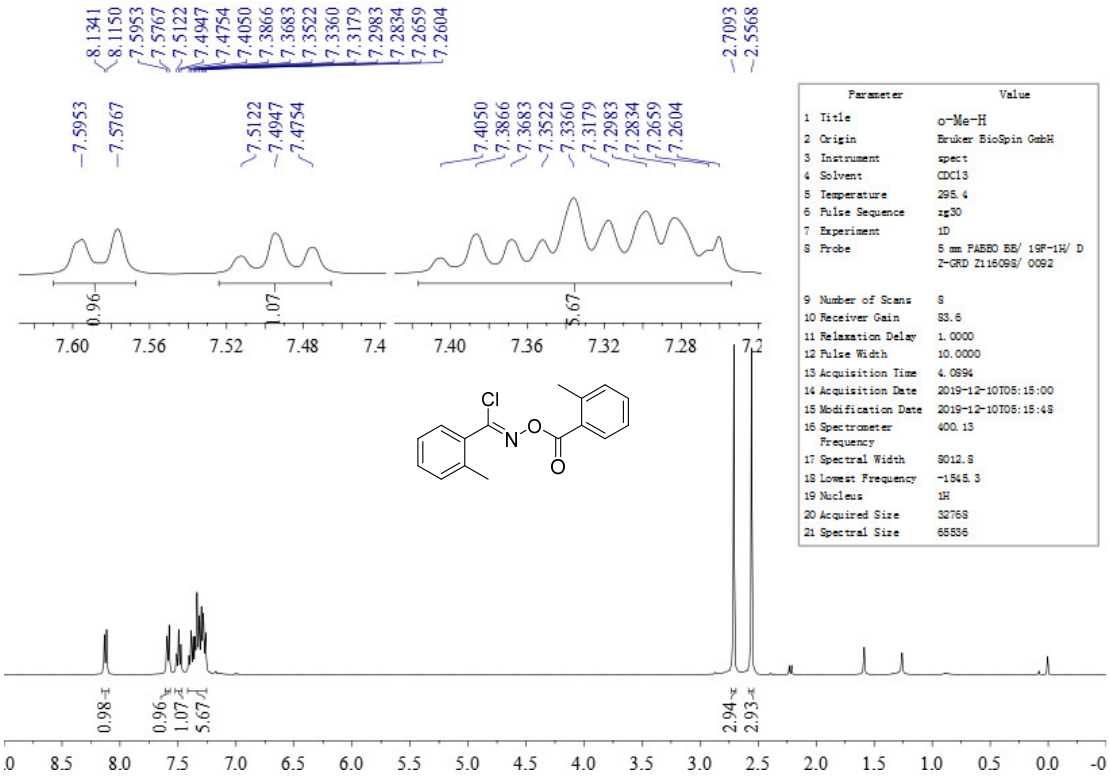


**Figure S23.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2j.

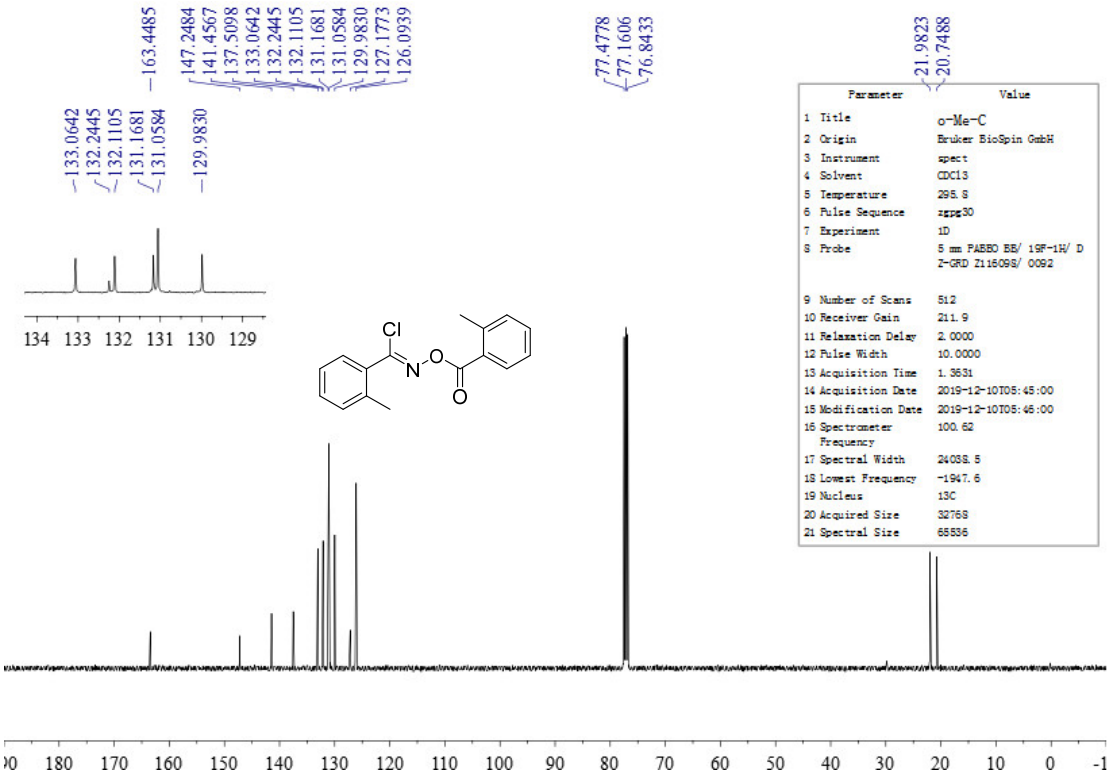


**Figure S24.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2j.

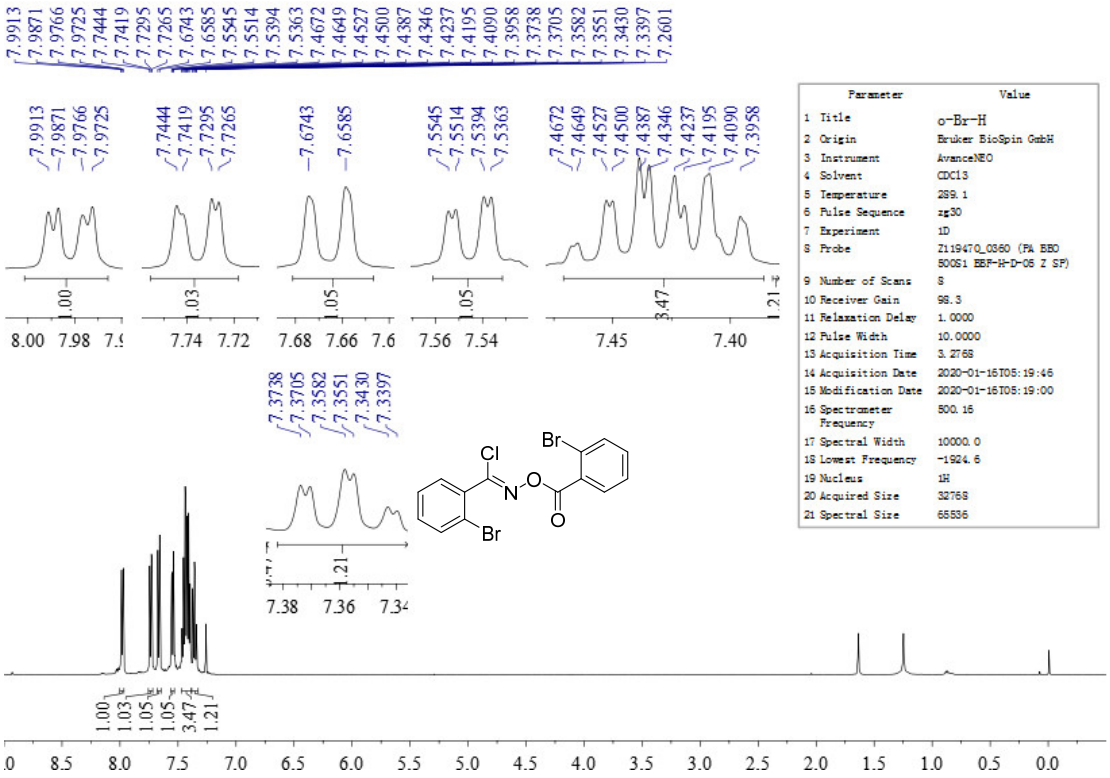


**Figure S25.** $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 2k.

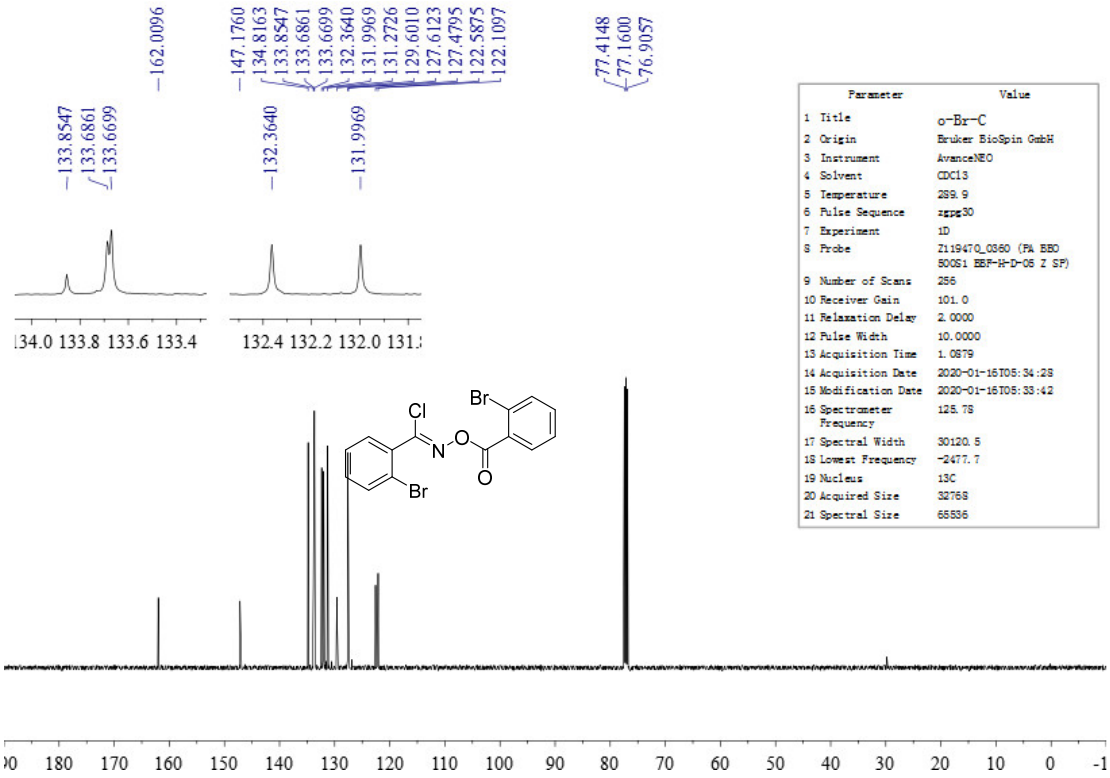


**Figure S26.** $^{13}$C NMR (126 MHz, $CDCl_3$) spectrum of compound 2k.

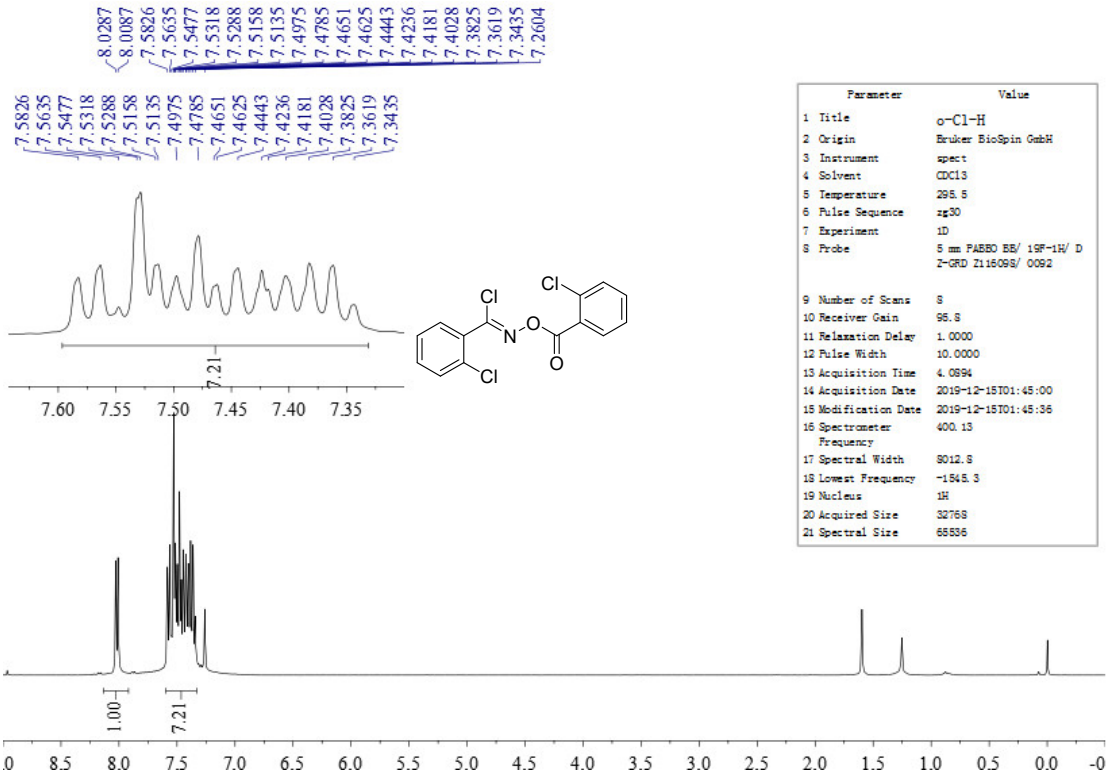


**Figure S27.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2l.

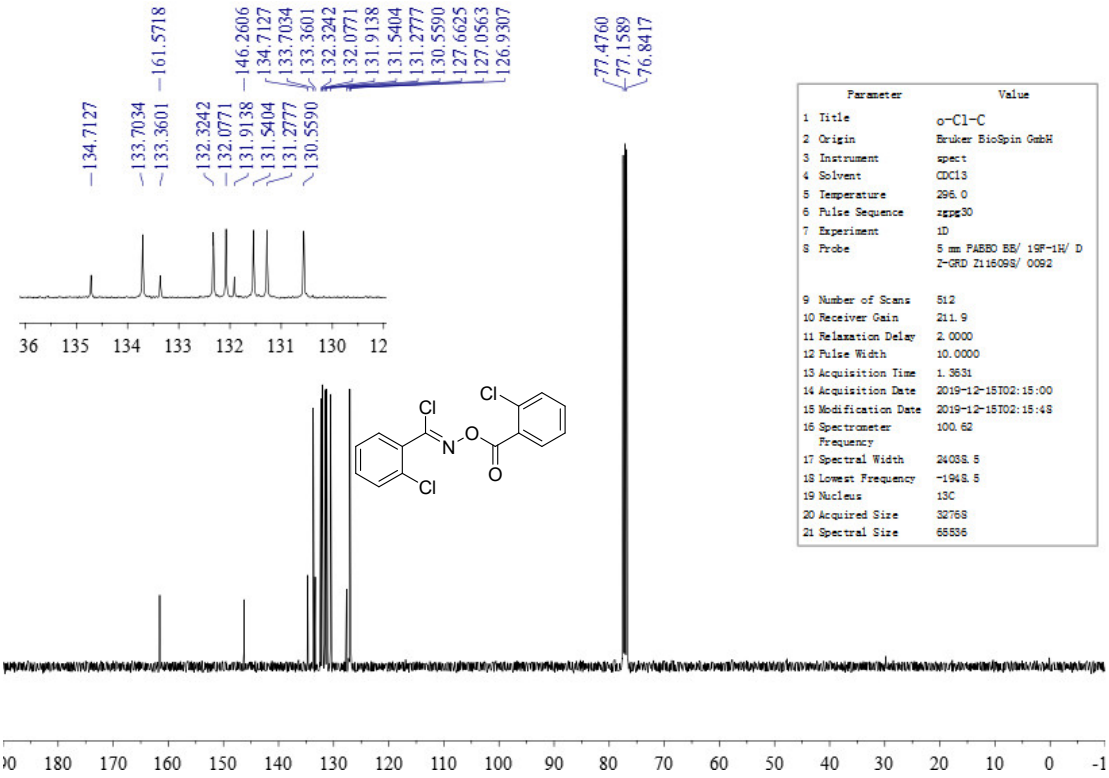


**Figure S28.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2l.

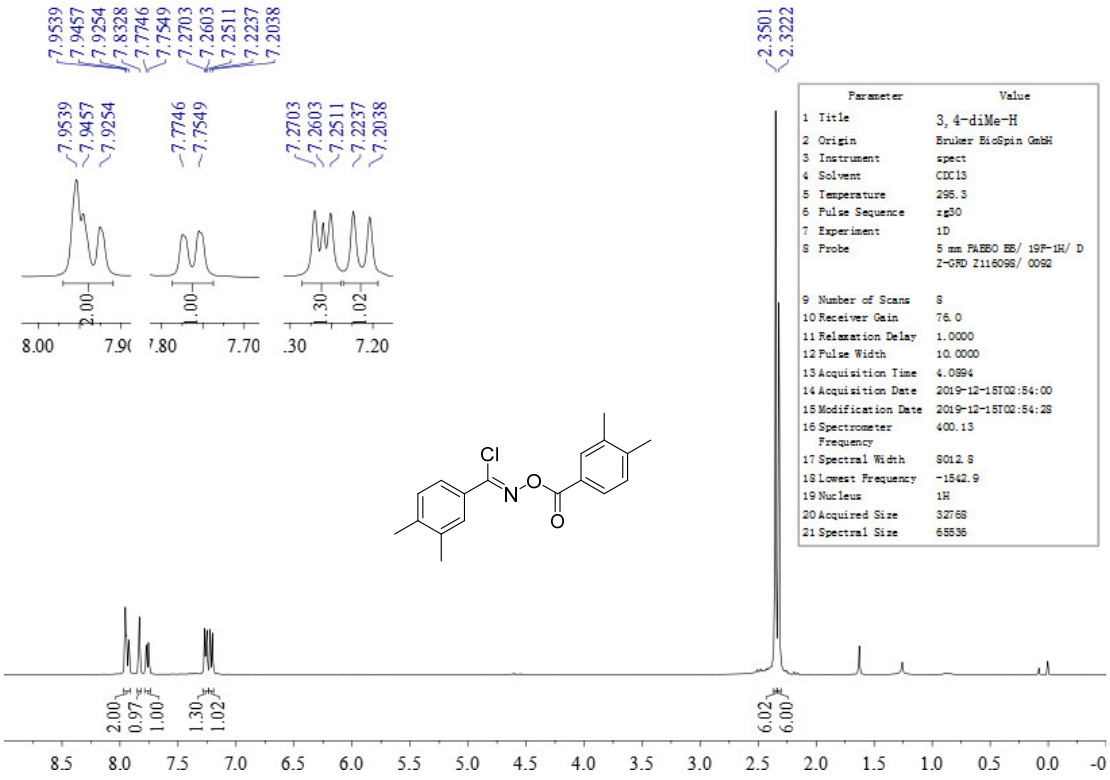


**Figure S29.** $^{1}$H NMR (400 MHz, $CDCl_3$) spectrum of compound 2m.

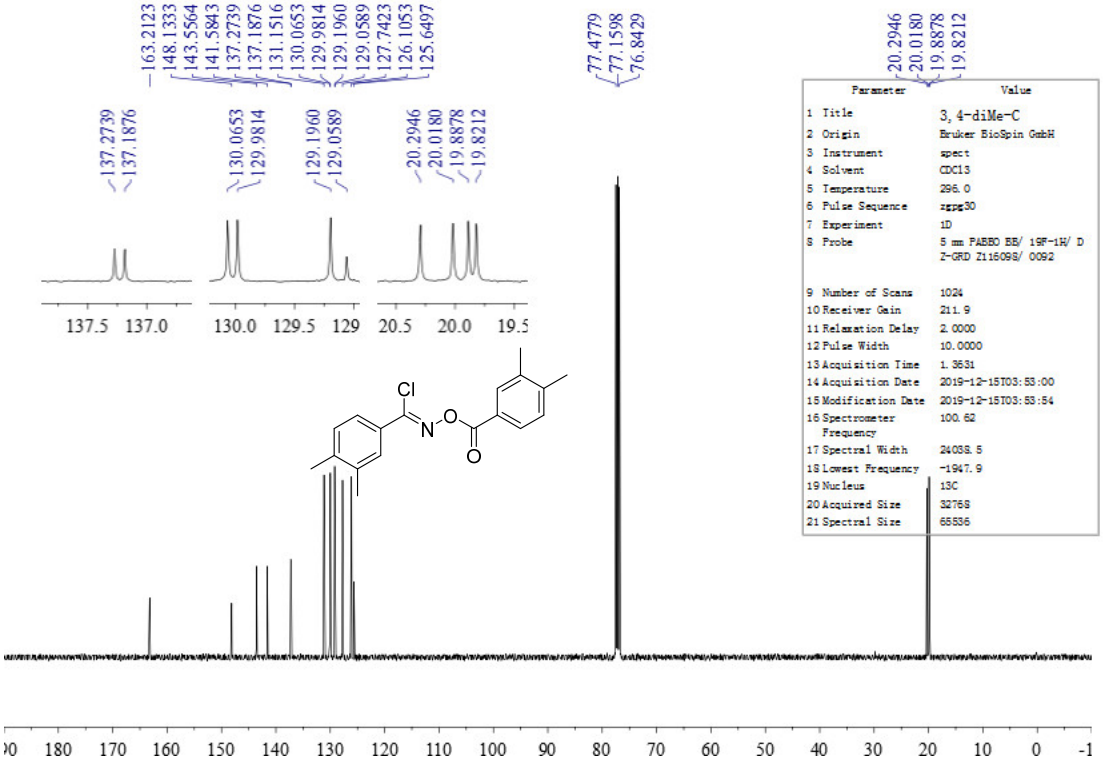


**Figure S30.** $^{13}$C NMR (101 MHz, $CDCl_3$) spectrum of compound 2m.

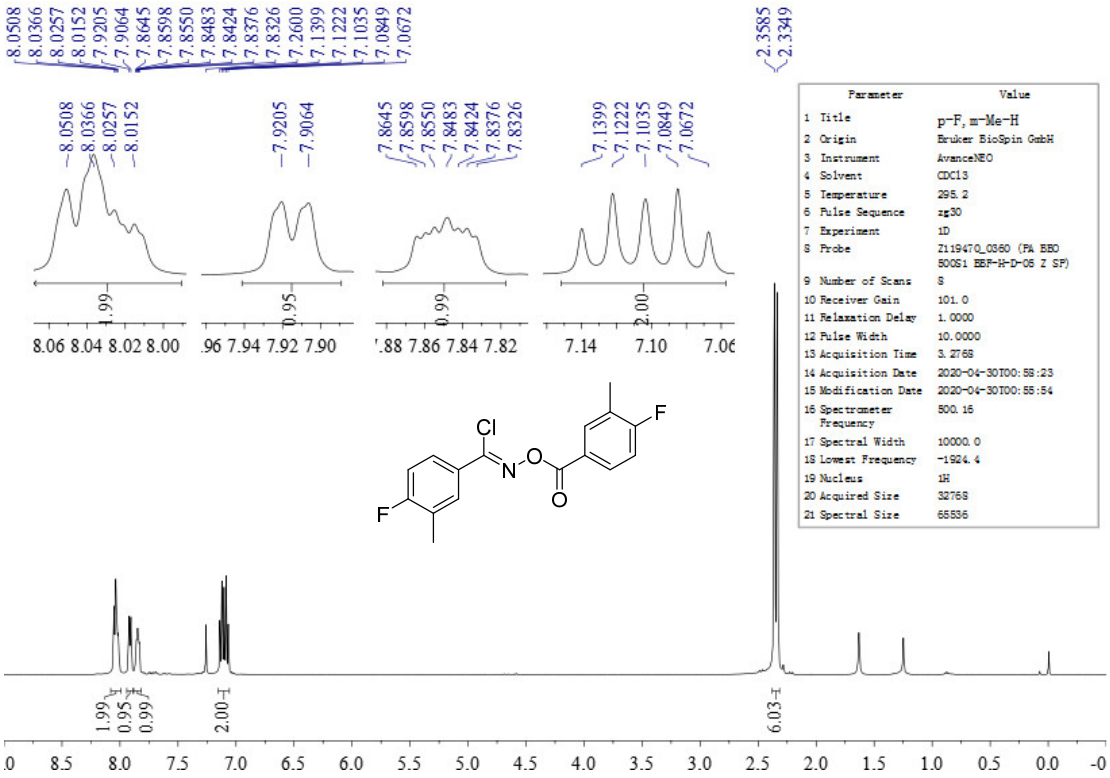


**Figure S31.** $^{1}$H NMR (500 MHz, $CDCl_3$) spectrum of compound 2n.

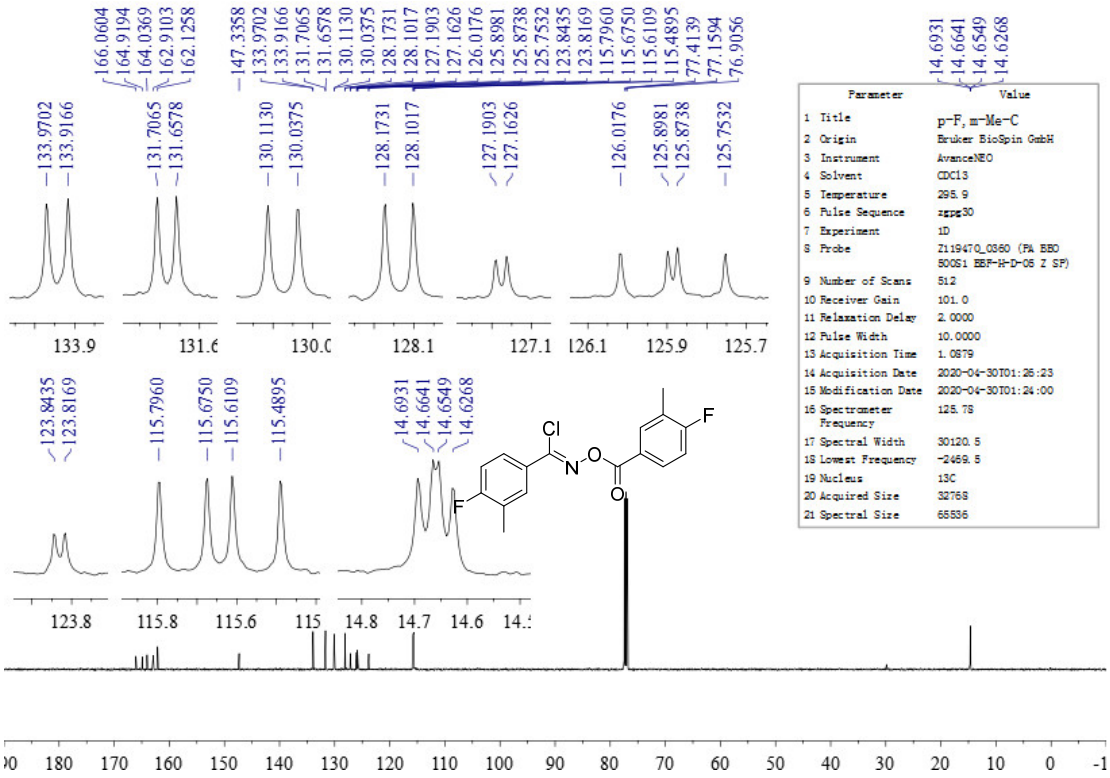


**Figure S32.** $^{13}$C NMR (126 MHz, $CDCl_3$) spectrum of compound 2n.

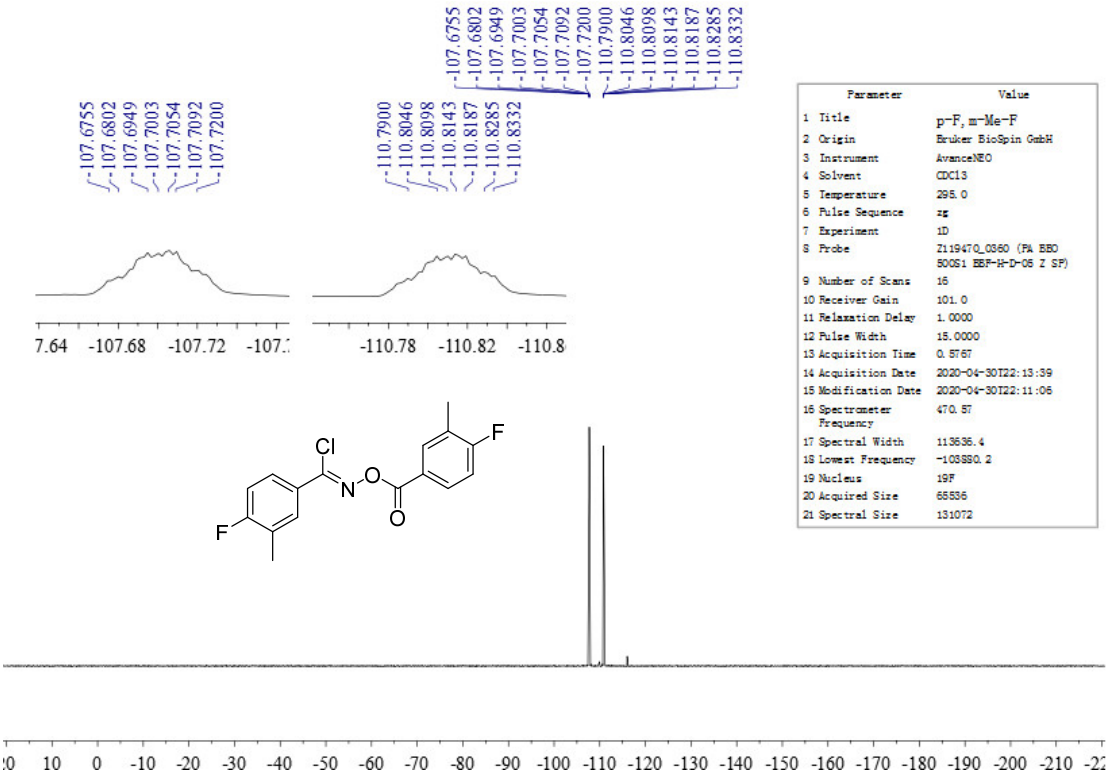


**Figure S33.** $^{19}F$ NMR (471 MHz, $CDCl_3$) spectrum of compound 2n.

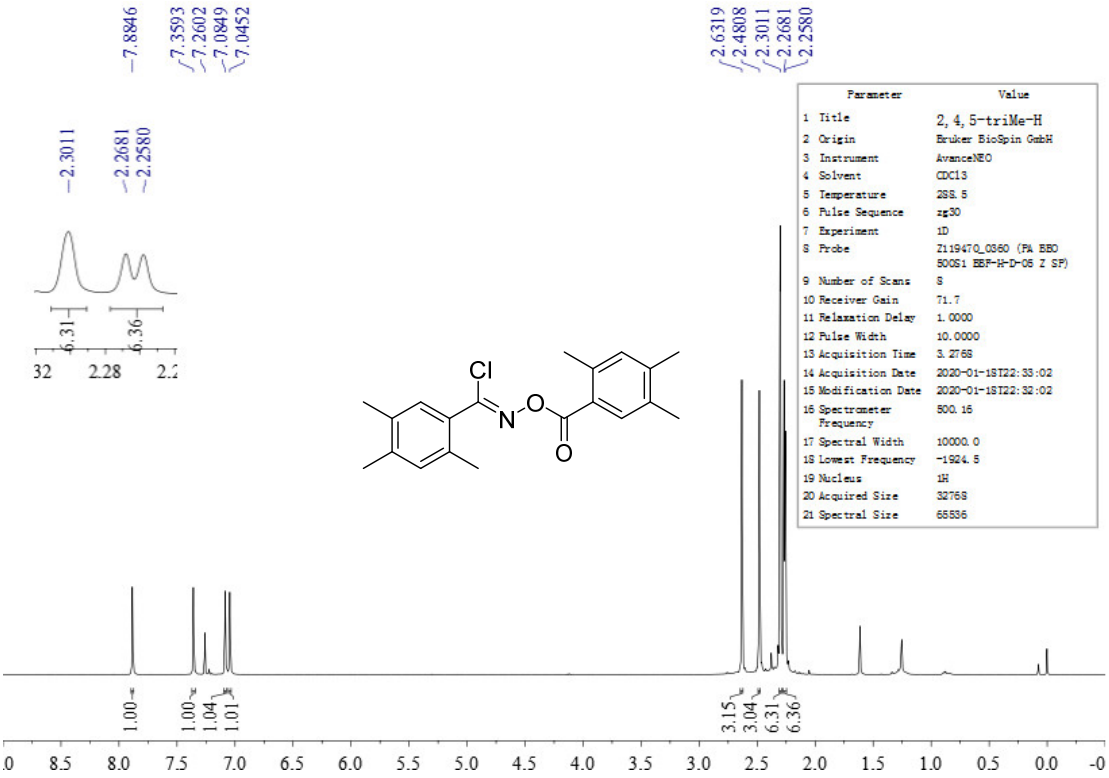


**Figure S34.** $^{1}H$ NMR (500 MHz, $CDCl_3$) spectrum of compound 2o.

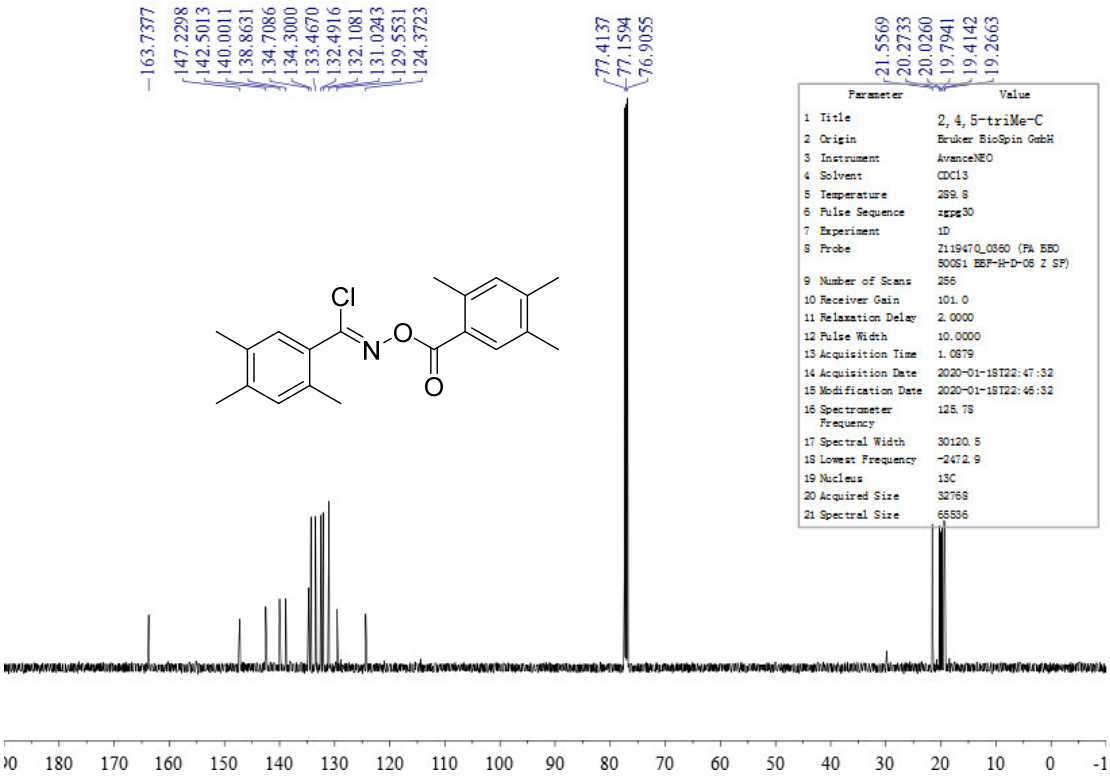


**Figure S35.** $^{13}C$ NMR (126 MHz, $CDCl_3$) spectrum of compound 2o.

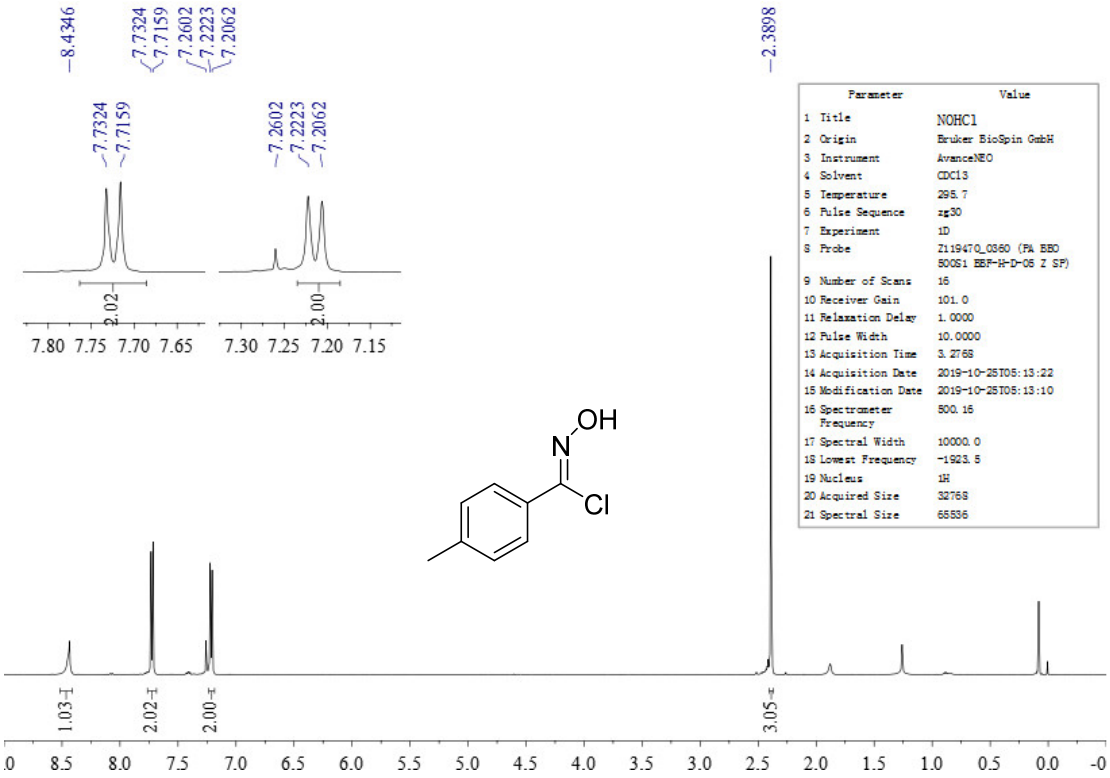


**Figure S36.** $^{1}H$ NMR (500 MHz, $CDCl_3$) spectrum of compound 3a.

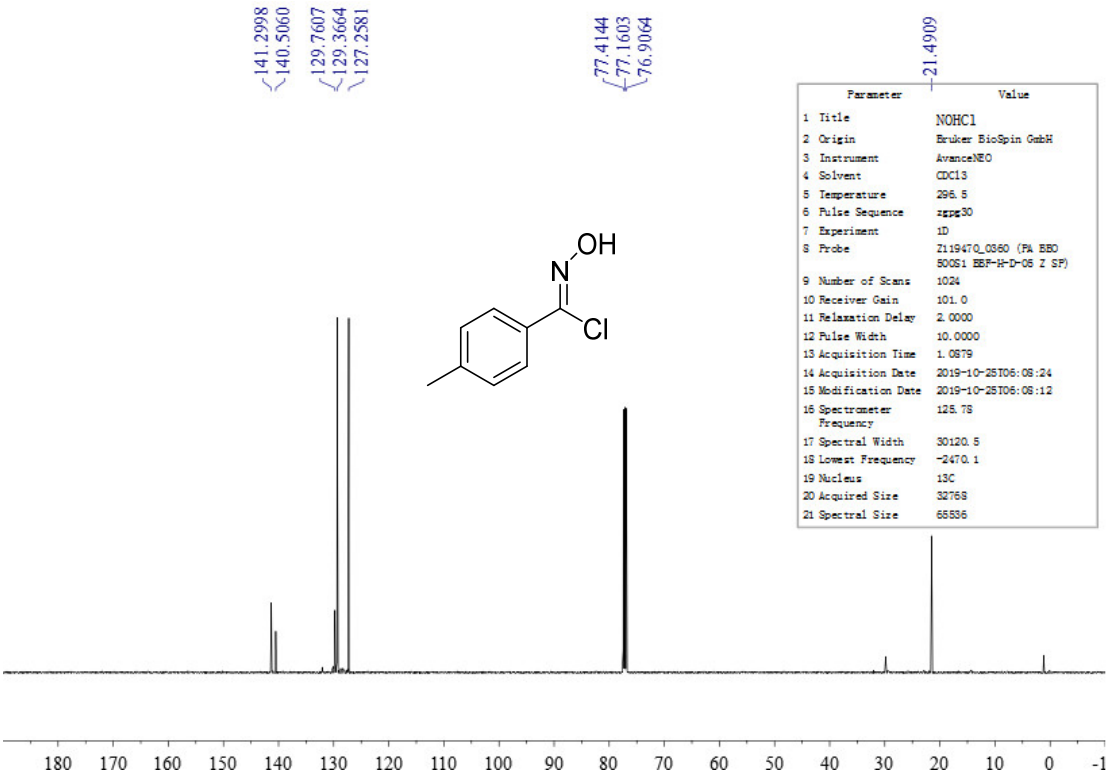


**Figure S37.** $^{13}$C NMR (126 MHz, $CDCl_3$) spectrum of compound 3a.

## 2. Single-Crystal X-ray Crystallography of 2a

Single crystals of **2a** were obtained by slow evaporation from a mixture of dichloromethane/*n*-hexane at 4 °C. Single-crystal X-ray diffraction data were collected on a diffractometer (Gemini S Ultra, Agilent Technologies) equipped with a CCD area detector using graphite-monochromated Cu K$\alpha$ radiation ($\lambda$ = 1.54184 Å) in the scan range 9.254° < 2$\theta$ < 146.750°. The structure was solved with direct methods using SHELXS-97 and refined with full-matrix least-squares refinement using the SHELXL-97 program within OLEX2. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 2003914.

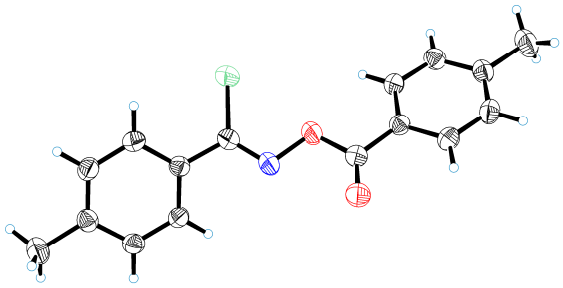

**Figure S38.** ORTEP Diagrams of **2a** with 30% Thermal Ellipsoids.

**Table S1.** Crystal Data and Structure Refinement for 2a.

| **Identification code** | **2003914** |
|---|---|
| Empirical formula | $C_{16}H_{14}ClNO_2$ |
| Formula weight | 287.73 |
| Temperature/K | 293(2) |
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |
| a/Å | 7.3283(3) |
| b/Å | 17.5340(8) |
| c/Å | 11.3990(5) |
| α/° | 90 |
| β/° | 90.530(4) |
| γ/° | 90 |
| Volume/Å$^3$ | 1464.65(11) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 1.305 |
| μ/mm$^{-1}$ | 2.313 |
| F(000) | 600.0 |
| Crystal size/mm$^3$ | 0.250 × 0.220 × 0.150 |
| Radiation | CuKα (λ = 1.54184) |
| 2Θ range for data collection/° | 9.254 to 146.750 |
| Index ranges | $-7 \le h \le 8$, $-21 \le k \le 21$, $-14 \le l \le 11$ |
| Reflections collected | 5918 |
| Independent reflections | 2866 [$R_{int}$ = 0.0196, $R_{sigma}$ = 0.0209] |
| Data/restraints/parameters | 2866/0/183 |
| Goodness-of-fit on $F^2$ | 1.059 |
| Final R indexes [I >= 2σ (I)] | $R_1$ = 0.0496, $wR_2$ = 0.1441 |
| Final R indexes [all data] | $R_1$ = 0.0568, $wR_2$ = 0.1551 |
| Largest diff. peak/hole/e Å$^{-3}$ | 0.33/−0.36 |