Supplementary Information

Visible-light-induced oxytrifluoromethylthiolation of α-diazo esters

Fei Tan,[a★] Jian Yang,[b] Tao Song,[a] Zhangqun Hou,[a] Xiao Huang,[a] Jian Wang,[a] Ling Mei[a] and Hongbo Dong[a★]

[a]Antibiotics Research and Re-evaluation Key Laboratory of Sichuan Province, Sichuan Industrial Institute of Antibiotics, School of Pharmacy, Chengdu University, Chengdu 610106, China

[b]Sichuan Insitute for Drug Control (Sichuan Testing Center of Medical Devices), Chengdu 611731, China

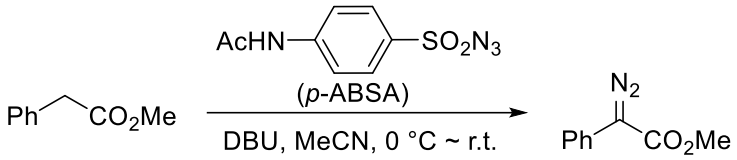
★E-mail: tanfei@cdu.edu.cn, donghongbo@cdu.edu.cn

CONTENTS:

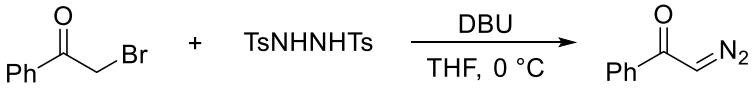
(A)General information

^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts were recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. ^{13}C{^{1}H} NMR data were collected on commercial instruments (101 MHz) with complete proton decoupling. ^{19}F{^{1}H} NMR spectra were collected on commercial instruments (376 MHz) with complete proton decoupling. Chemical shifts were reported in ppm from the tetramethylsilane with the solvent resonance as internal standard. HRMS was recorded on a commercial apparatus (FTMS+c ESI). Amide-based electrophilic trifluoromethylthiolating reagents **1a**−**1d**, PhtSCHF$_2$ (**6a**) and PhtSCCl$_3$ (**9a**) were purchased from domestic suppliers, and used directly. PhtSMe (**4a**) and PhtOCF$_3$ (**12a**) are known compounds, which were synthesized according to the documents. Diazo compounds **2a**−**2t**, **2v**−**2z** are known compounds, which were prepared through the previously reported literature methods, diazo ester **2u** was obtained from domestic supplier and used directly. Chromatography: Qingdao Hailang silica gel (300−400 mesh). The photochemical reactor and LEDs used in this manuscript were purchased from Technology Research: PhotoSyn 3.0.

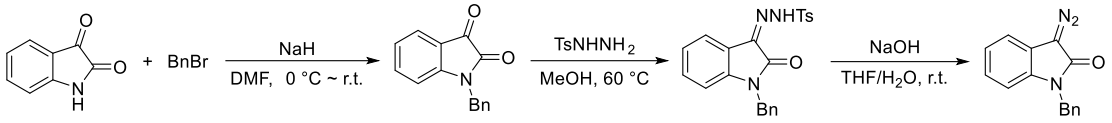
(B)Typical procedure for α-diazo carbonyls preparation[1]



To a solution of methyl 2-phenylacetate (5.0 mmol, 1.0 equiv) and *p*-acetamidobenzenesulfonylazide (*p*-ABSA) (7.5 mmol, 1.5 equiv) in dry CH$_3$CN (25 mL) was added DBU (15 mmol, 1.5 equiv) dropwise at 0 °C. Then the mixture was stirred overnight at room temperature. The reaction was then quenched with aqueous ammonium chloride solution, followed by extraction with Et$_2$O (2 × 10 mL). The combined organic extracts were anhydrous Na$_2$SO$_4$, filtered and concentrated under reduced pressure. The yellow crude product was purified by column chromatography (silica gel, Pet: Et$_2$O = 20:1) to give the product as an orange oil. For other α-diazoesters, this synthesis method could be applied.



2-Bromo-1-phenylethan-1-one (5 mmol, 1.0 equiv) and *N*,*N*′-ditosylhydrazine (5.5 mmol, 1.1 equiv) were dissolved in THF (25 mL) and cooled to 0 °C. DBU (7.5 mmol, 1.5 equiv) was added dropwise and stirred at the temperature for 1 hour. Then the mixture was quenched by the addition of saturated NaHCO$_3$ solution, followed by the extraction with ethyl acetate three times (3 × 30 mL). The organic phase was washed with brine, dried over MgSO$_4$ and evaporated to give the residue, which was purified by column chromatography (petroleum/EA = 10/1) to give the product as a yellow solid.

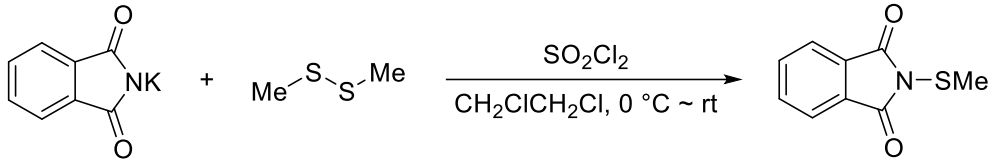


A solution of isatin (20 mmol, 1.0 equiv) in DMF (36 mL) was cooled to 0 °C (ice bath). NaH (60% dispersion in mineral oil, 22 mmol, 1.1 equiv) was added portionwise to the orange solution. The mixture was stirred for 10 min at 0 °C, followed by the addition of benzyl bromide (22 mmol, 1.1 equiv). Then the mixture was stirred at room temperature for 15 min, and cool brine was introduced to precipitate the product. After filtration and drying, the product was used directly for the next step.

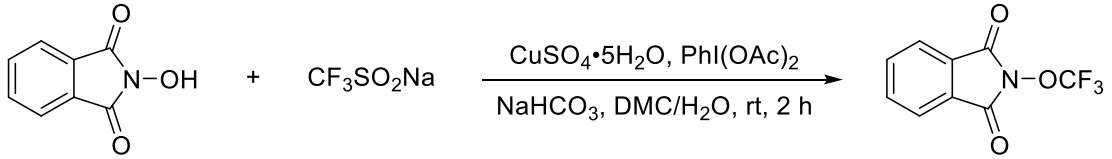
1-Benzyl-isatin (4.2 mmol, 1.0 equiv) was suspended in MeOH (20 mL). The suspension was heated to 60 °C and tosylhydrazine (4.6 mmol, 1.1 equiv) was added in portions. Yellow precipitate appeared and the reaction mixture was stirred at 60 °C for 8 h, then cooled to room temperature. The tosylhydrazone product (yellow precipitate) was collected by filtration and was used without further purification.

To a solution of the tosylhydrazone (3 mmol, 1.0 equiv) in THF (10 mL) was added aqueous solution of NaOH (4.5 mmol, 1.5 equiv, in 10 mL H_2O). The reaction mixture was stirred at room temperature for 3 h. H_2O (30 mL) was added to the reaction mixture and followed by the extraction with EtOAc (30 mL). The organic layer was washed by brine twice and dried over Na_2SO_4, filtered, evaporated to give the residue. Flash chromatography (PE:EA = 10:1) afforded the 2-diazo-1-phenylethan-1-one as a deep-orange crystal.

(C)Procedure for PhtSMe and PhtOCF$_3$ preparation[2]



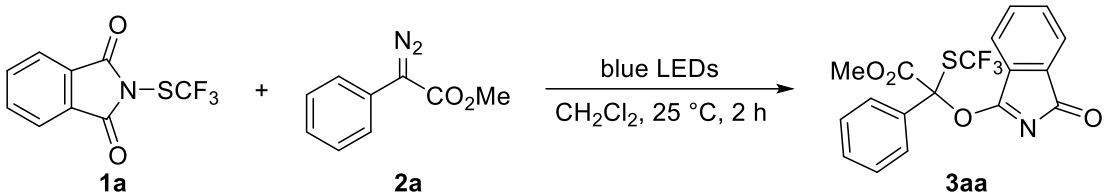
Dimethyldisulfide (10 mmol, 1.0 equiv) was dissolved in 1,2-dichloroethane (20 mL) and cooled to 0 ºC. A 1,2-dichloroethane solution (20 mL) of sulfuryl chloride (11 mmol) was added dropwise over 15 min via an addition funnel to the cooled dimethyldisulfide solution. The reaction was stirred at 0 ºC for an additional 5 min and was then rapidly charged with potassium phthalimide (20 mmol). After 20 min at 0 ºC and 90 min at room temperature, the reaction mixture was filtered under vacuum and the resultant yellow filtrate was concentrated to solids. The solids were triturated with MeOH overnight to afford PhtSMe as a white fluffy solid.



An oven-dried reaction tube with a stirrer bar was charged with $NaHCO_3$ (5 mmol, 0.5 equiv), $CuSO_4 \cdot 5H_2O$ (0.5 mmol, 0.05 equiv), and CF_3SO_2Na (20 mmol, 2.0 equiv). Then, H_2O (8.0 mL) and dimethyl carbonate (30.0 mL) were added. The reaction mixture was cooled to 0 °C. $PhI(OAc)_2$ (20 mmol, 2.0 equiv) and *N*-

hydroxyphthalimide (10 mmol, 1.0 equiv) were added at 0 °C. Then, the reaction mixture was stirred at room temperature for 2 h. The aqueous layer was separated and extracted with EtOAc three times. The combined organic layer was washed with saturated aqueous $NaHCO_3$, dried over anhydrous $MgSO_4$, then filtered and concentrated in vacuo to give a residue. The residue was purified with silica gel column chromatography (hexane/EtOAc = 20:1) to afford $PhtOCF_3$ as a white solid.

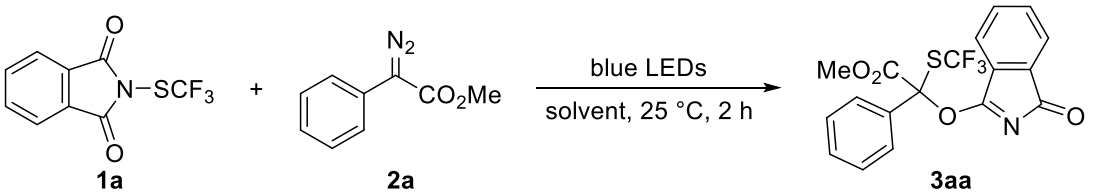
(D)General procedure for the photochemical reaction



A dry reaction tube was charged with **1a** (0.1 mmol) under N_2 atmosphere, then dichloromethane (0.5 mL) was added. Subsequently, α-diazo ester **2a** (0.2 mmol) was added and the mixture was stirred under the irradiation of blue LEDs (460 ± 5 nm, 3 W) at 25 °C for 2 h. Finally, the crude mixture was purified by column chromatography on silica gel (petroleum/ethyl acetate = 10:1 as eluent) at -20 °C to afford the product **3aa** as a colorless gum.

(E)Optimization of the reaction conditions

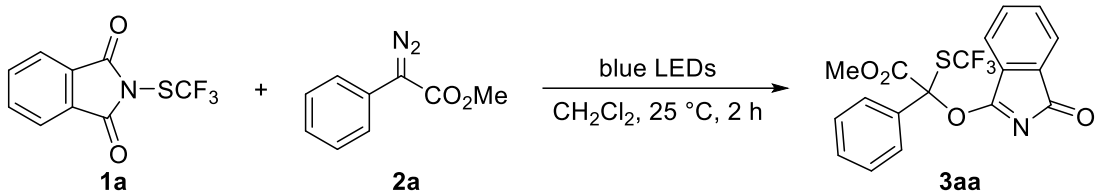
Table S1: Screen of the solvent[a]



Entry	Solvent	Yield (%)
1	CH_2Cl_2	50
2	$CHCl_3$	26
3	CH_2ClCH_2Cl	49
4	$CHCl_2CH_2Cl$	33
5	$CHCl_2CHCl_2$	—[b]
6	EA	46
7	Methyl Acetate	45
8	Ethyl Formate	37
9	MeCN	48
10	DMF	—[b]
11	DMSO	—[b]
12	toluene	—[b]

[a]Unless otherwise noted, all reactions were performed with **1a** (0.1 mmol) and **2a** (0.1 mmol) in solvent (0.5 mL) under blue LEDs (460 ± 5 nm, 40 W) at 25 °C for 2 h. Isolated yield. [b]**2a** decomposed and **3aa** was undetected.

Table S2: Screen of the ratio between 1a and 2a[a]

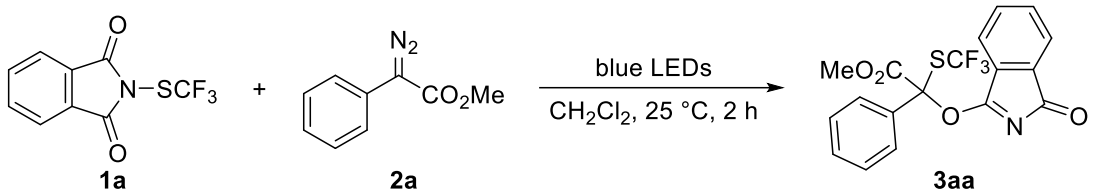


1a x mmol **2a** y mmol **3aa**

Entry	x	y	Yield (%)
1	0.2	0.1	64
2	0.15	0.1	55
3	0.1	0.1	50
4	0.1	0.15	67

[a]Unless otherwise noted, all reactions were performed with **1a** (x mmol) and **2a** (y mmol) in CH_2Cl_2 (0.5 mL) under blue LEDs (460 ± 5 nm, 40 W) at 25 °C for 2 h. Isolated yield.

Table S3: Screen of the irradiation conditions[a]

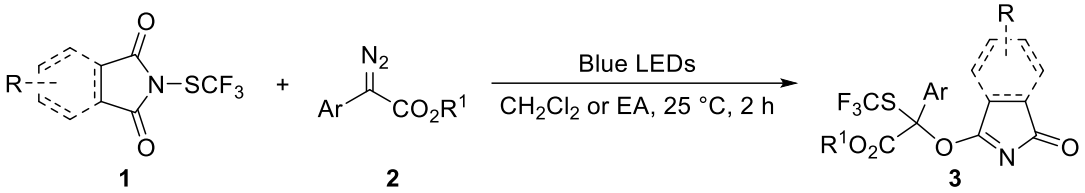


1a **2a** **3aa**

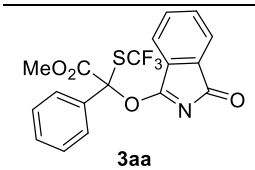
Entry	Blue LEDs	Yield (%)
1	460 ± 5 nm, 20 W	31
2	460 ± 5 nm, 10 W	53
3	460 ± 5 nm, 5 W	53
4	460 ± 5 nm, 3 W	67
5	460 ± 5 nm, 2 W	58
6[b]	460 ± 5 nm, 2 W	64
7[c]	480 ± 5 nm, 3 W	37
8[c]	470 ± 5 nm, 3 W	46
9	450 ± 5 nm, 3 W	39
10	440 ± 5 nm, 3 W	47
11[d]	460 ± 5 nm, 3 W	76
12[d,e]	460 ± 5 nm, 3 W	39
13[d,f]	460 ± 5 nm, 3 W	49
14[d,g]	460 ± 5 nm, 3 W	34
15[h]	—	N.R.
16[i]	—	N.R.
17[j]	—	trace

[a]Unless otherwise noted, all reactions were performed with **1a** (0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (0.5 mL) under blue LEDs at 25 °C for 2 h. Isolated yield. [b]Reaction time: 3 h. [c]Reaction time: 4 h. [d]0.2 mmol **2a** was used. [e]0.2 mL CH_2Cl_2 was used. [f]1.0 mL CH_2Cl_2 was used. [g]At -10 °C. [h]In the dark at 35 °C for 2 h, N.R. = no reaction. [i]In the dark at 40 °C for 6 h. [j]In the dark at 80 °C for 6 h, most of **2a** decomposed and **1a** remained, α-keto ester **16** and (*E*)-dimethyl 2,3-diphenylfumarate were detected.

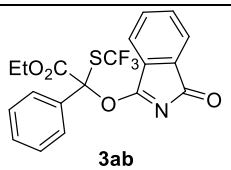
Table S4: Full list of the products[a]



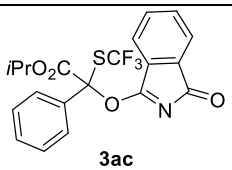
1 2 3



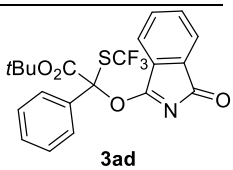
3aa
76% (72%)[b] yield



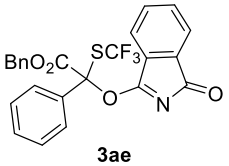
3ab
72% (74%)[b] yield



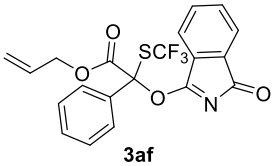
3ac
71% (66%)[b] yield



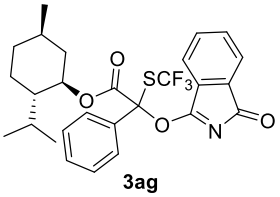
3ad
36% (39%)[b] yield



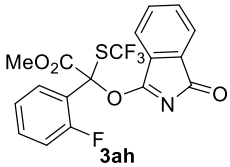
3ae
74% (70%)[b] yield



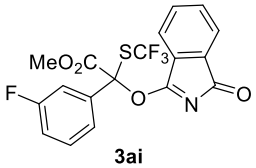
3af
58% (64%)[b] yield



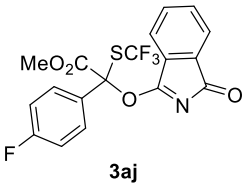
3ag
73% (69%)[b] yield, 1:1 dr



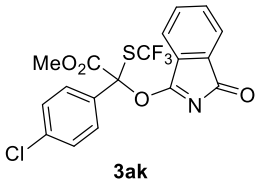
3ah
39% (37%)[b] yield



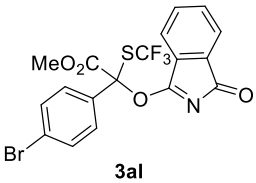
3ai
<44% (<27%)[b] yield



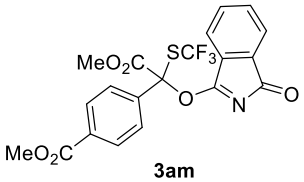
3aj
40% (30%)[b] yield



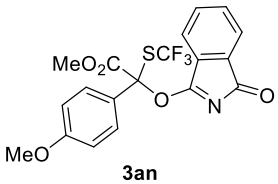
3ak
79% (65%)[b] yield



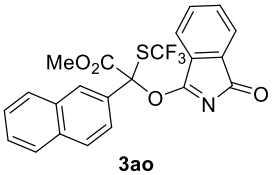
3al
71% (66%)[b] yield



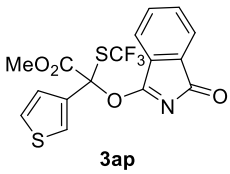
3am
<38% (<16%)[b] yield



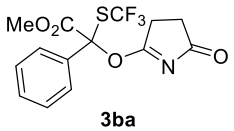
3an
22% (8%)[b] yield



3ao
60% (53%)[b] yield

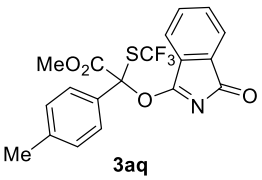


3ap
39% (9%)[b] yield

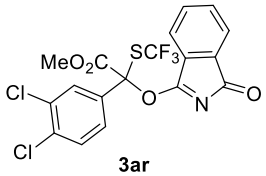


3ba
75% (69%)[b] yield

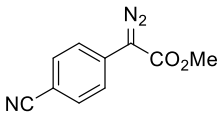
limitations:



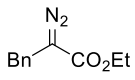
3aq
difficult to purify
mixture (<49% yield)



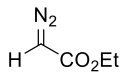
3ar
difficult to purify
mixture (<85% yield)



2s
decomposed



2t
decomposed



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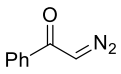
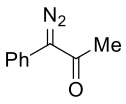
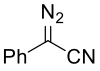
decomposed



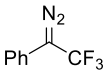
2v
decomposed



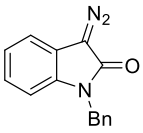
2w
decomposed



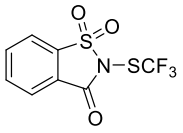
2x
decomposed



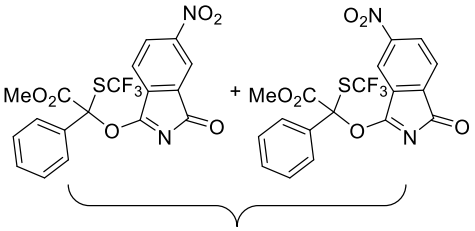
2y
decomposed



2z
decomposed



1c
messy



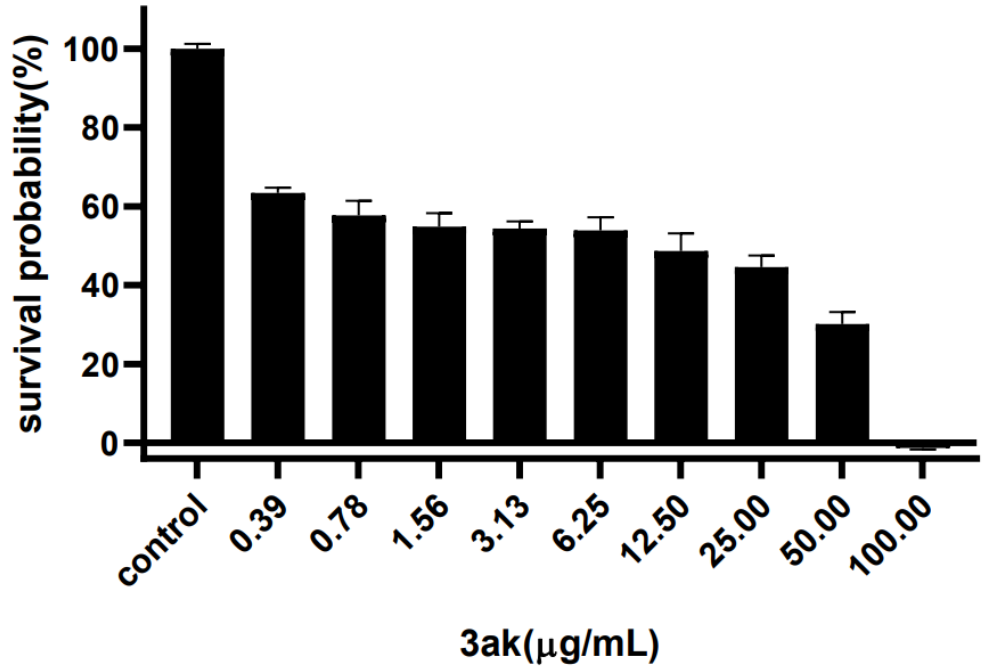
3da + **3da'**
unstable, difficult to purify
mixture (<34% yield)

[a]Unless otherwise noted, all reactions were performed with **1** (0.1 mmol) and **2** (0.2 mmol) in CH_2Cl_2 (0.5 mL) under blue LEDs (460 ± 5 nm, 3 W) at 25 °C for 2 h. Isolated yield. [b]Using EA (0.5 mL) as the solvent.

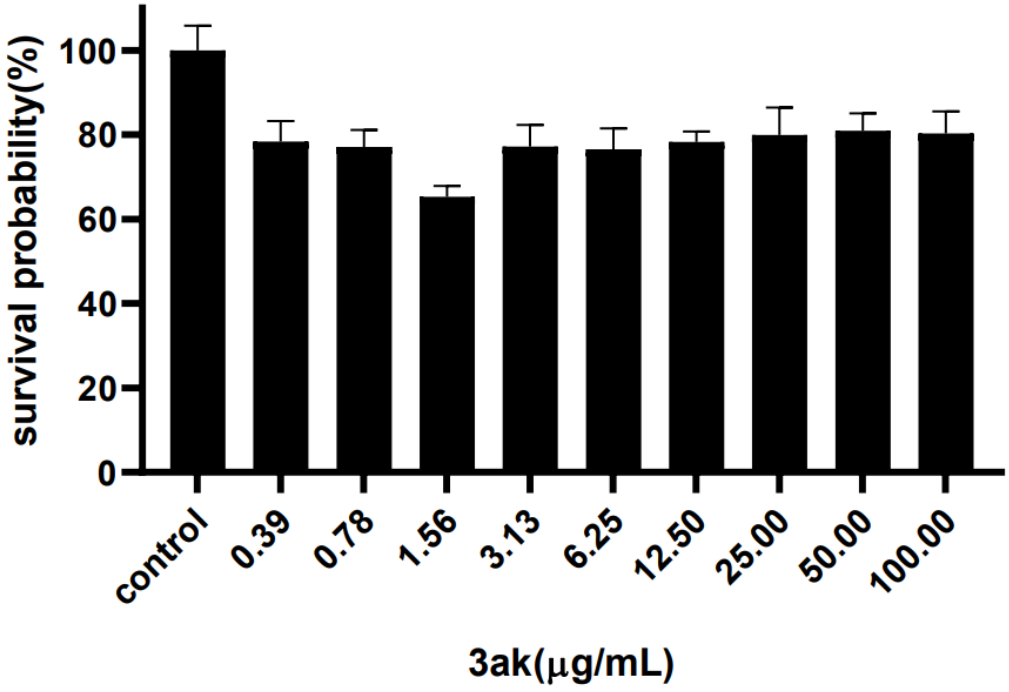
(F) Antimicrobial evaluation of 3ak

The Gram-positive *Staphylococcus aureus* ATCC25923 and Gram-negative *Escherichia coli* ATCC25922 were treated with the DMSO solution of **3ak** (0.39 μg/mL, 0.78 μg/mL. 1.56 μg/mL, 3.13 μg/mL, 6.25 μg/mL, 12.50 μg/mL, 25.00 μg/mL, 50.00 μg/mL, 100.00 μg/mL), respectively.

a) Survival probability of *S. aureus* ATCC25923

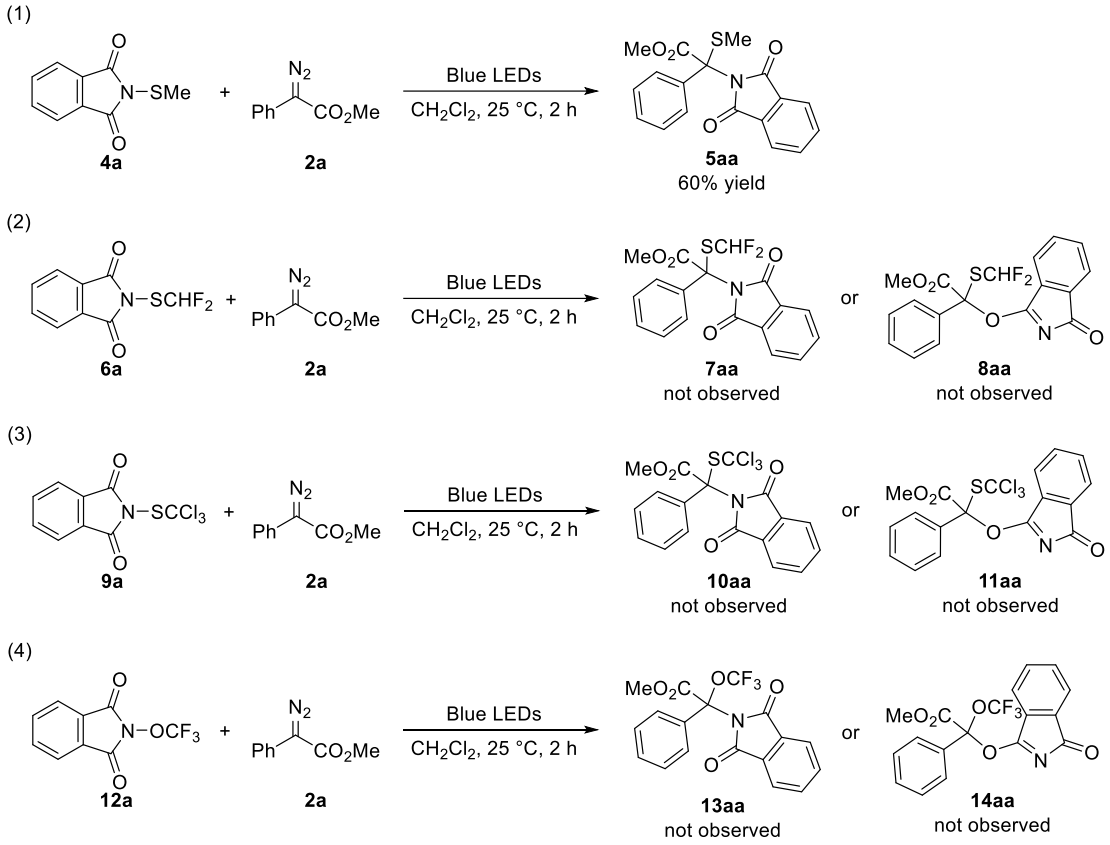


b) Survival probability of *E. coli* ATCC25922



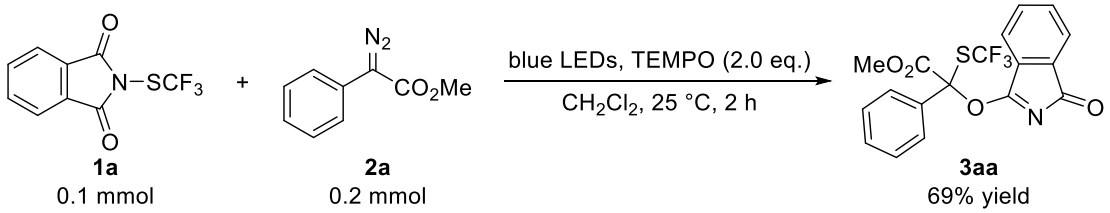
(G) Control and transformation experiments

a) Variation of the trifluoromethylthio group



A dry reaction tube was charged with *N*-substituted phthalimide **4a**/**6a**/**9a**/**12a** (0.1 mmol), then CH_2Cl_2 (0.5 mL) was added. Subsequently, α-diazo ester **2a** (0.2 mmol) was added and the mixture was stirred under the irradiation of blue LEDs (460 ± 5 nm, 3 W) at 25 °C for 2 h. The reaction was monitored by TLC and purified by column chromatography on silica gel (petroleum/ethyl acetate = 10:1 as eluent) at -20 °C. For substrate **4a**, the crude mixture was purified by column chromatography on silica gel (petroleum/ethyl acetate = 8:1 as eluent) at room temperature to afford product **5aa** as a colorless gum.

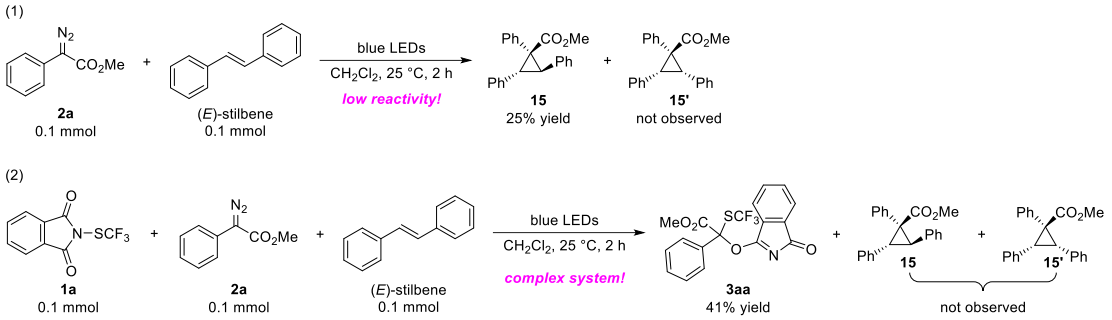
b) Radical termination experiment



A dry reaction tube was charged with **1a** (0.1 mmol) and TEMPO (0.2 mmol) under N_2 atmosphere, then dichloromethane (0.5 mL) was added. Subsequently, α-diazo ester **2a** (0.2 mmol) was added and the mixture was stirred under the irradiation of blue LEDs (460 ± 5 nm, 3 W) at 25 °C for 2 h. Finally, the crude mixture was purified by column

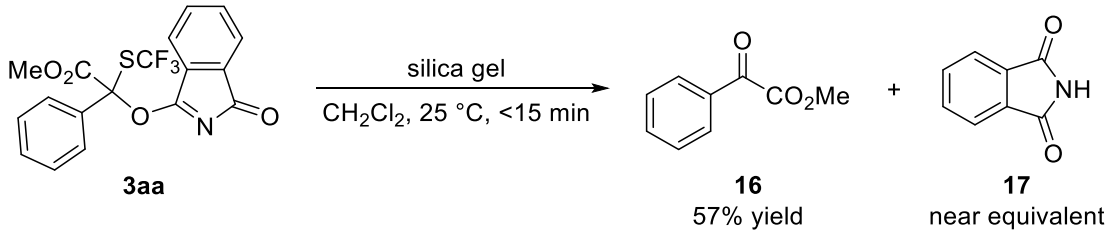
chromatography on silica gel (petroleum/ethyl acetate = 10:1 as eluent) at -20 °C to afford the product **3aa** as a colorless gum.

c) Carbene capture with (*E*)-stilbene



A dry reaction tube was charged with **1a** (0.1 mmol) and/or (*E*)-stilbene (0.1 mmol) under N_2 atmosphere, then dichloromethane (0.5 mL) was added. Subsequently, α-diazo ester **2a** (0.1 mmol) was added and the mixture was stirred under the irradiation of blue LEDs (460 ± 5 nm, 3 W) at 25 °C for 2 h. Finally, the crude mixture was purified by column chromatography on silica gel (petroleum/ethyl acetate = 20:1 as eluent) to afford product **15** as a white solid, or by column chromatography on silica gel (petroleum/ethyl acetate = 10:1 as eluent) at -20 °C to afford the product **3aa** as a colorless gum.

d) Decomposition of 3aa

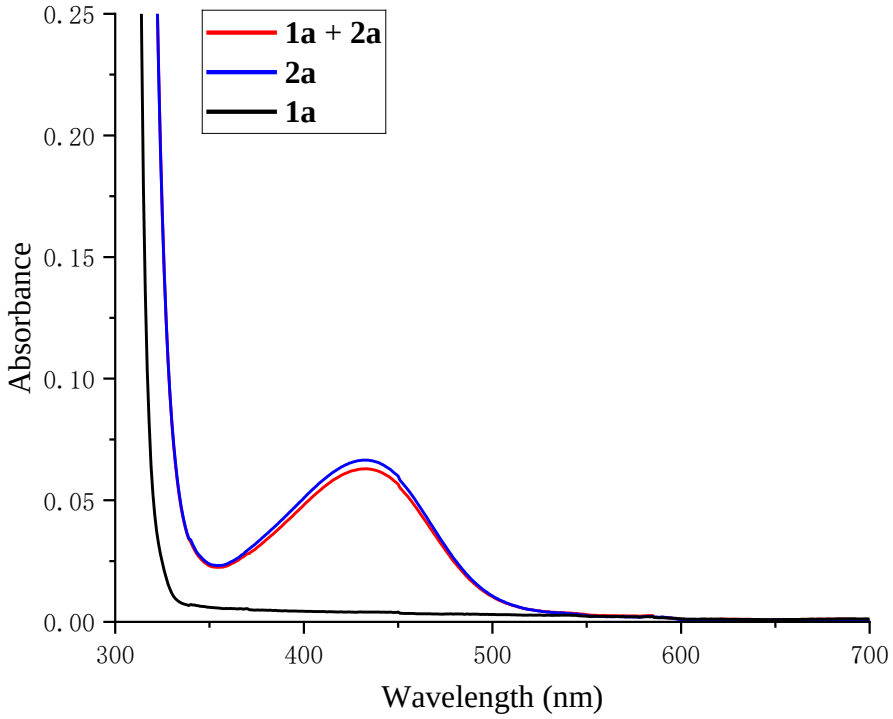


A vial was charged with phosphate **3aa** (0.12 mmol), then CH_2Cl_2 (0.5 mL) was added. Subsequently, silica gel (200~300 mesh, 20 mg) was added and the mixture was stirred at 25 °C for ~15 min. Finally, the crude mixture was purified by column chromatography on silica gel (petroleum/ethyl acetate = 10:1~0:1 as eluent) to afford α-keto ester **16** as a yellow oil in 57% yield and phthalimide **17** as a white solid in near quantitative yield.

(H)UV-vis absorption and fluorescence experiments

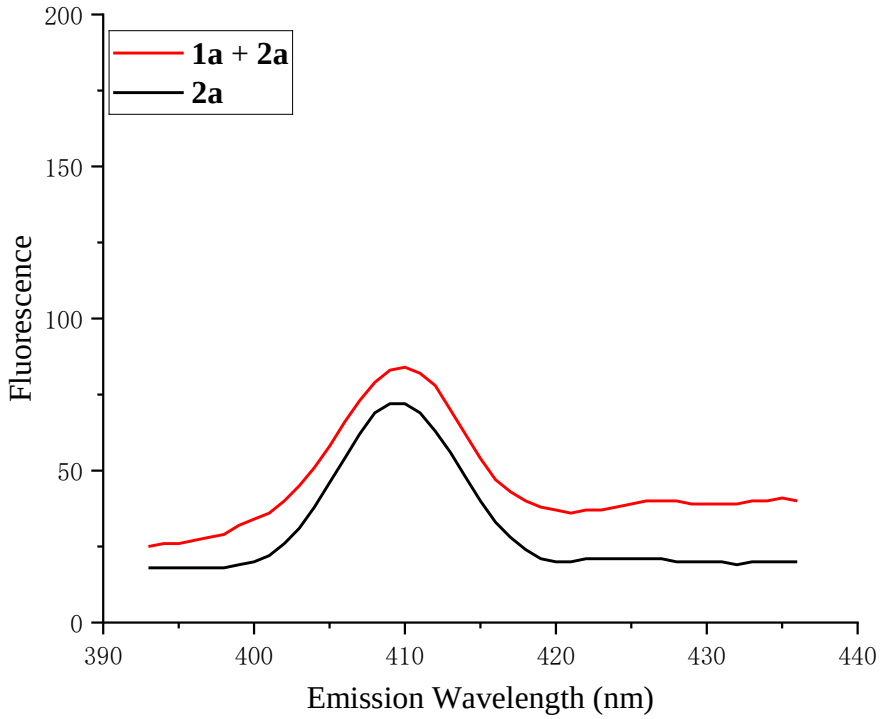
a) UV-vis absorption spectra

The fresh solutions of **1a** (0.5 mM in DCM) and **2a** (0.5 mM in DCM) were prepared and measured by UV-2450 (A10834534597CS). The solution of the mixture of **1a** and **2a** (0.5 mM in DCM) was prepared by mixing equal volumes of **1a** (1.0 mM in DCM) and **2a** (1.0 mM in DCM) in *situ* before measurement.



b) fluorescence spectra

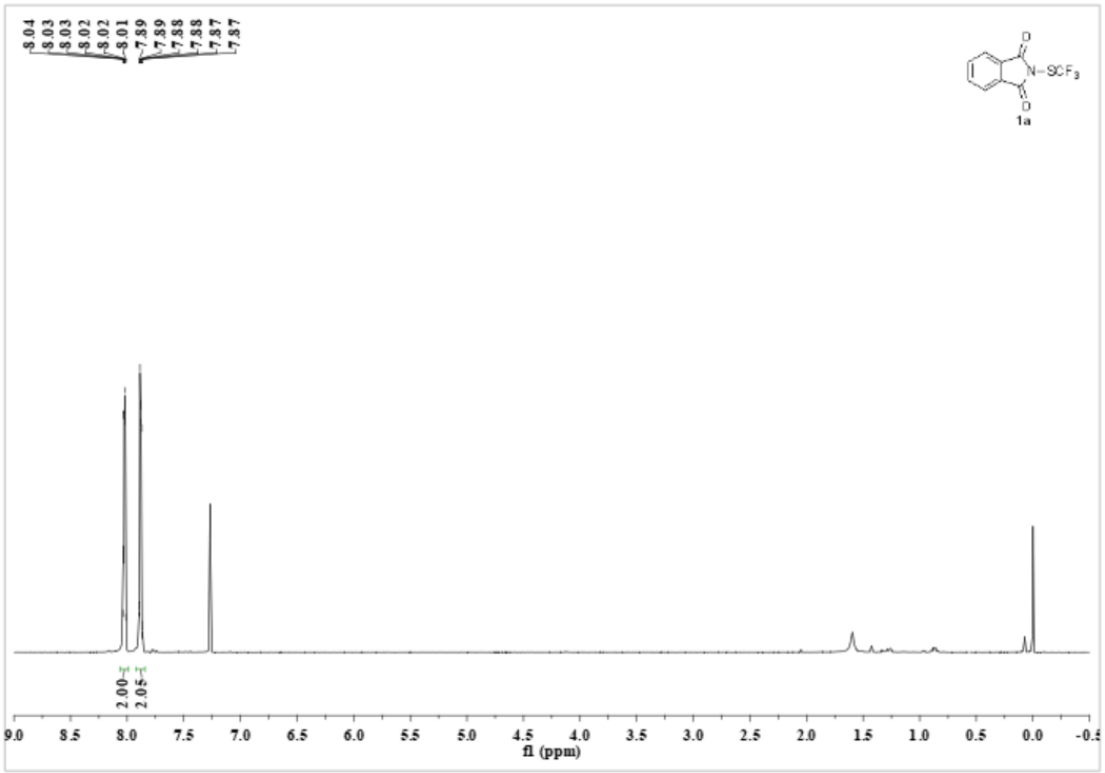
The fresh solution of **2a** (0.5 mM in DCM) was prepared and measured by FL970. The solution of the mixture of **1a** and **2a** (0.5 mM in DCM) was prepared by mixing equal volumes of **1a** (1.0 mM in DCM) and **2a** (1.0 mM in DCM) in *situ* before measurement.

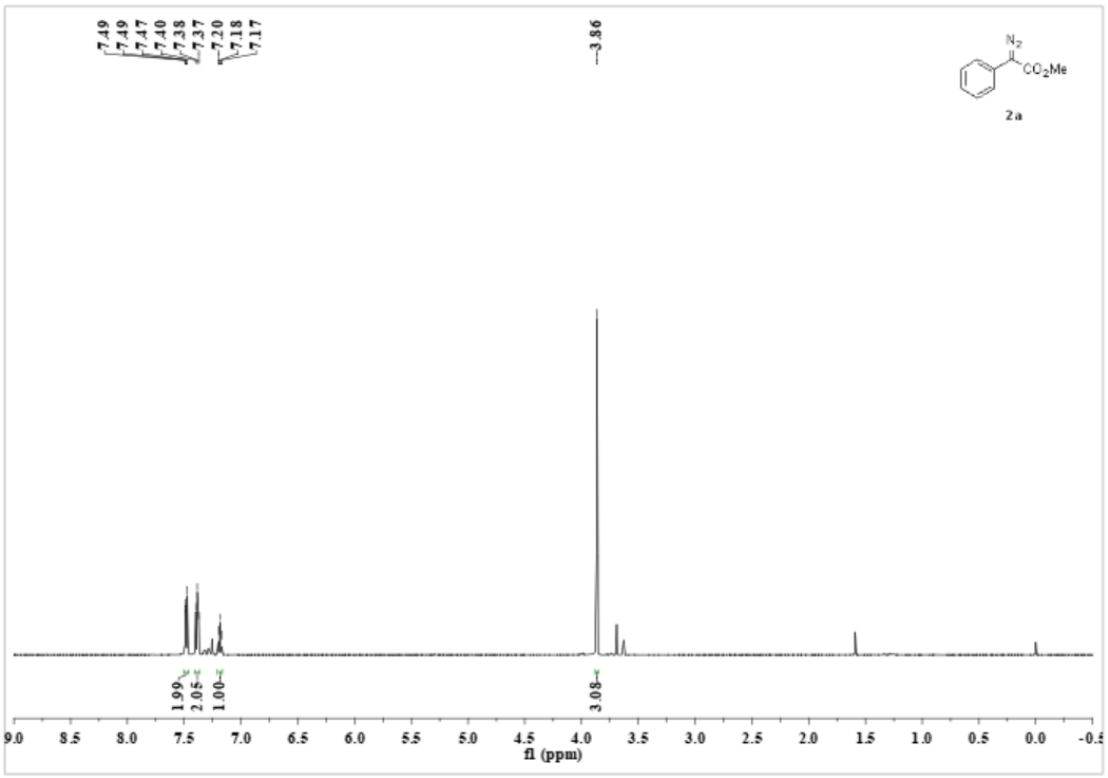


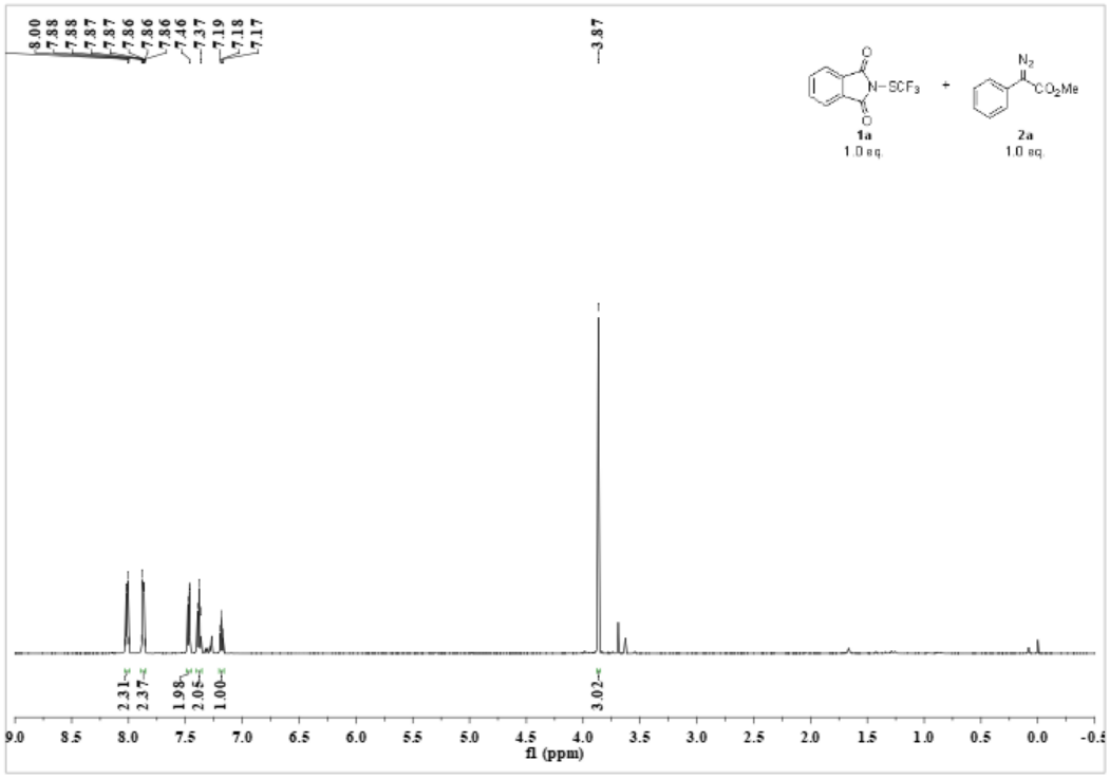
(I) NMR experiments

All the samples were dissolved in $CDCl_3$ and tested.

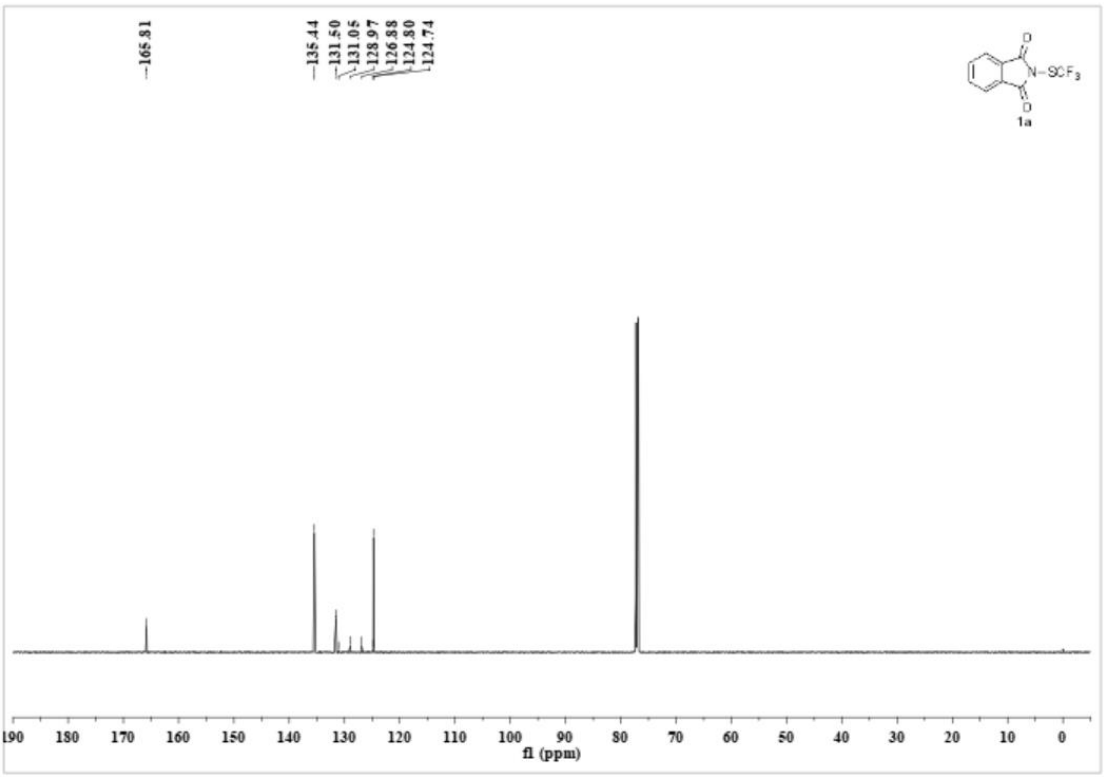
a) ^{1}H spectra of 1a, 2a and the mixture of 1a & 2a

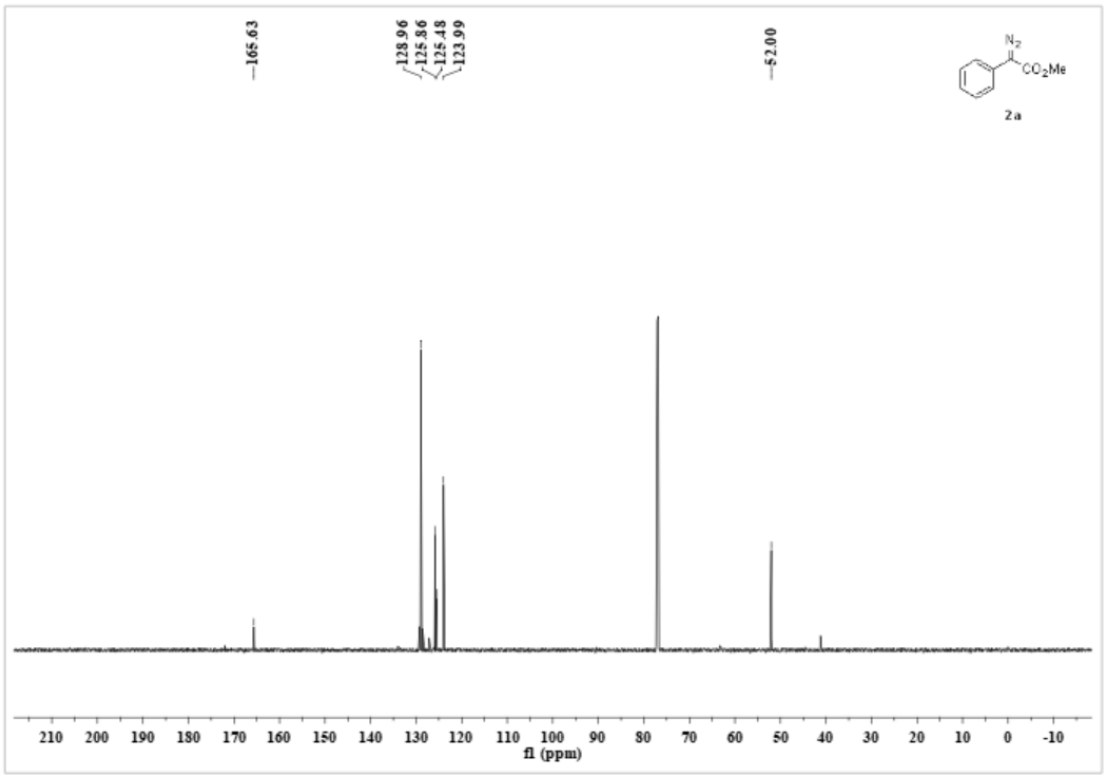


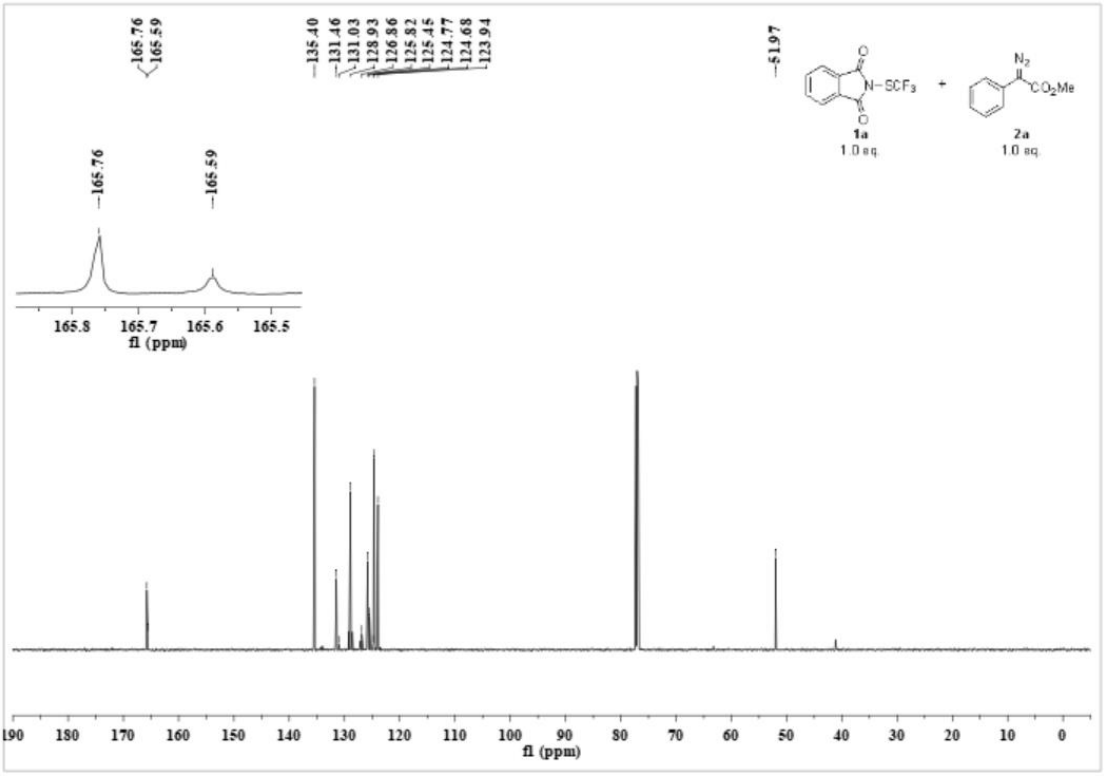

7.49
7.49
7.47
7.40
7.38
7.37
7.20
7.18
7.17
3.86
N2
CO2Me
2a
1.99
2.05
1.00
3.08
fl (ppm)


8.00
7.88
7.88
7.87
7.87
7.87
7.86
7.86
7.86
7.46
7.37
7.19
7.18
7.17
3.87
N-SCF3
1a
1.0 eq.
N2
CO2Me
2a
1.0 eq.
2.31
2.37
1.98
2.05
1.00
3.02
fl (ppm)

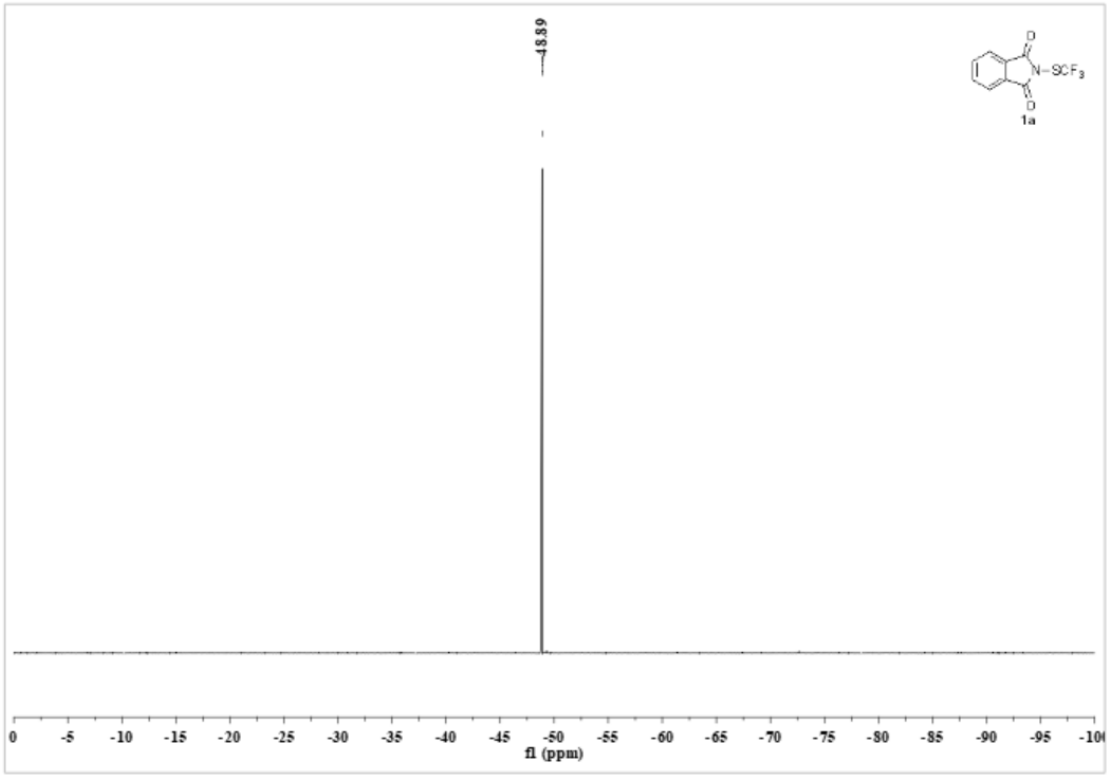
b) $^{13}C\{^{1}H\}$ spectra of 1a, 2a and the mixture of 1a & 2a

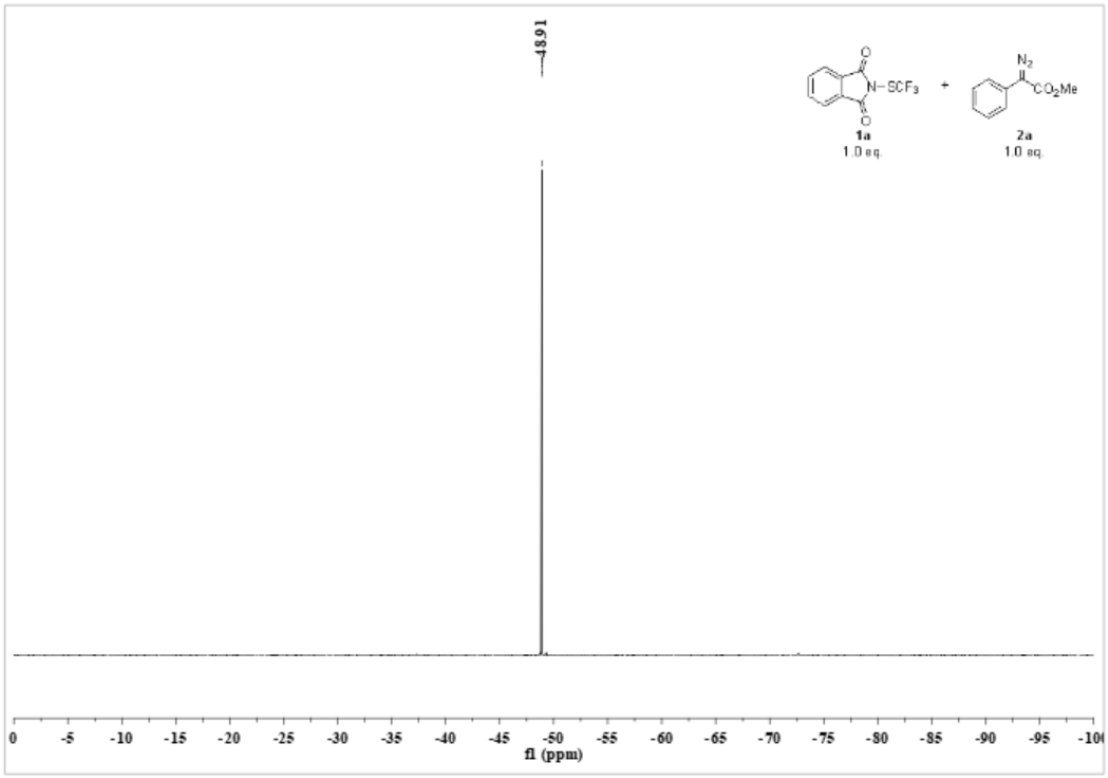






c) $^{19}F\{^{1}H\}$ spectra of 1a and the mixture of 1a & 2a



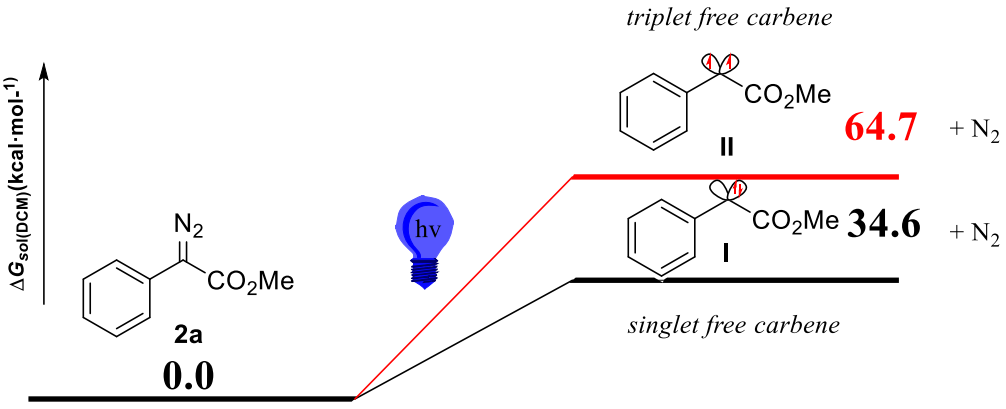


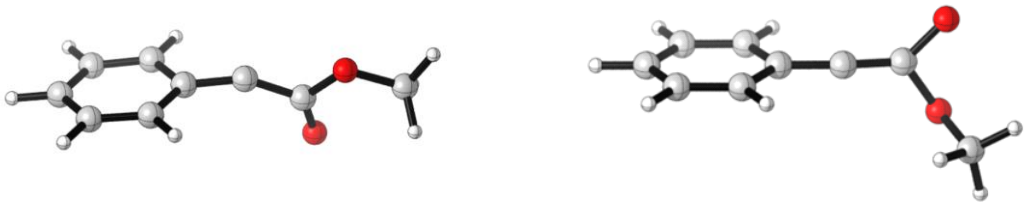
(J) DFT analysis

a) Computational details

Geometry optimizations were performed using Gaussian 16[3], employing the B3LYP functional[4] and 6-31G*[5] basis set. The geometry optimizations were run with dispersion corrections from Grimme's D3 model[6] with Becke-Johnson damping factors[7]. Harmonic vibrational frequencies calculations were performed to confirm the stationary points as true minima or transition states, as well as to provide thermodynamic corrections to the SCF energies. According to the excited state calculation of TDDFT method, it is found that the wavelength band of 455~465nm corresponds to the second excited state of two carbenes, so the energies of two carbenes are calculated by setting spin multiplicity 1 and 3 respectively with high accuracy scheme. Single point energy calculations were carried out using def2-TZVP[8] basis set, SMD solvation model[9] with dichloromethane as the solvent. The ball and stick structures were created using GaussView 6.1.1[10]. In addition, to correct the Gibbs free energies under pressure of 1 atm to the standard state in solution (1 mol/L), a correction of RT ln(cs/cg) (1.89 kcal/mol) is added to energies of all species, cs is the standard molar concentration in solution (1 mol/L), cg is the standard molar concentration in gas phase (0.0446 mol/L), and R is the gas constant. The 3D diagrams of molecules were generated using CYLView[11].

b) The energies of the excited singlet carbene I and triplet carbene II





singlet free carbene I

G = -312230.862419 kcal/mol
E = -312208.500487 kcal/mol

triplet free carbene II

G = -312200.745723 kcal/mol
E = -312177.199053kcal/mol

c) Energies and coordinates

Singlet carbene

C	-2.96064100	-1.15265200	-0.18526600
C	-1.58016400	-1.17250700	-0.33353100
C	-0.80366300	0.01861100	-0.17281800
C	-1.51168100	1.22782700	0.10126600
C	-2.89549900	1.23970400	0.19990800
C	-3.62400900	0.05181600	0.06741000
H	-3.53025300	-2.07323700	-0.28548000
H	-1.05601600	-2.09740800	-0.54931700
H	-0.93645100	2.13906700	0.22966900
H	-3.41510200	2.17238900	0.40384200
H	-4.70598300	0.06531800	0.16211700
C	0.58032400	0.00415400	-0.31943000
C	1.89815400	-0.11424700	0.17520500
O	2.85267900	0.36206000	-0.67328200
C	4.20304300	0.19017000	-0.22405600
H	4.82884000	0.42892700	-1.08508800
H	4.42789100	0.87228400	0.60252900
H	4.38352600	-0.83612900	0.10805200
O	2.16836700	-0.66562000	1.24347600

Thermal correction to Gibbs Free Energy = 0.108399

E(DCM) = -497.679844

Temperature	298.150	Kelvin
Pressure	1.00000	atm
Frequencies scaled by	1.0000	
Electronic Energy (EE)	-497.679844	Hartree
Zero-point Energy Correction	0.144035	Hartree
Thermal Correction to Energy	0.153563	Hartree
Thermal Correction to Enthalpy	0.154508	Hartree
Thermal Correction to Free Energy	0.108399	Hartree
EE + Zero-point Energy	-497.535809	Hartree
EE + Thermal Energy Correction	-497.526280	Hartree
EE + Thermal Enthalpy Correction	-497.525336	Hartree
EE + Thermal Free Energy Correction	-497.571445	Hartree
E (Thermal)	96.363	kcal/mol
Heat Capacity (Cv)	35.341	cal/mol-kelvin
Entropy (S)	97.044	cal/mol-kelvin

Triplet carbene

C 2.82872900 1.24531800 0.28028600
C 1.45374700 1.16747200 0.42705800
C 0.74948700 -0.04301300 0.12829800
C 1.52297400 -1.15995100 -0.32797300
C 2.89702000 -1.06111100 -0.47042900
C 3.56162400 0.13686700 -0.16884600
H 3.34242000 2.17388400 0.51522500
H 0.88374800 2.02326100 0.77504900
H 1.00572900 -2.08577700 -0.55920300
H 3.46346800 -1.92105700 -0.81823200
H 4.63971300 0.20571900 -0.28237900
C -0.61960100 -0.13057200 0.27529300
C -1.89501100 -0.42244900 0.31962600
O -2.78718700 -0.20923900 -0.75959900
C -3.54003100 1.01346100 -0.69279800
H -3.87679200 1.19816000 -1.71595900
H -4.40754900 0.90497700 -0.03685000
H -2.90409200 1.83604300 -0.35187600
O -2.70034700 -0.89217800 1.24099100

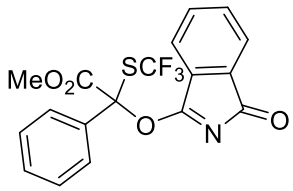
Thermal correction to Gibbs Free Energy = 0.106166

E(DCM) = -497.629617

Temperature	298.150	Kelvin
Pressure	1.00000	atm
Frequencies scaled by	1.0000	
Electronic Energy (EE)	-497.629617	Hartree
Zero-point Energy Correction	0.143690	Hartree
Thermal Correction to Energy	0.153981	Hartree
Thermal Correction to Enthalpy	0.154926	Hartree
Thermal Correction to Free Energy	0.106166	Hartree
EE + Zero-point Energy	-497.485927	Hartree
EE + Thermal Energy Correction	-497.475636	Hartree
EE + Thermal Enthalpy Correction	-497.474691	Hartree
EE + Thermal Free Energy Correction	-497.523451	Hartree
E (Thermal)	96.625	kcal/mol
Heat Capacity (Cv)	37.861	cal/mol-kelvin
Entropy (S)	102.623	cal/mol-kelvin

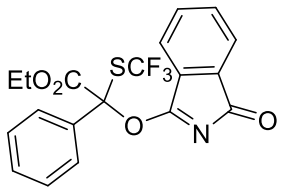
(K)Spectral characterization data for the products

Methyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3aa)



following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3aa** (76% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, J = 8.0 Hz, 2H), 7.83 – 7.78 (m, 1H), 7.75 – 7.70 (m, 1H), 7.69 – 7.62 (m, 2H), 7.54 – 7.43 (m, 3H), 3.78 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.6, 179.5, 165.8, 135.2, 135.0, 134.6, 133.54, 133.51, 129.9, 128.91 (q, J = 311.1 Hz), 128.87, 125.7, 124.6, 121.0, 93.4 (q, J = 2.0 Hz), 54.5. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.3. HRMS (FTMS-ESI) calcd for $C_{18}H_{12}F_3NNaO_4S^+$ ([M+Na^+]): 418.0331, found: 418.0331.

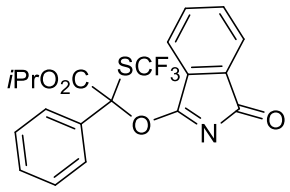
Ethyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3ab)



following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ab** (72% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 8.0 Hz, 2H), 7.84 – 7.78 (m, 1H), 7.76 – 7.70 (m, 1H), 7.69 – 7.62 (m, 2H), 7.53 – 7.42 (m, 3H), 4.36 – 4.17 (m, 2H), 1.19 (t, J = 8.0 Hz, 3H). ^{13}C{^{1}H}

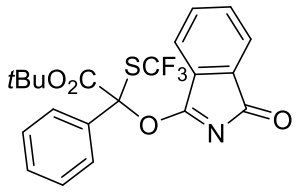
NMR (101 MHz, $CDCl_3$) δ 182.5, 179.5, 165.1, 135.4, 135.25, 135.19, 134.6, 133.5, 129.8, 129.0 (q, *J* = 311.1 Hz), 128.8, 125.7, 124.5, 121.0, 93.5 (q, *J* = 2.0 Hz), 64.1, 13.6. $^{19}F\{^1H\}$ NMR (376 MHz, $CDCl_3$) δ -37.3. HRMS (FTMS-ESI) calcd for $C_{19}H_{14}F_3NNaO_4S^+$ ([M+Na^+]): 432.0488, found: 432.0487.

Isopropyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3ac)



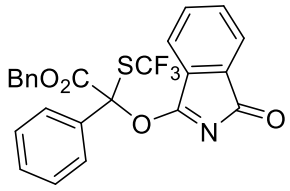
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 15:1) at -20 °C to afford **3ac** (71% yield) as a colorless gum. ^1H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.83 – 7.78 (m, 1H), 7.76 – 7.70 (m, 1H), 7.69 – 7.62 (m, 2H), 7.53 – 7.41 (m, 3H), 5.15 – 5.04 (m, 1H), 1.19 (d, *J* = 8.0 Hz, 3H), 1.16 (d, *J* = 8.0 Hz, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 182.4, 179.4, 164.4, 135.4, 135.2, 134.6, 133.4, 129.7, 129.0 (q, *J* = 311.1 Hz), 128.7, 125.6, 124.5, 121.0, 93.6 (d, *J* = 1.0 Hz), 72.6, 21.2, 21.1. $^{19}F\{^1H\}$ NMR (376 MHz, $CDCl_3$) δ -37.4. HRMS (FTMS-ESI) calcd for $C_{20}H_{16}F_3NNaO_4S^+$ ([M+Na^+]): 446.0644, found: 446.0636.

***tert*-Butyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3ad)**



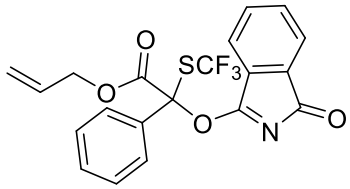
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 15:1) at -20 °C to afford **3ad** (36% yield) as a colorless gum. ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.0 Hz, 2H), 7.83 – 7.78 (m, 1H), 7.74 – 7.69 (m, 1H), 7.68 – 7.61 (m, 2H), 7.52 – 7.41 (m, 3H), 1.41 (s, 9H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 182.4, 179.6, 163.5, 135.9, 135.3, 134.8, 133.39, 133.38, 129.6, 129.1 (q, *J* = 311.1 Hz), 128.7, 125.6, 124.4, 121.0, 93.8, 86.2, 27.5. $^{19}F\{^1H\}$ NMR (376 MHz, $CDCl_3$) δ -37.5. HRMS (FTMS-ESI) calcd for $C_{21}H_{18}F_3NNaO_4S^+$ ([M+Na^+]): 460.0801, found: 460.0788.

Benzyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3ae)



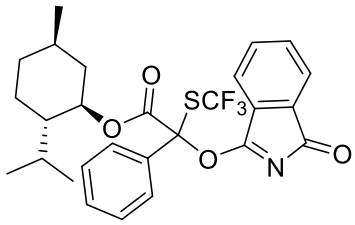
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ae** (74% yield) as a colorless gum. ^1H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.83 (m, 2H), 7.83 – 7.78 (m, 1H), 7.75 – 7.69 (m, 1H), 7.69 – 7.62 (m, 2H), 7.54 – 7.42 (m, 3H), 7.30 – 7.21 (m, 3H), 7.14 (d, *J* = 8.0 Hz, 2H), 5.22 (dd, *J* = 16.0, 12.0 Hz, 2H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 182.4, 179.3, 165.0, 135.4, 135.0 (d, *J* = 9.1 Hz), 134.6, 134.0, 133.5 (d, *J* = 4.0 Hz), 129.9, 129.0 (q, *J* = 311.1 Hz), 128.8, 128.6, 128.5, 128.2, 125.8, 124.7, 124.5, 121.0, 93.6 (d, *J* = 2.0 Hz), 69.3. $^{19}F\{^1H\}$ NMR (376 MHz, $CDCl_3$) δ -37.2. HRMS (FTMS-ESI) calcd for $C_{24}H_{17}F_3NO_4S^+$ ([M+H^+]): 472.0825, found: 472.0814.

Allyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3af)



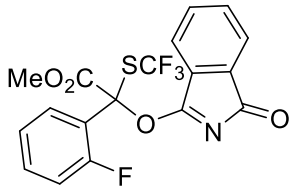
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3af** (58% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.0 Hz, 2H), 7.84 – 7.78 (m, 1H), 7.76 – 7.70 (m, 1H), 7.69 – 7.62 (m, 2H), 7.55 – 7.42 (m, 3H), 5.86 – 5.68 (m, 1H), 5.21 – 5.10 (m, 2H), 4.68 (d, *J* = 4.0 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.5, 179.4, 164.9, 135.4, 135.1 (d, *J* = 6.1 Hz), 134.6, 133.5 (d, *J* = 3.0 Hz), 130.3, 129.9, 128.9 (q, *J* = 311.1 Hz), 128.8, 125.8, 124.7, 124.5, 121.0, 119.7, 93.5 (q, *J* = 2.0 Hz), 68.2. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.3. HRMS (FTMS-ESI) calcd for $C_{20}H_{15}F_3NO_4S^+$ ([M+H$^+$]): 422.0668, found: 422.0661.

(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3ag)



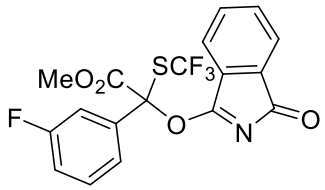
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 15:1) at -20 °C to afford **3ag** (73% yield, 1:1 dr) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 – 7.83 (m, 4H), 7.83 – 7.77 (m, 2H), 7.76 – 7.70 (m, 2H), 7.69 – 7.61 (m, 4H), 7.53 – 7.40 (m, 6H), 4.78 – 4.70 (m, 1H) [4.70 – 4.62 (m, 1H)], 2.02 (d, *J* = 12.0 Hz, 1H) [1.94 (d, *J* = 12.0 Hz, 1H)], 1.63 – 1.52 (m, 5H), 1.45 – 1.36 (m, 2H), 1.35 – 1.18 (m, 3H), 1.18 – 1.08 (m, 1H), 0.96 (d, *J* = 8.0 Hz, 1H), 0.94 – 0.86 (m, 1H), 0.84 (d, *J* = 8.0 Hz, 6H), 0.78 (d, *J* = 12.0 Hz, 1H), 0.76 – 0.68 (m, 2H), 0.65 (d, *J* = 8.0 Hz, 3H), 0.61 (d, *J* = 8.0 Hz, 3H) [0.59 (d, *J* = 8.0 Hz, 3H), 0.44 (d, *J* = 4.0 Hz, 3H)]. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.4 [182.2], 179.3 [179.1], 164.6, 135.57 [135.56], 135.20 [135.17], 134.72 [134.70], 133.43, 133.40, 129.7 [129.6], 129.10 (q, *J* = 310.1 Hz) [129.06 (q, *J* = 310.1 Hz)], 128.7 [128.6], 125.7 [125.6], 124.5 [124.4], 121.00 [120.96], 93.9 [93.8], 79.2 [78.5], 47.0 [46.6], 39.6 [39.0], 34.0 [33.9], 31.32 [31.26], 25.8 [25.3], 23.0 [20.6], 22.8 [20.4], 21.84 [21.81], 15.7 [15.4]. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.26 [-37.30]. HRMS (FTMS-ESI) calcd for $C_{27}H_{28}F_3NNaO_4S^+$ ([M+Na$^+$]): 542.1583, found: 542.1589. (Note: **3ag** is a mixture of two inseparable diastereomers, so there are two sets of peaks on ^{1}H, ^{13}C and ^{19}F spectra. On the ^{1}H spectrum, most of the peaks of the two diastereomers could not be well distinguished, which were integrated together, except for the methyl of isopropyl group and the hydrogen of the tertiary carbon linked to the oxygen atom, so the total number of hydrogen doubled. For ^{1}H, ^{13}C and ^{19}F spectra, the peaks in the square brackets belong to the diastereomer.)

Methyl 2-(2-fluorophenyl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3ah)



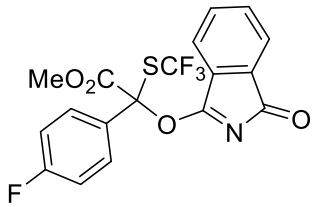
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ah** (39% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (td, *J* = 7.6, 1.6 Hz, 1H), 7.90 – 7.85 (m, 1H), 7.81 – 7.73 (m, 1H), 7.69 – 7.60 (m, 2H), 7.31 (td, *J* = 7.6, 0.8 Hz, 2H), 7.21 – 7.13 (m, 2H), 3.97 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 183.8, 167.8, 164.4, 162.8 (d, *J* = 258.6 Hz), 136.9, 136.8, 134.4, 132.6, 130.9 (d, *J* = 1.4 Hz), 129.1 (q, *J* = 293.9 Hz), 124.9 (d, *J* = 4.0 Hz), 123.6, 121.8 (d, *J* = 11.1 Hz), 116.6 (d, *J* = 21.2 Hz), 93.1, 53.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ -31.1, -111.6. HRMS (FTMS-ESI) calcd for $C_{18}H_{11}F_4NNaO_4S^+$ ([M+Na^+]): 436.0237, found: 436.0243.

Methyl 2-(3-fluorophenyl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3ai)



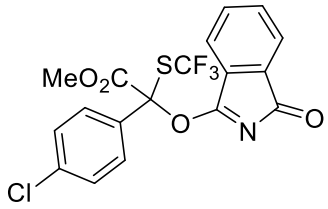
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ai** (<44% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.80 (m, 1H), 7.74 – 7.70 (m, 1H), 7.68 – 7.62 (m, 3H), 7.61 – 7.56 (m, 1H), 7.51 – 7.44 (m, 1H), 7.16 (td, *J* = 12.0, 4.0 Hz, 1H), 3.80 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.4, 179.3, 165.4, 162.8 (d, *J* = 248.5 Hz), 135.0, 134.6, 134.4, 133.6 (d, *J* = 6.1 Hz), 130.6 (d, *J* = 8.1 Hz), 128.8 (q, *J* = 311.1 Hz), 126.1 (d, *J* = 4.0 Hz), 124.7, 123.6, 121.0, 117.0 (d, *J* = 21.2 Hz), 113.2 (d, *J* = 25.2 Hz), 93.6 (d, *J* = 3.0 Hz), 54.7. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.3, -110.9. HRMS (FTMS-ESI) calcd for $C_{18}H_{11}F_4NNaO_4S^+$ ([M+Na^+]): 436.0237, found: 436.0239. (Note: According to the NMR, there should be a small amount of impurity in the product **3ai**, which was hard to be separated and identified.)

Methyl 2-(4-fluorophenyl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3aj)



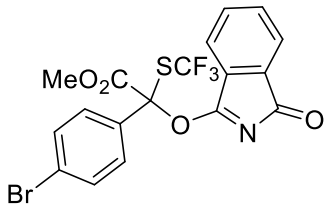
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3aj** (40% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.79 (m, 3H), 7.72 – 7.63 (m, 3H), 7.22 – 7.14 (m, 2H), 3.79 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.5, 179.3, 165.6, 163.4 (d, *J* = 251.5 Hz), 135.0, 134.6, 133.63, 133.55, 130.9 (d, *J* = 3.0 Hz), 128.9 (q, *J* = 311.1 Hz), 128.0 (d, *J* = 9.1 Hz), 124.7, 121.0, 116.0 (d, *J* = 22.2 Hz), 93.1, 54.6. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.3, -110.8. HRMS (FTMS-ESI) calcd for $C_{18}H_{11}F_4NNaO_4S^+$ ([M+Na^+]): 436.0237, found: 436.0243.

Methyl 2-(4-chlorophenyl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3ak)



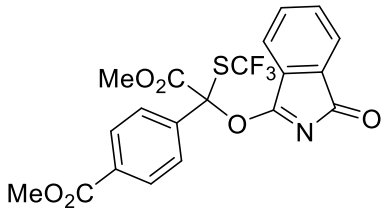
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ak** (79% yield) as a white solid, mp: 136 – 138 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.77 (m, 3H), 7.72 – 7.64 (m, 3H), 7.50 – 7.44 (m, 2H), 3.78 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.4, 179.3, 165.5, 136.2, 135.0, 134.6, 133.7, 133.64, 133.57, 129.2, 128.8 (q, J = 311.1 Hz), 127.2, 124.7, 121.0, 93.0 (d, J = 2.0 Hz), 54.6. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.2. HRMS (FTMS-ESI) calcd for $C_{18}H_{11}ClF_3NNaO_4S^+$ ([M+Na$^+$]): 451.9942, 453.9912, found: 451.9946, 453.9913.

Methyl 2-(4-bromophenyl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3al)



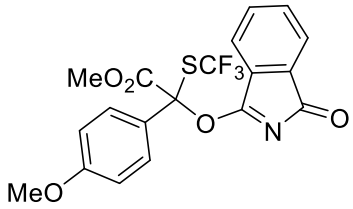
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3al** (71% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.79 (m, 1H), 7.76 – 7.72 (m, 2H), 7.72 – 7.65 (m, 3H), 7.65 – 7.61 (m, 2H), 3.78 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.4, 179.2, 165.4, 135.0, 134.6, 134.3, 133.64, 133.57, 132.1, 128.8 (q, J = 311.1 Hz), 127.5, 124.7, 124.4, 121.0, 93.0 (d, J = 1.0 Hz), 54.6. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.2. HRMS (FTMS-ESI) calcd for $C_{18}H_{11}BrF_3NNaO_4S^+$ ([M+Na$^+$]): 495.9436, 497.9416, found: 495.9443, 497.9421.

Methyl 2-(4-acetoxyphenyl)-2-((1-oxo-1H-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3am)



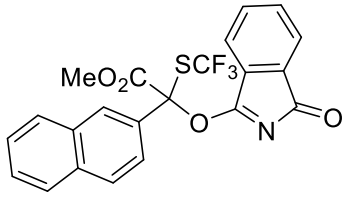
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 6:1) at -20 °C to afford **3am** (<38% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, J = 8.0 Hz, 2H), 7.95 (d, J = 8.0 Hz, 2H), 7.86 – 7.80 (m, 1H), 7.76 – 7.71 (m, 1H), 7.71 – 7.63 (m, 2H), 3.96 (s, 3H), 3.79 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 182.4, 179.2, 166.2, 165.3, 139.7, 134.9, 134.6, 133.7, 133.6, 131.6, 130.1, 128.4 (d, J = 246.4 Hz), 125.9, 124.7, 121.1, 93.0 (d, J = 1.0 Hz), 54.7, 52.5. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.2. HRMS (FTMS-ESI) calcd for $C_{20}H_{14}F_3NNaO_6S^+$ ([M+Na$^+$]): 476.0386, found: 476.0384.

Methyl 2-(4-methoxyphenyl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3an)



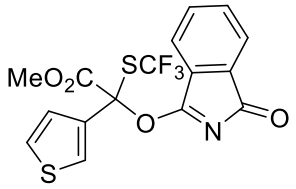
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 5:1) at -20 °C to afford **3an** (22% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.85 (m, 2H), 7.81 – 7.77 (m, 2H), 7.74 (d, *J* = 8.0 Hz, 2H), 6.92 (d, *J* = 8.0 Hz, 2H), 3.84 (s, 3H), 3.77 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 189.1, 181.1, 166.2, 160.4, 134.8, 131.45, 131.42, 130.78, 129.8, 129.0 (d, *J* = 341.4 Hz), 123.93, 123.90, 123.6, 113.5, 91.7, 55.33, 54.32. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -37.9. HRMS (FTMS-ESI) calcd for $C_{19}H_{14}F_3NNaO_5S^+$ ([M+Na$^+$]): 448.0437, found: 448.0444.

Methyl 2-(naphthalen-2-yl)-2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-((trifluoromethyl)thio)acetate (3ao)



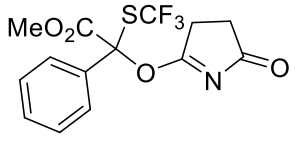
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3an** (60% yield) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.57 (s, 1H), 8.06 (d, *J* = 12.0 Hz, 1H), 7.99 (d, *J* = 8.0 Hz, 1H), 7.96 – 7.89 (m, 2H), 7.88 – 7.86 (m, 2H), 7.79 – 7.73 (m, 2H), 7.66 (t, *J* = 8.0 Hz, 1H), 7.59 (t, *J* = 8.0 Hz, 1H), 4.04 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 186.0, 167.9, 164.2, 136.4, 134.4, 133.6, 132.7, 132.3, 130.1, 129.8, 129.6, 129.3 (d, *J* = 309.1 Hz), 129.0, 128.0, 127.2, 124.0, 123.6, 90.5, 52.9. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -31.1. HRMS (FTMS-ESI) calcd for $C_{22}H_{14}F_3NNaO_4S^+$ ([M+Na$^+$]): 468.0488, found: 468.0490.

Methyl 2-((1-oxo-1*H*-isoindol-3-yl)oxy)-2-(thiophen-3-yl)-2-((trifluoromethyl)thio)acetate (3ap)



following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ao** (39% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (d, *J* = 1.2 Hz, 1H), 7.91 – 7.83 (m, 2H), 7.80 – 7.73 (m, 2H), 7.70 (d, *J* = 4.0 Hz, 1H), 7.39 – 7.34 (m, 1H), 3.96 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.7, 167.8, 162.7, 138.0, 137.6, 134.4, 132.7, 131.5 (d, *J* = 307.0 Hz), 127.9, 126.7, 123.6, 85.5, 53.0. ^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) δ -31.1. HRMS (FTMS-ESI) calcd for $C_{16}H_{10}F_3NNaO_4S_2^+$ ([M+Na$^+$]): 423.9896, found: 423.9903.

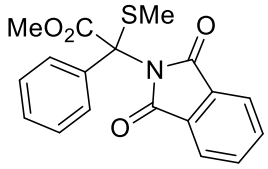
Methyl 2-((2-oxo-3,4-dihydro-2*H*-pyrrol-5-yl)oxy)-2-phenyl-2-((trifluoromethyl)thio)acetate (3ba)



following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 10:1) at -20 °C to afford **3ba** (75% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 – 7.73

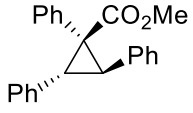
(m, 2H), 7.48 – 7.39 (m, 3H), 3.75 (s, 3H), 3.12 – 3.01 (m, 1H), 2.97 – 2.88 (m, 1H), 2.83 – 2.78 (m, 2H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 192.1, 189.8, 165.7, 135.0, 129.8, 129.0 (q, *J* = 311.1 Hz), 128.8, 125.7, 93.4, 54.4, 32.2, 29.6. $^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$) δ -37.5. HRMS (FTMS-ESI) calcd for $C_{14}H_{12}F_3NNaO_4S^+$ ([M+Na^+]): 370.0331, found: 370.0338.

Methyl 2-(1,3-dioxoisoindolin-2-yl)-2-(methylthio)-2-phenylacetate (5aa)



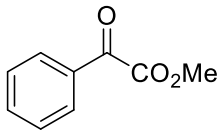
following the general procedure, the reaction mixture was purified by silica gel column chromatography (petroleum/EtOAc = 8:1) to afford **5aa** (60% yield) as a colorless gum. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.82 (m, 2H), 7.80 – 7.75 (m, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.42 – 7.31 (m, 3H), 3.79 (s, 3H), 2.10 (s, 3H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 167.7, 167.1, 134.5, 134.1, 131.4, 128.6, 128.22, 128.18, 123.7, 73.1, 53.6, 14.8. HRMS (FTMS-ESI) calcd for $C_{18}H_{16}NO_4S^+$ ([M+H^+]): 342.0795, found: 342.0786.

(2*R*,3*R*)/(2*S*,3*S*)-Methyl 1,2,3-triphenylcyclopropane-1-carboxylate (15)



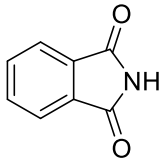
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.38 (t, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.25 – 7.18 (m, 5H), 7.16 – 7.09 (m, 3H), 6.97 (d, *J* = 8.0 Hz, 2H), 3.88 (d, *J* = 8.0 Hz, 1H), 3.57 (d, *J* = 8.0 Hz, 1H), 3.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 136.4, 136.3, 135.7, 131.3, 129.0, 128.3, 128.2, 128.0, 127.9, 127.2, 127.1, 126.4, 52.4, 46.0, 36.8, 34.8. This compound is known and the characterization is in consistence with the reported literature.[12]

Methyl 2-oxo-2-phenylacetate (16)



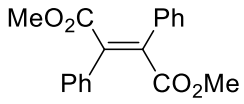
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.99 (m, 2H), 7.71 – 7.63 (m, 1H), 7.55 – 7.49 (m, 2H), 3.99 (s, 3H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 186.1, 164.1, 135.0, 132.4, 130.1, 128.9, 52.8. This compound is known and the characterization is in consistence with the reported literature.[13]

Isoindoline-1,3-dione (17)



^{1}H NMR (400 MHz, DMSO-d6) δ 11.29 (s, 1H), 7.77 (s, 4H). This compound is known and the characterization is in consistence with the reported literature.[14]

(*E*)-Dimethyl 2,3-diphenylfumarate



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.35 (m, 10H), 3.57 (s, 6H). This compound is known and the characterization is in consistence with the reported literature.[15]

(L) References

(1) For the synthesis of α-diazoester and α-diazoketone, see: (a) Li, W.; Wang, J.; Hu, X. L.; Shen, K.; Wang, W. T.; Chu, Y. Y.; Lin, L. L.; Liu, X. H.; Feng, X. M. Catalytic asymmetric Roskamp reaction of α-alkyl-α-diazoesters with aromatic aldehydes: Highly enantioselective synthesis of α-alkyl-β-keto esters. *J. Am. Chem. Soc*. **2010**, *132*, 8532−8533. (b) Tan, F.; Liu, X. H.; Hao, X. Y.; Tang, Y.; Lin, L. L.; Feng, X. M. Asymmetric catalytic insertion of α-diazo carbonyl compounds into O−H bonds of carboxylic acids. *ACS Catal*. **2016**, *6*, 6930−6934. (c) Xia, Z. L.; Hu, J. D.; Gao, Y.-Q.; Yao, Q. Z.; Xie, W. Q. Facile access to 2,2-disubstituted indolin-3-ones via a cascade Fischer indolization/Claisen rearrangement reaction. *Chem. Commun.* **2017**, *53*, 7485−7488. For the synthesis of 1-benzyl-3-diazoindolin-2-one, see: (d) Marti, C.; Carreira, E. M. Total synthesis of (−)-spirotryprostatin B: Synthesis and related studies. *J. Am. Chem. Soc.* **2005**, *127*, 11505–11515. For the synthesis of (1-diazo-2,2,2-trifluoroethyl)benzene, see: (e) Denton, J. R.; Sukumaran, D.; Davies, H. M. L. Enantioselective synthesis of trifluoromethyl-substituted cyclopropanes. *Org. Lett*. **2007**, *9*, 2625–2628. For the synthesis of 2-diazo-2-phenylacetonitrile, see: (f) Jiang, Z.; Niu, S.-L.; Zeng, Q.; Ouyang, Q.; Chen, Y.-C.; Xiao, Q. Selective alkynylallylation of the C−C bond of cyclopropenes. *Angew. Chem. Int. Ed*. **2021**, *60*, 297−303.

(2) For the synthesis of PhtOCF_3, see: (a) Yuan, W.-J.; Tong, C.-L.; Xu, X.-H.; Qing, F.-L. *N*-Triffuoromethoxyphthalimide: A shelf-stable reagent for nucleophilic triffuoromethoxylation. *J. Org. Chem*. **2023**, *88*, 4434−444. For the synthesis of PhtSMe, see: (b) Cesario, C.; Miller, M. J. Syntheses of carbocyclic uracil polyoxin C analogs: Application of Pd(0)/InI-allylation of 4-acetoxy-2-azetidinone. *J. Org. Chem.* **2009**, *74*, 5730–5733.

(3) Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Petersson, G. A.; Nakatsuji, H.; Li, X.; Caricato, M.; Marenich, A. V.; Bloino, J.; Janesko, B. G.; Gomperts, R.; Mennucci, B.; Hratchian, H. P.; Ortiz, J. V.; Izmaylov, A. F.; Sonnenberg, J. L.; Williams-Young, D.; Ding, F.; Lipparini, F.; Egidi, F.; Goings, J.; Peng, B.; Petrone, A.; Henderson, T.; Ranasinghe, D.; Zakrzewski, V. G.; Gao, J.; Rega, N.; Zheng, G.; Liang, W.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Throssell, K.; Montgomery, J. A., Jr.; Peralta, J. E.; Ogliaro, F.; Bearpark, M. J.; Heyd, J. J.; Brothers, E. N.; Kudin, K. N.; Staroverov, V. N.; Keith, T. A.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A. P.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Millam, J. M.; Klene, M.; Adamo, C.; Cammi, R.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Farkas, O.; Foresman, J. B.; Fox, D. J. *Gaussian 16, Rev. A.03*; Gaussian, Inc.: Wallingford, CT, 2016.

(4) Becke, A. D. *J. Chem. Phys*. **1993**, *98*, 5648–5652.

(5) Lee, C.; Yang, W.; Parr, R. G. *Phys. Rev. B: Condens. Matter Mater. Phys*. **1988**, *37*, 785–789.

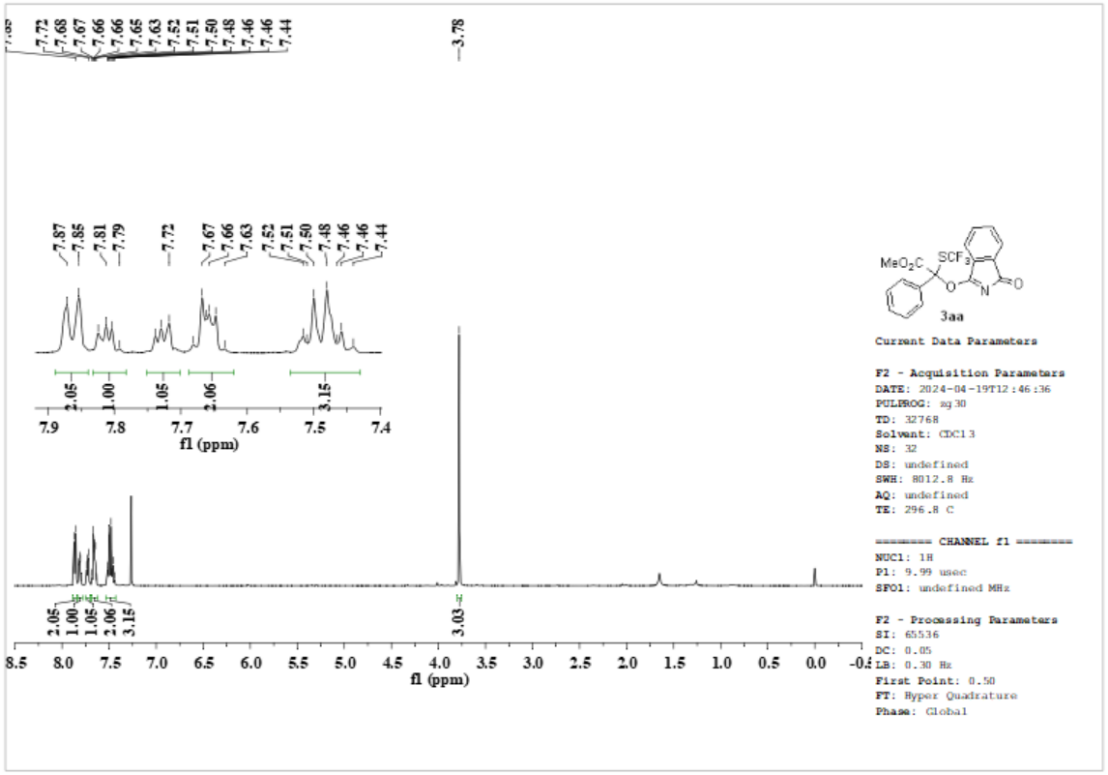
(6) Grimme, S.; Antony, J.; Ehrlich, S.; Krieg, H. *J. Chem. Phys*. **2010**, *132*, 154104.

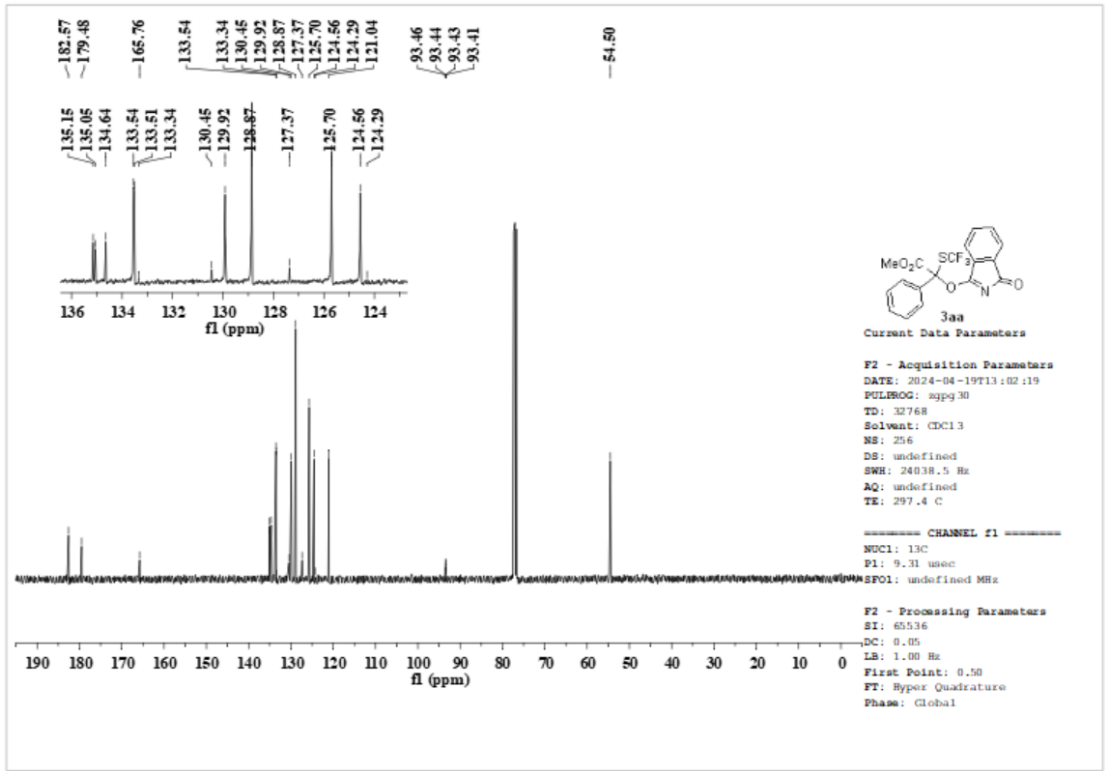
(7) Johnson, E. R.; Becke, A. D. *J. Chem. Phys*. **2006**, *124*, 174104.

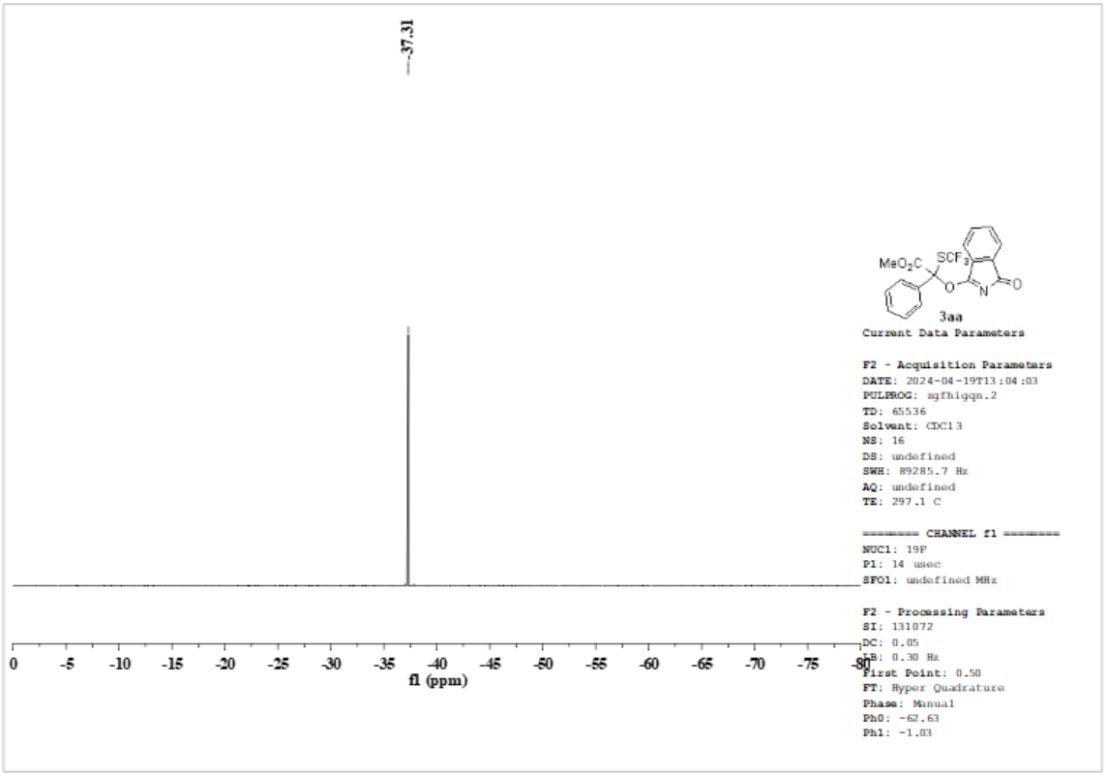
(8) Weigend, F. *Phys. Chem. Chem. Phys*. **2006**, *8*, 1057–1065.

(9) Marenich, A. V.; Cramer, C. J.; Truhlar, D. G. *J. Phys. Chem. B* **2009**, *113*, 6378–6396.
(10) Dennington, R.; Keith, T.; Millam, J. *GaussView, Version 6.1.1*; Semichem Inc.: Shawnee Mission, KS, 2019.
(11) Legault, C. Y. *CYLview, 1.0b*: Universitéde Sherbrooke, 2009 (http://www.cylview.org).
(12) Thompson, J. L.; Davies, H. M. L. *J. Am. Chem. Soc.* **2007**, *129*, 6090–6091.
(13) Bannon, R.; Morrison, G.; Smyth, M.; Moody, T. S.; Wharry, S.; Roth, P. M. C.; Gauron, G.; Baumann, M. Continuous flow approach for benzylic photo-oxidations using compressed air. *Org. Process Res. Dev*. **2024**, *28*, 3307−3312.
(14) Xin, X. Q.; Xiang, D. X.; Yang, J. M.; Zhang, Q.; Zhou, F. G.; Dong, D. W. Homogeneous and stereoselective copper(II)-catalyzed monohydration of methylenemalononitriles to 2-cyanoacrylamides. *J. Org. Chem.* **2013**, *78*, 11956−11961.
(15) Zhao, R.; Teng, J.; Wang, Z.; Dong, W.; Lin J.; Chu, C. Copper on charcoal: Cu^0 nanoparticle catalysed aerobic oxidation of α-diazo esters. *Org. Biomol. Chem*. **2021**, *19*, 6120–6126.

(M) Copies of NMR spectra for the products



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O
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LB: 0.30 Hz
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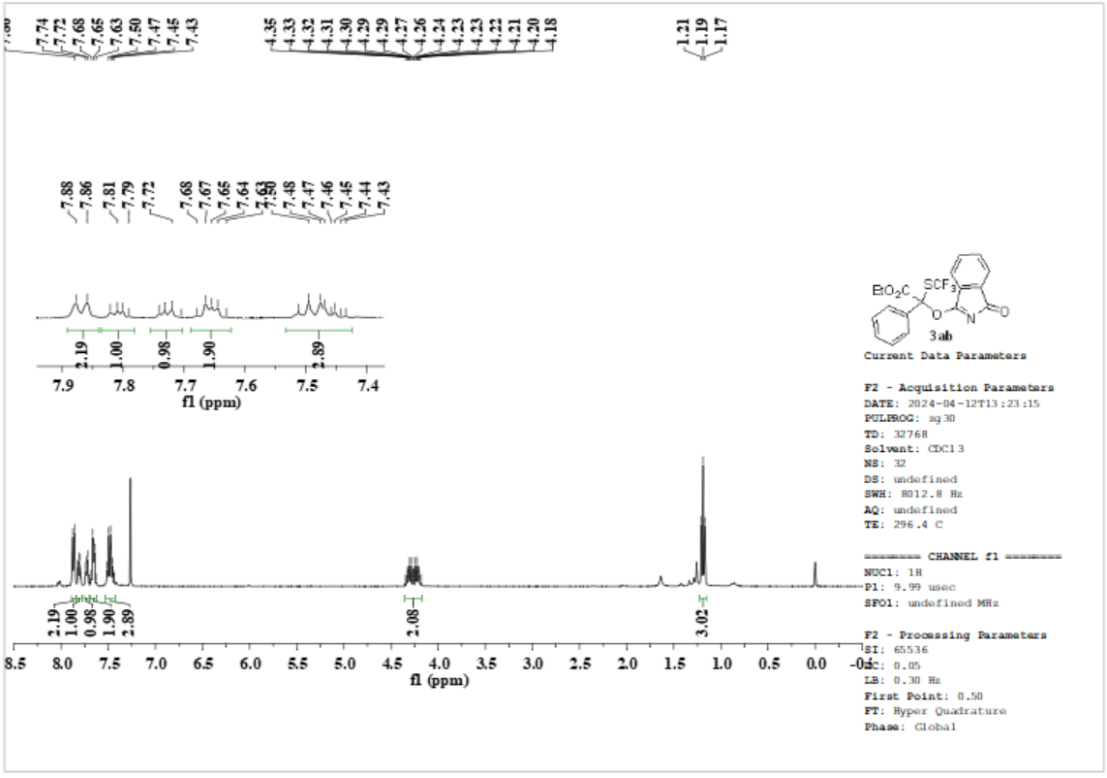
f1 (ppm)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4.35 4.33 4.32 4.31 4.30 4.29 4.29 4.27 4.26 4.24 4.23 4.23 4.22 4.21 4.20 4.18
1.21 1.19 1.17
7.88 7.86 7.81 7.79 7.72 7.68 7.67 7.65 7.64 7.63 7.60 7.48 7.47 7.46 7.45 7.44 7.43
2.19 1.00 0.98 1.90 2.89
7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
EtO2C
SCF3
O
N
O
3ab
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2.08
3.02
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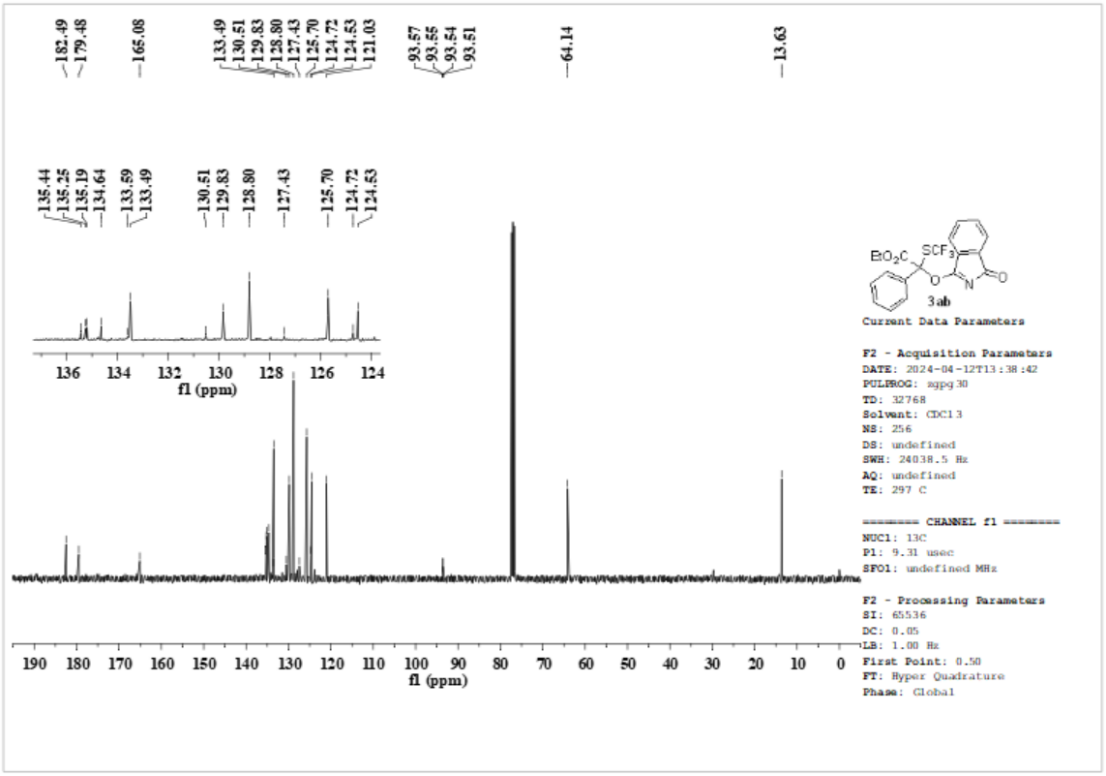
f1 (ppm)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93.55
93.54
93.51
64.14
13.63
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135.25
135.19
134.64
133.59
133.49
130.51
129.83
128.80
127.43
125.70
124.72
124.53
136 134 132 130 128 126 124
f1 (ppm)
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f1 (ppm)
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SCF3
O
N
O
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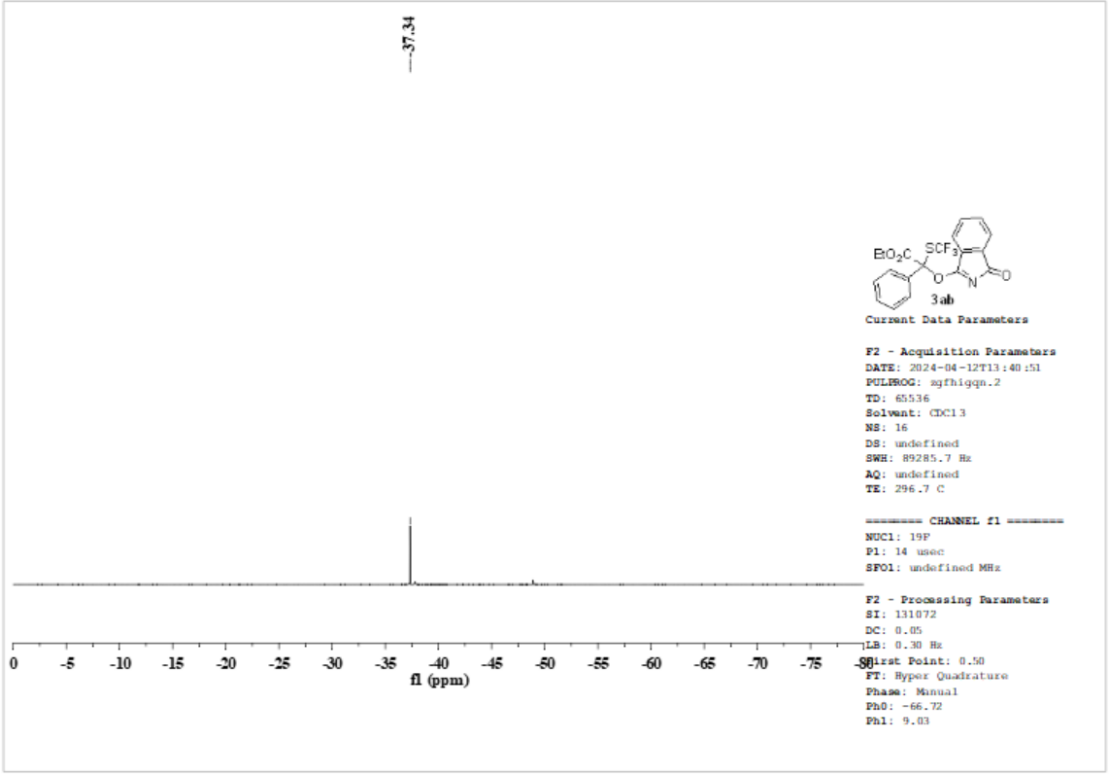
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SCF3
O
N
O
3ab
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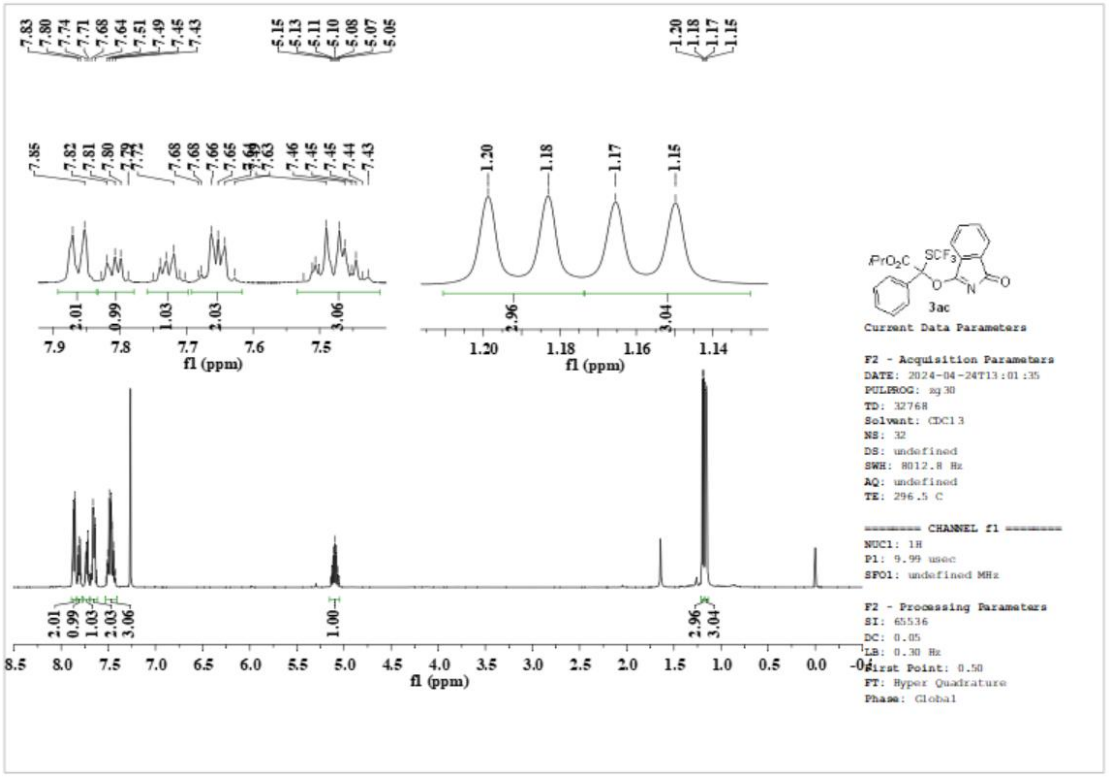
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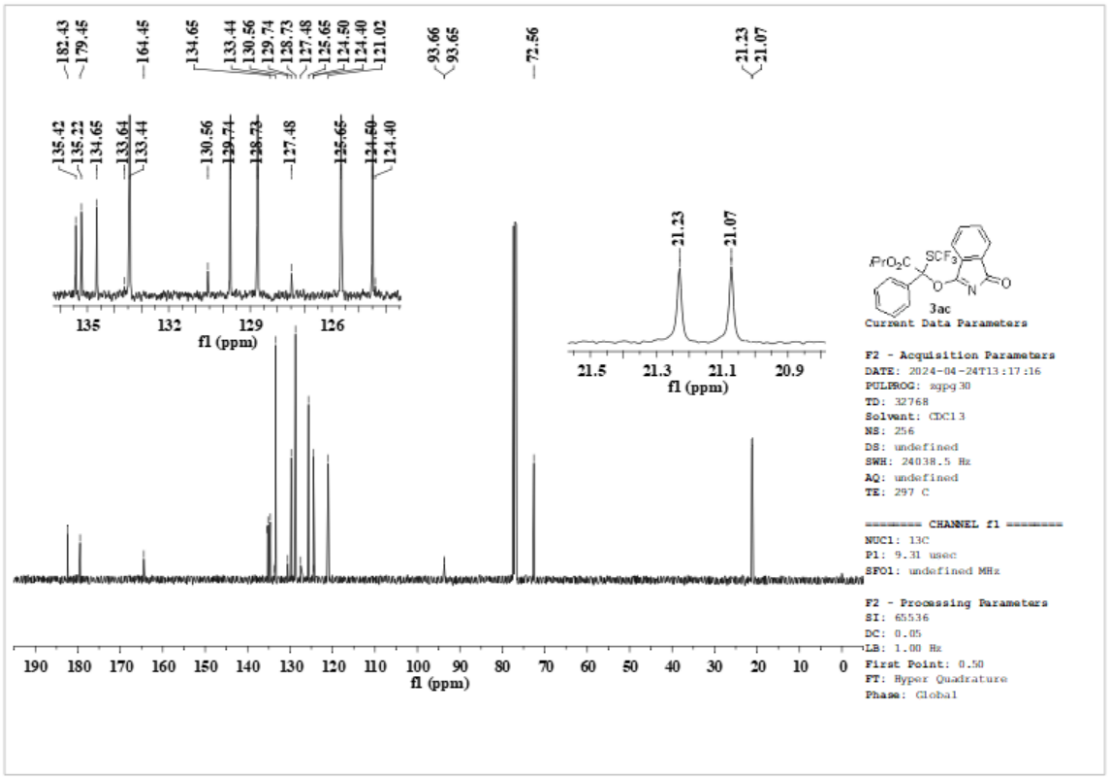
fl (ppm)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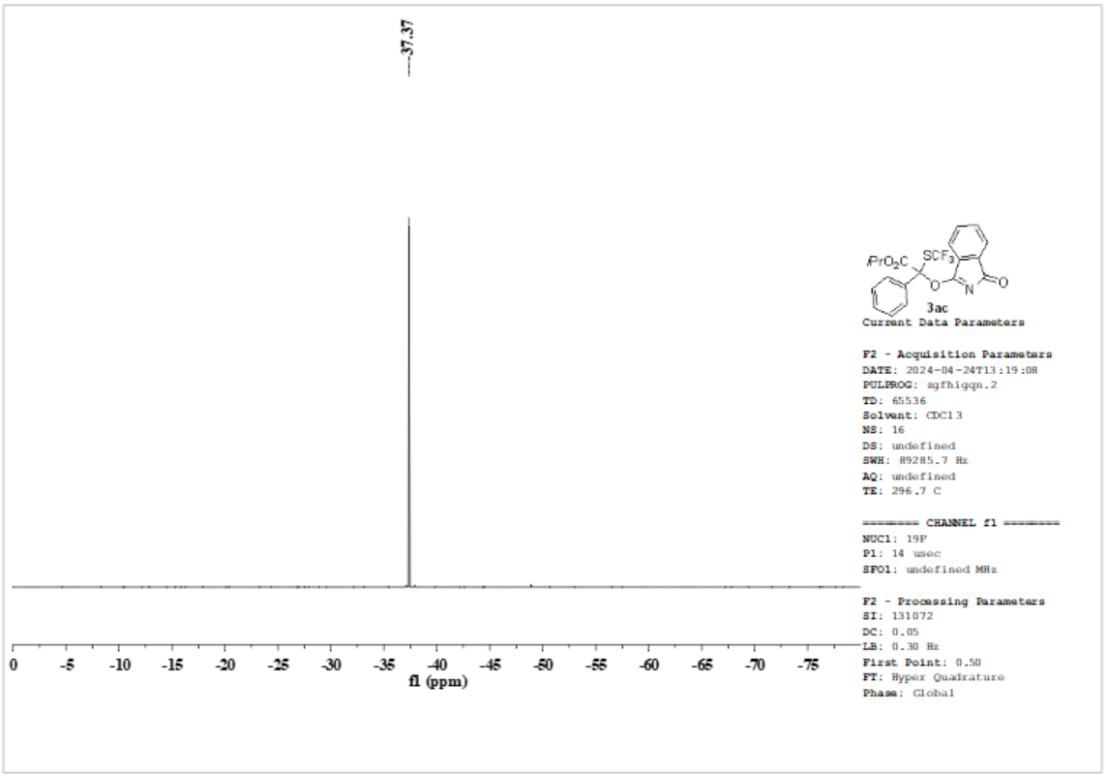
fl (ppm)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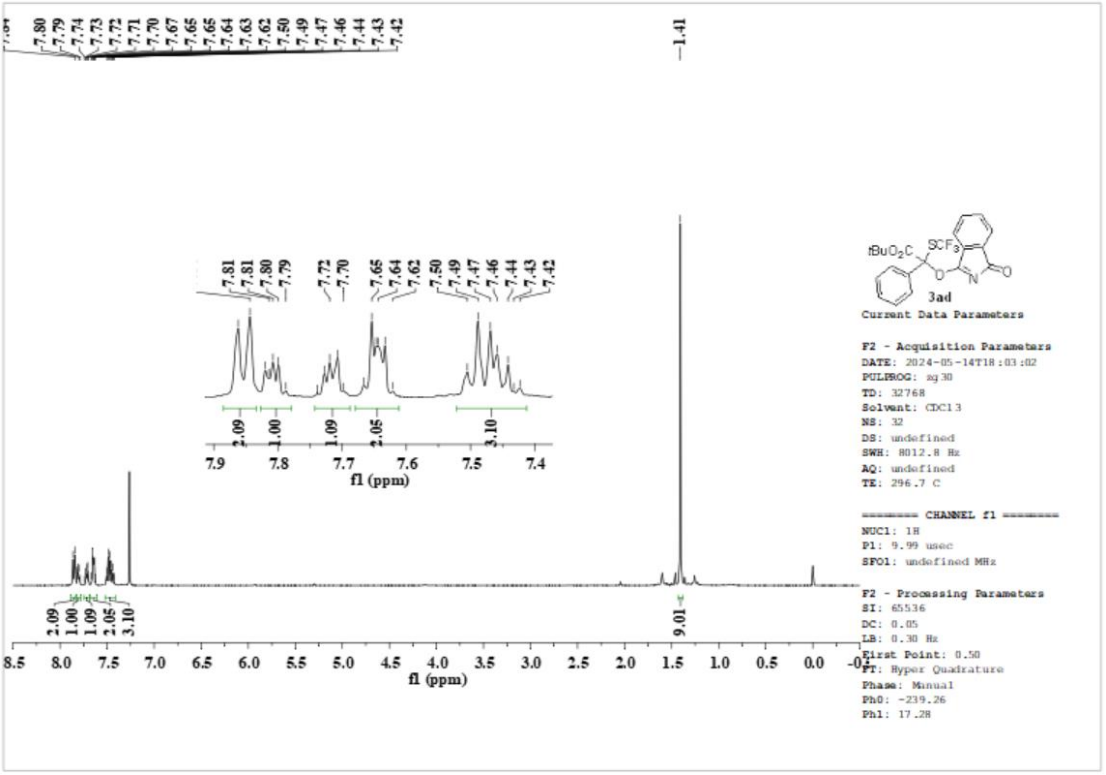
f1 (ppm)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3.10
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3.10
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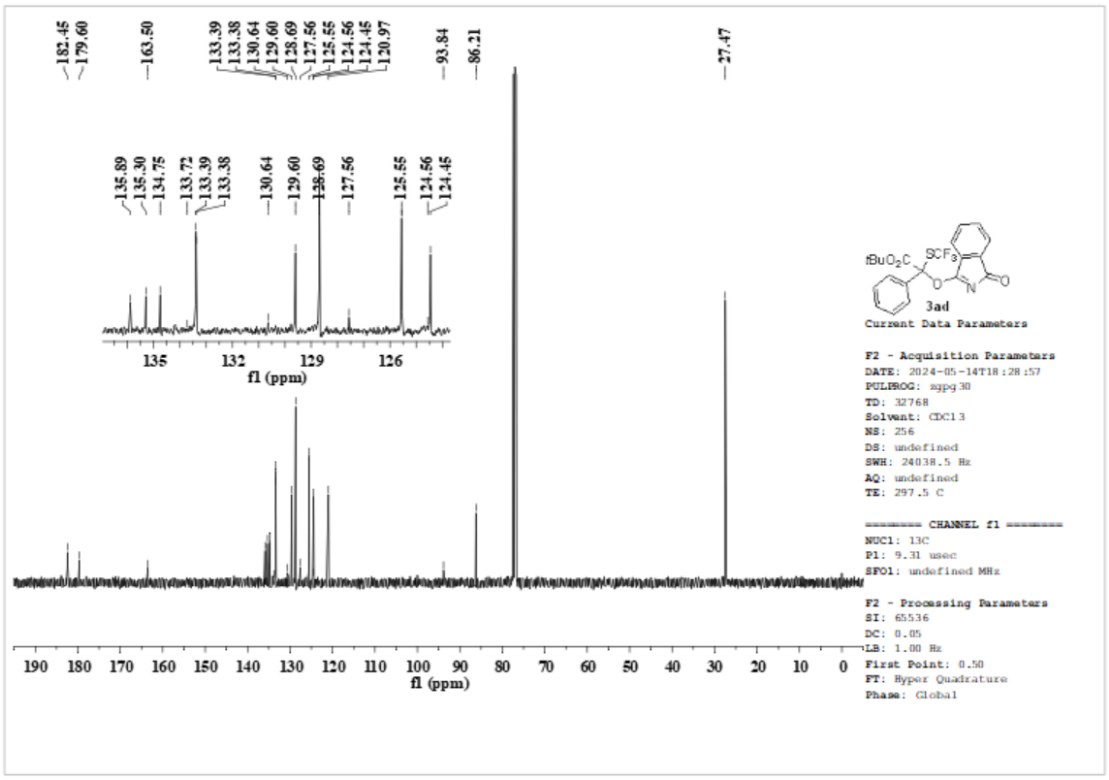
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f1 (ppm)
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N
O
3ad
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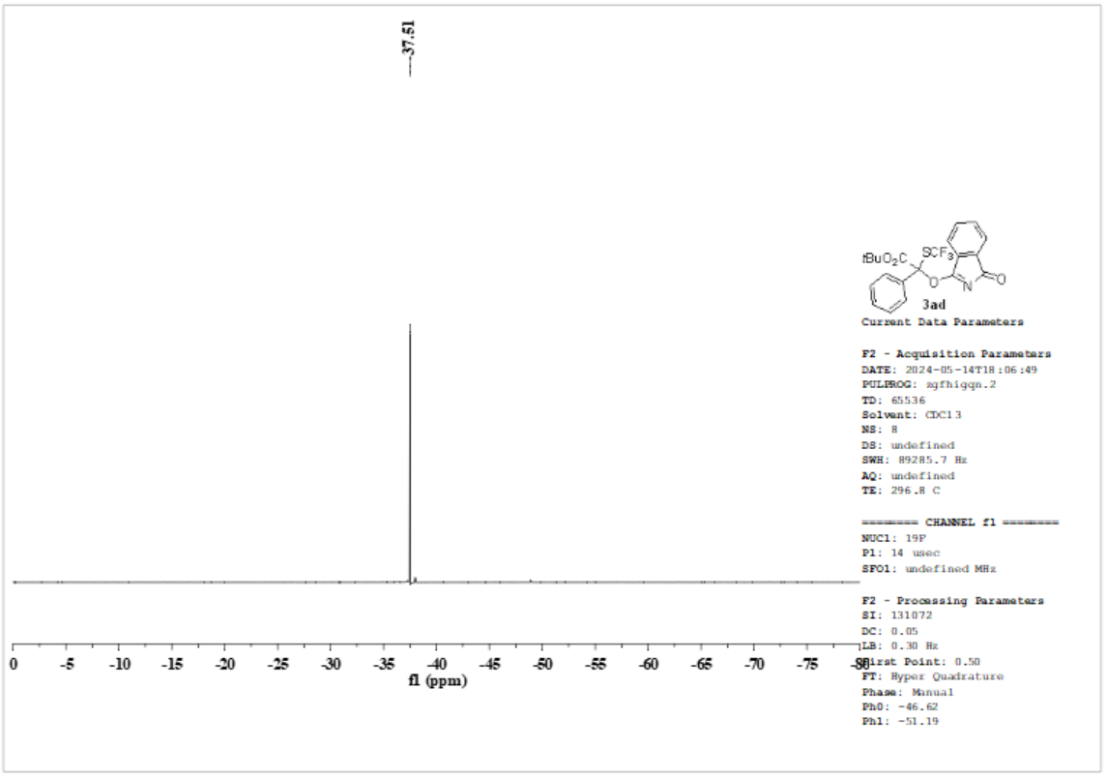
Phase: Global


37.51
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80
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SCF3
O
N
O
3ad
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SFO1: undefined MHz
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LB: 0.30 Hz
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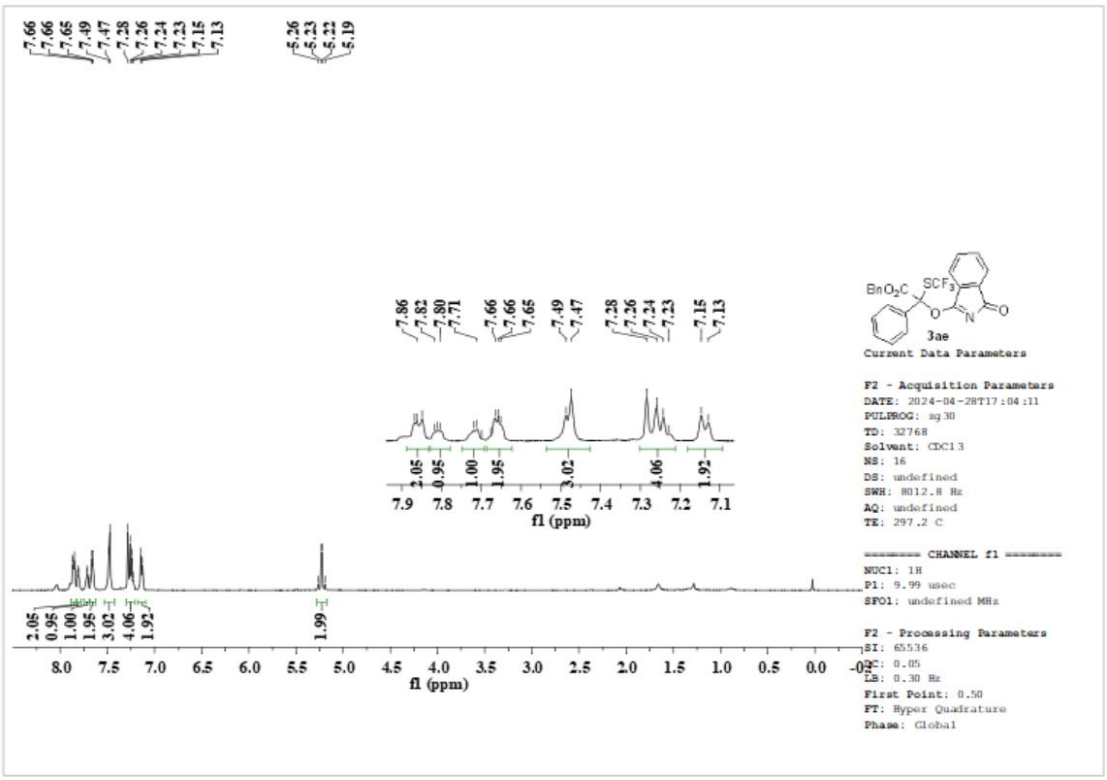
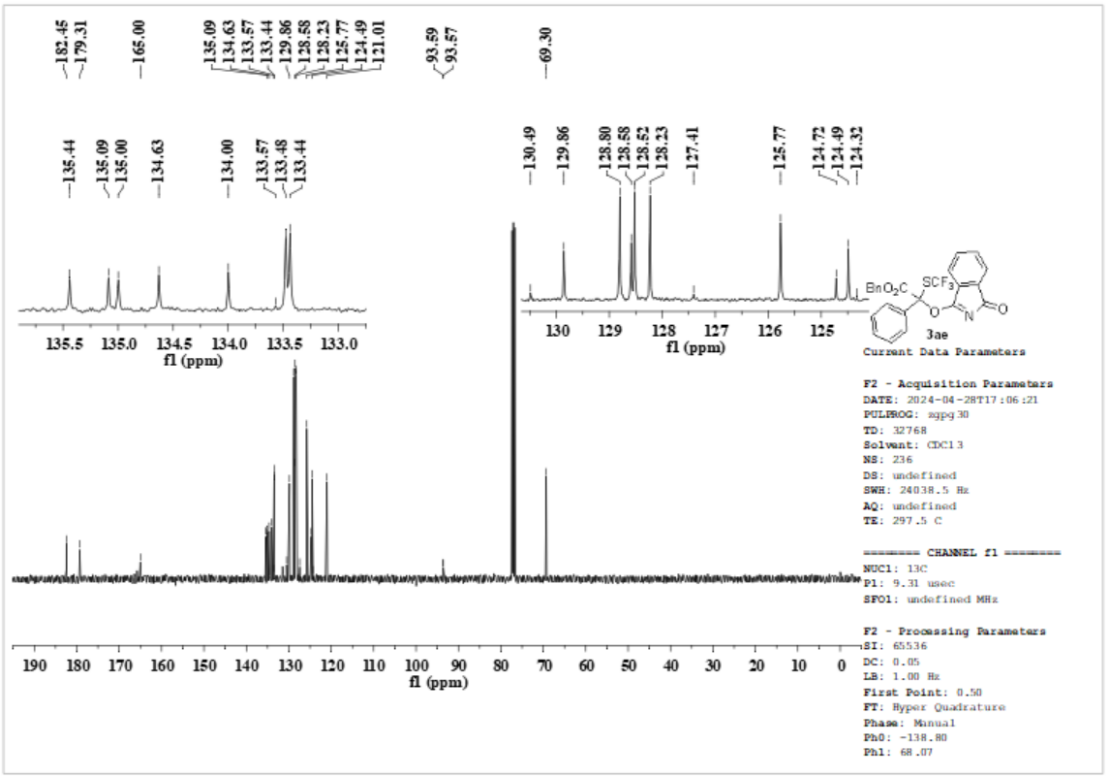
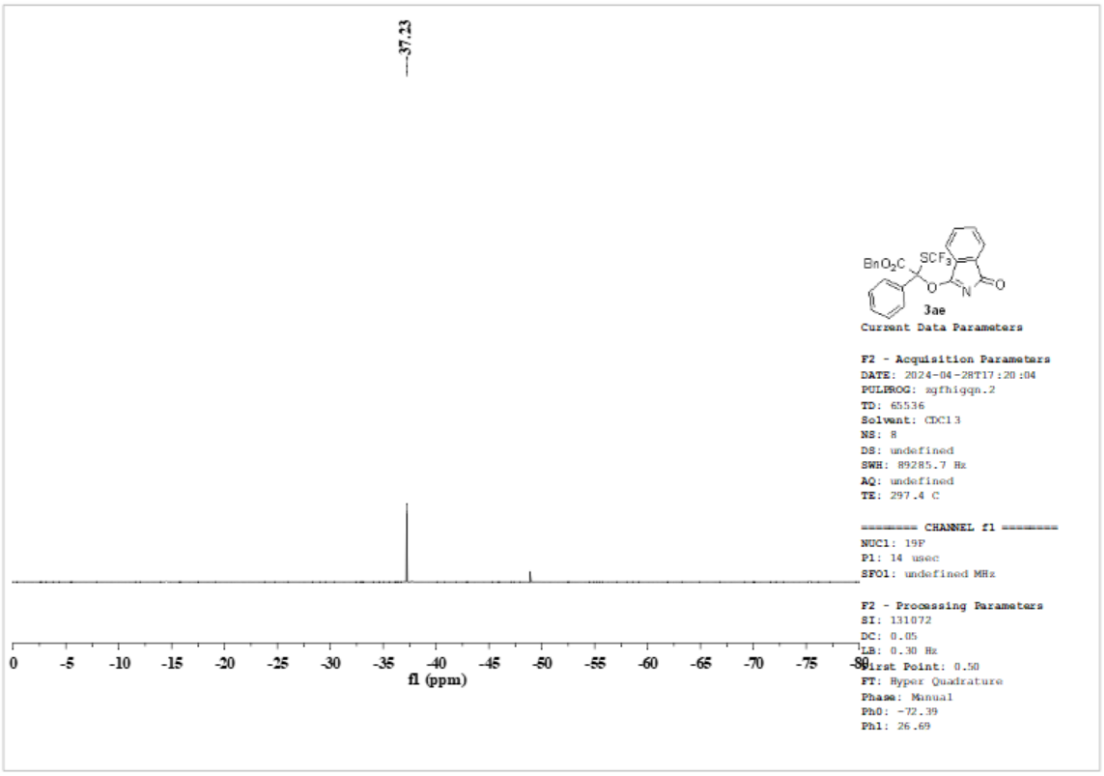
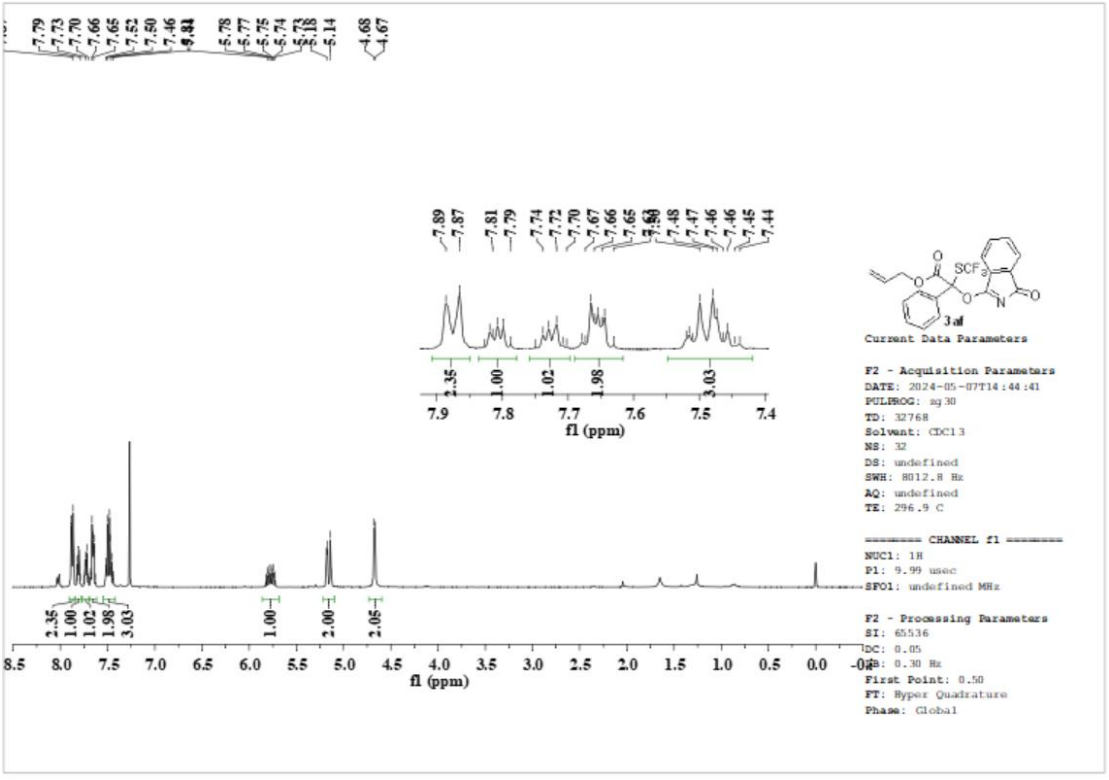
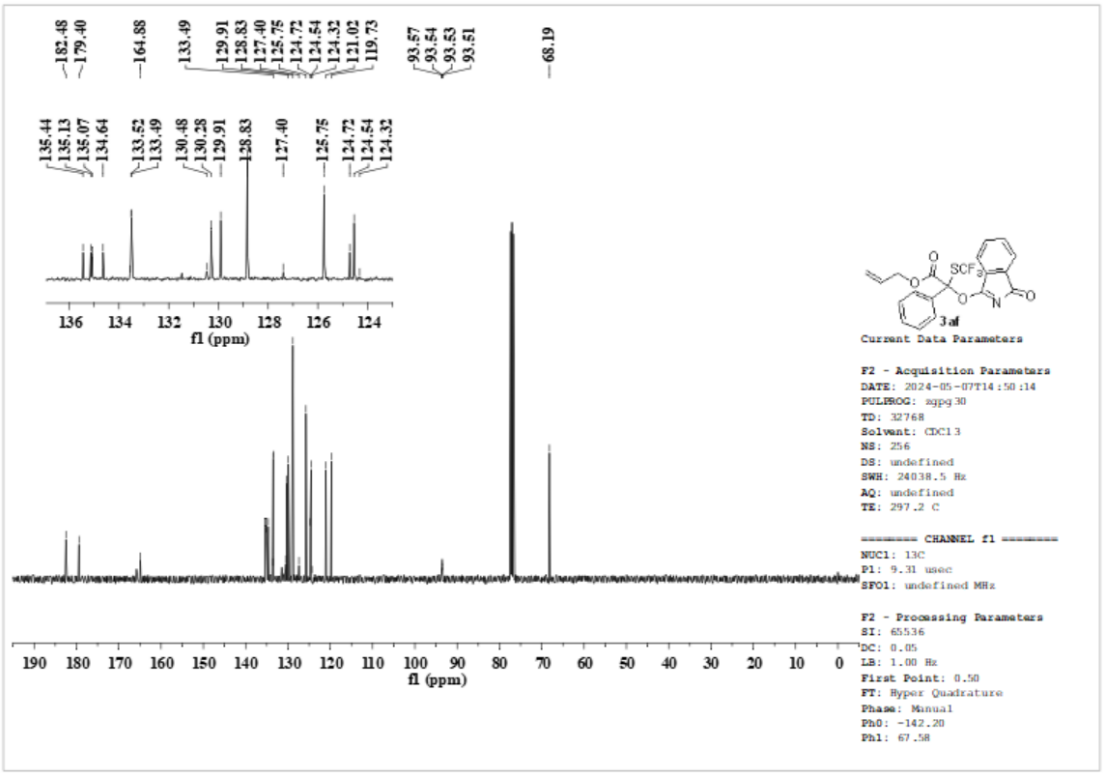
Ph0: -46.62
Ph1: -51.19

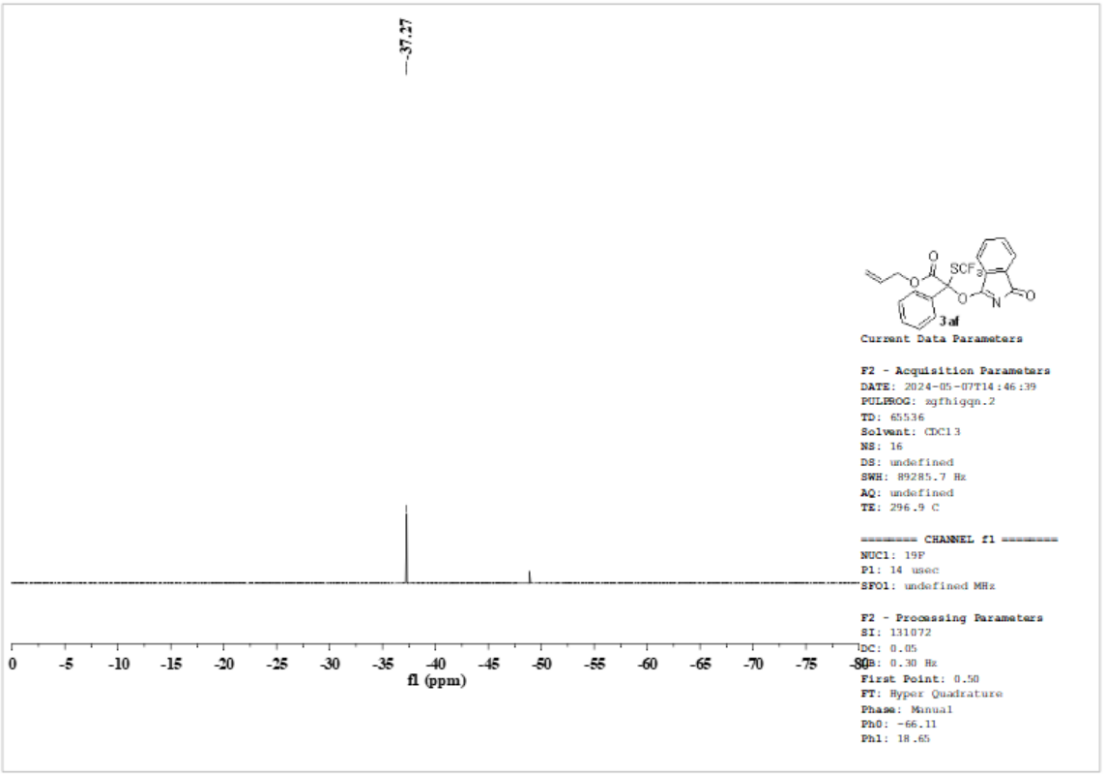

7.66
7.66
7.65
7.49
7.47
7.28
7.26
7.24
7.23
7.15
7.13
5.26
5.23
5.22
5.19
7.86
7.82
7.80
7.71
7.66
7.66
7.65
7.49
7.47
7.28
7.26
7.24
7.23
7.15
7.13
2.05
0.95
1.00
1.95
3.02
4.06
1.92
1.99
f1 (ppm)
3ae
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-04-28T17:04:11
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.2 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

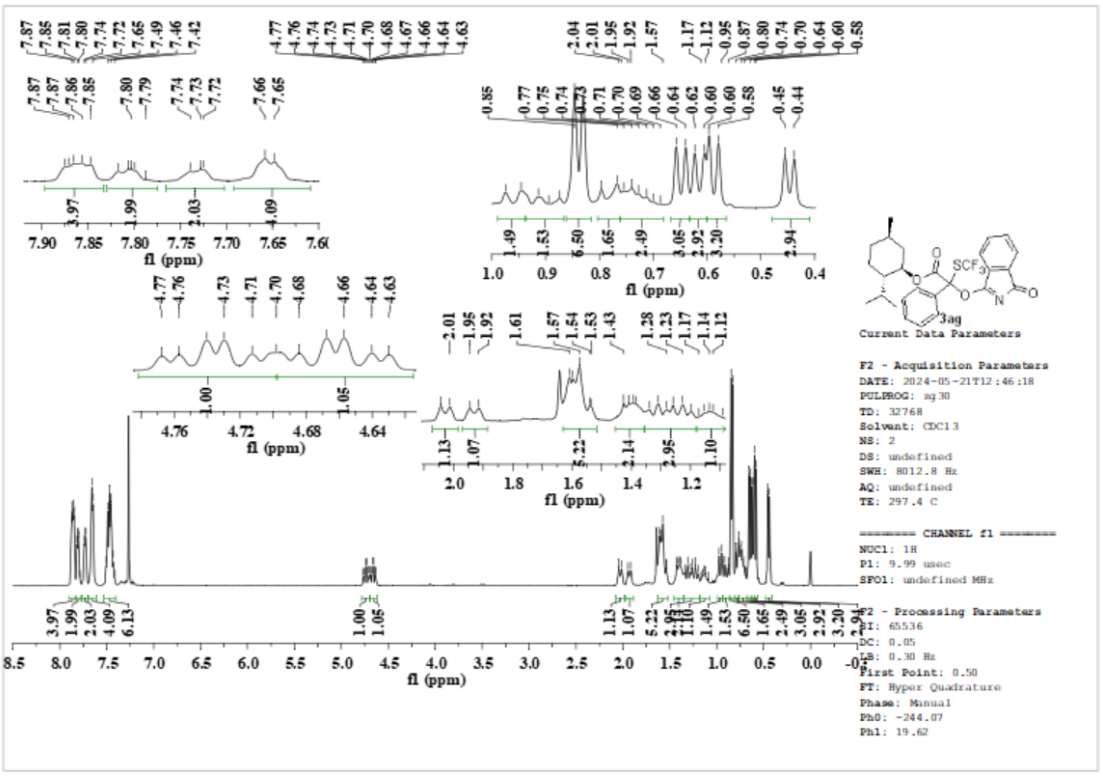

182.45
179.31
165.00
135.09
134.63
133.57
133.44
129.86
128.58
128.23
125.77
124.49
121.01
93.59
93.57
69.30
135.44
135.09
135.00
134.63
134.00
133.57
133.48
133.44
130.49
129.86
128.80
128.58
128.52
128.23
127.41
125.77
124.72
124.49
124.32
f1 (ppm)
3ae
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-04-28T17:06:21
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 236
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 297.5 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -138.80
Ph1: 68.07

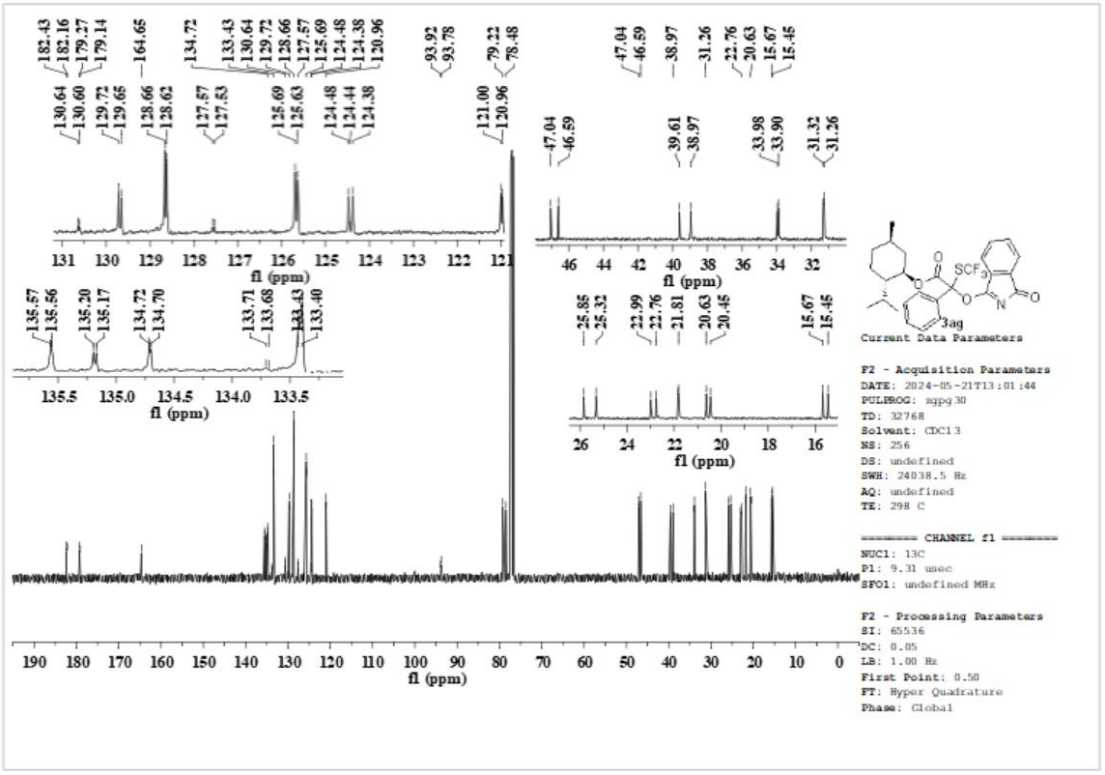

-37.23
BnO2C
SCF3
O
N
O
3ae
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-04-28T17:20:04
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 8
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.4 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -72.39
Ph1: 26.69
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80
f1 (ppm)

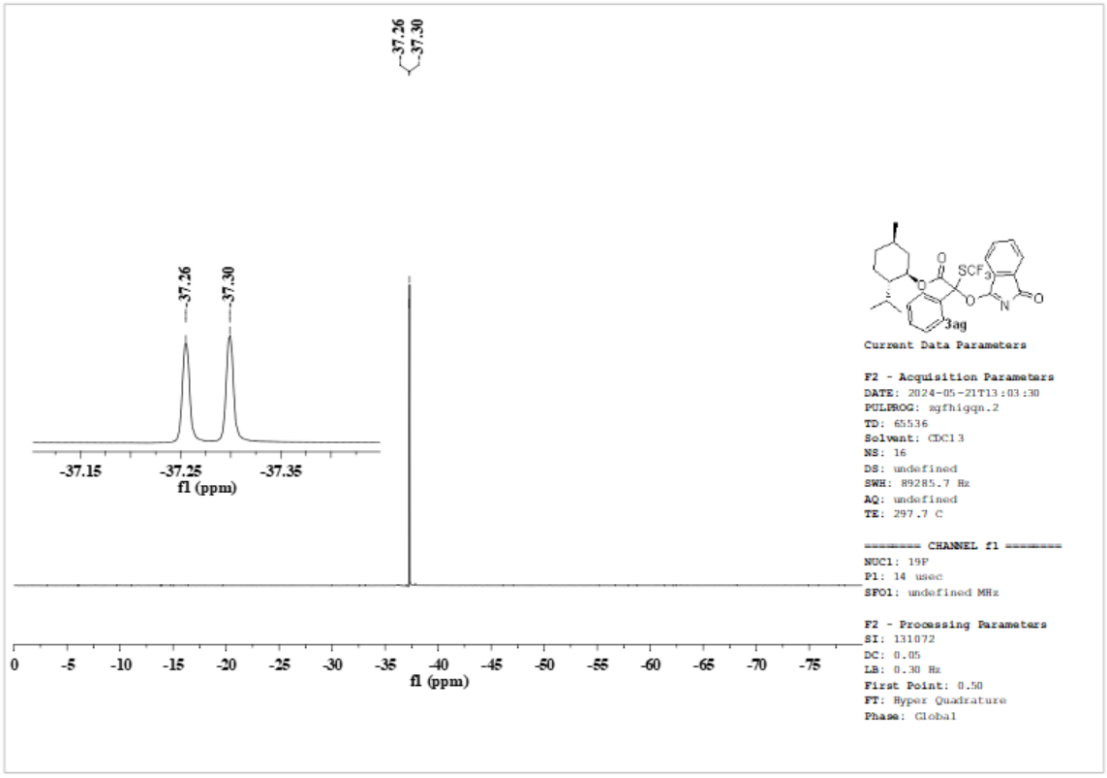

7.89 7.87 7.81 7.79 7.74 7.72 7.70 7.67 7.66 7.65 7.48 7.47 7.46 7.46 7.45 7.44
2.35 1.00 1.02 1.98 3.03
7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
7.79 7.73 7.70 7.66 7.65 7.52 7.50 7.46 5.78 5.77 5.75 5.74 5.73 5.18 5.14 4.68 4.67
2.35 1.00 1.02 1.98 3.03 1.00 2.00 2.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
O
SCF3
O
O
N
O
3af
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-07T14:44:41
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 296.9 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

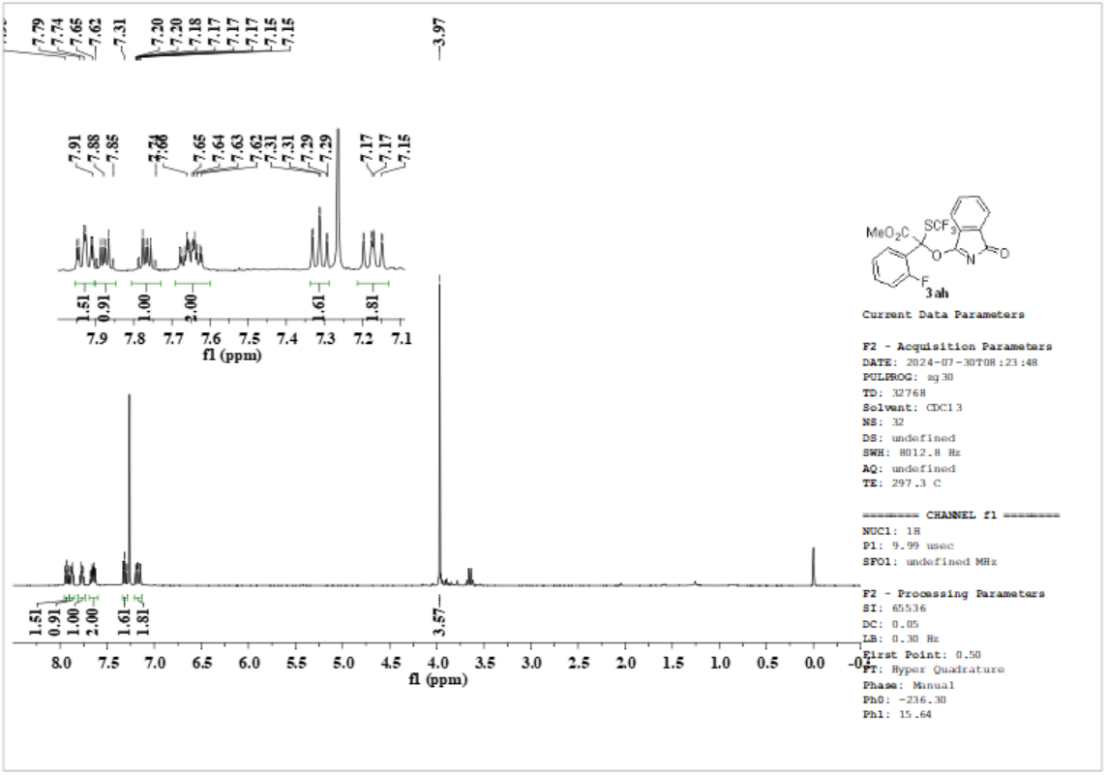

182.48
179.40
164.88
133.49
129.91
128.83
127.40
125.75
124.72
124.54
124.32
121.02
119.73
93.57
93.54
93.53
93.51
68.19
135.44
135.13
135.07
134.64
133.52
133.49
130.48
130.28
129.91
128.83
127.40
125.75
124.72
124.54
124.32
136 134 132 130 128 126 124
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
SCF3
3af
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-07T14:50:14
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 297.2 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -142.20
Ph1: 67.58

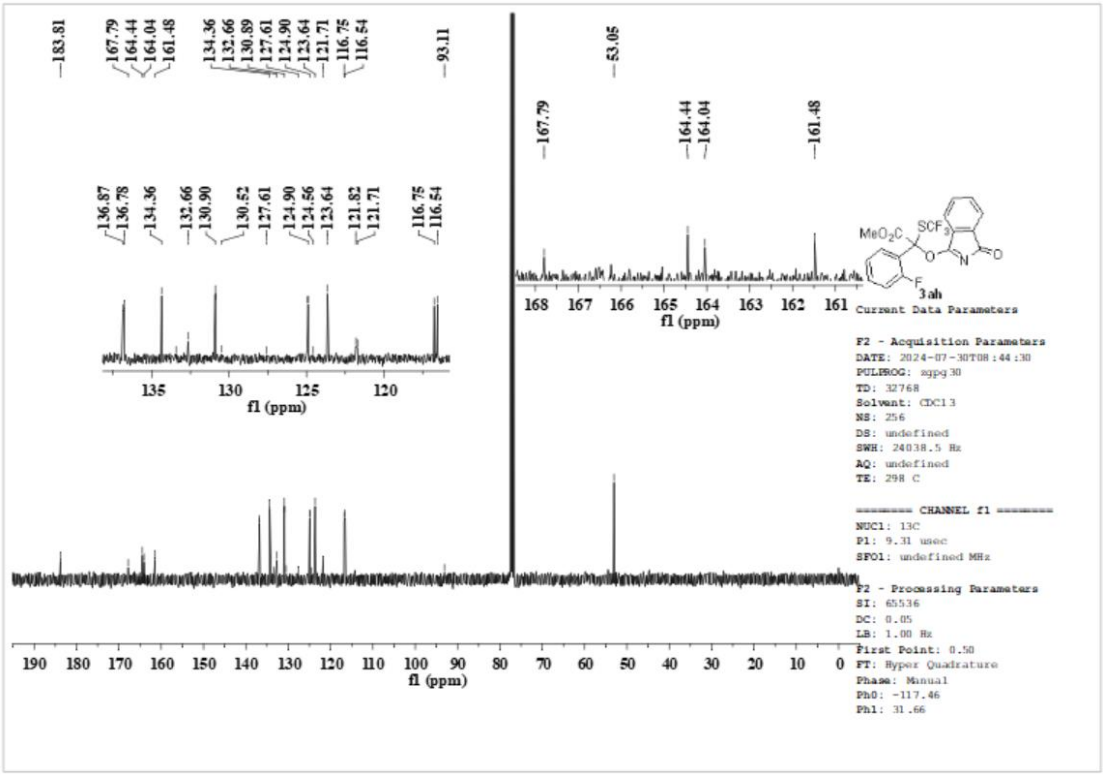

37.27
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80
f1 (ppm)
SCF3
3af
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-07T14:46:39
PULPROG: zgfhiqqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 296.9 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -66.11
Ph1: 18.65

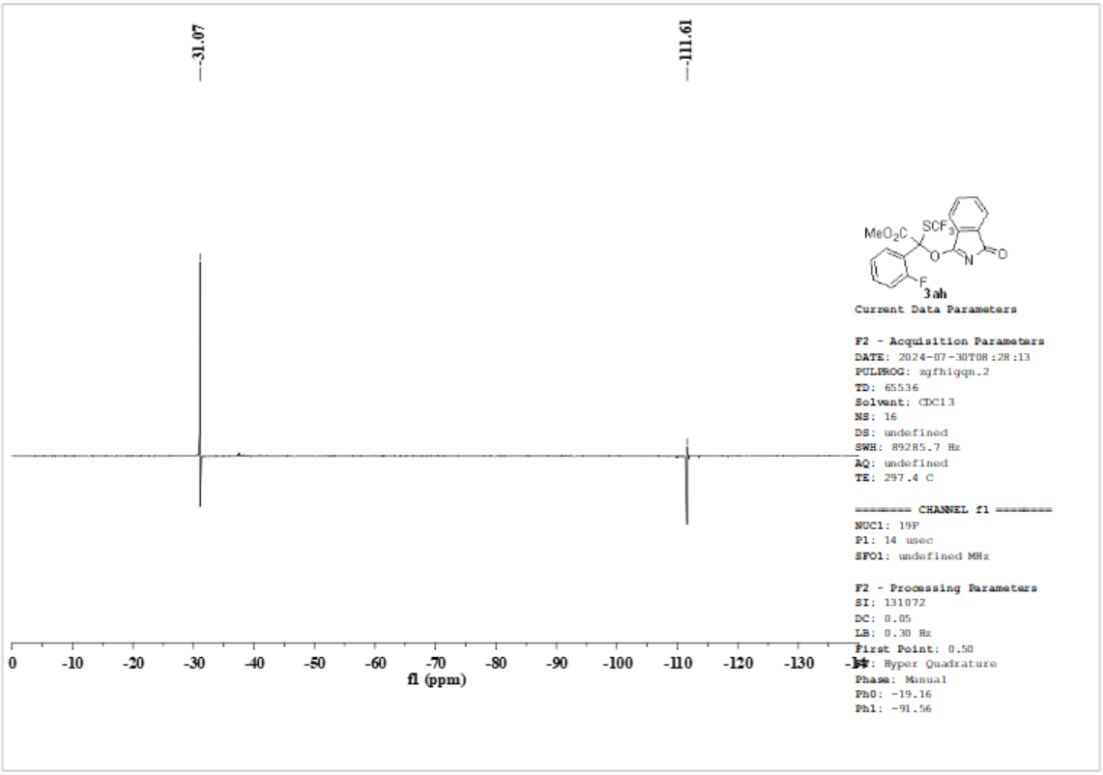

3ag
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-21T12:46:18
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 2
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.4 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -244.07
Ph1: 19.62
fl (ppm)

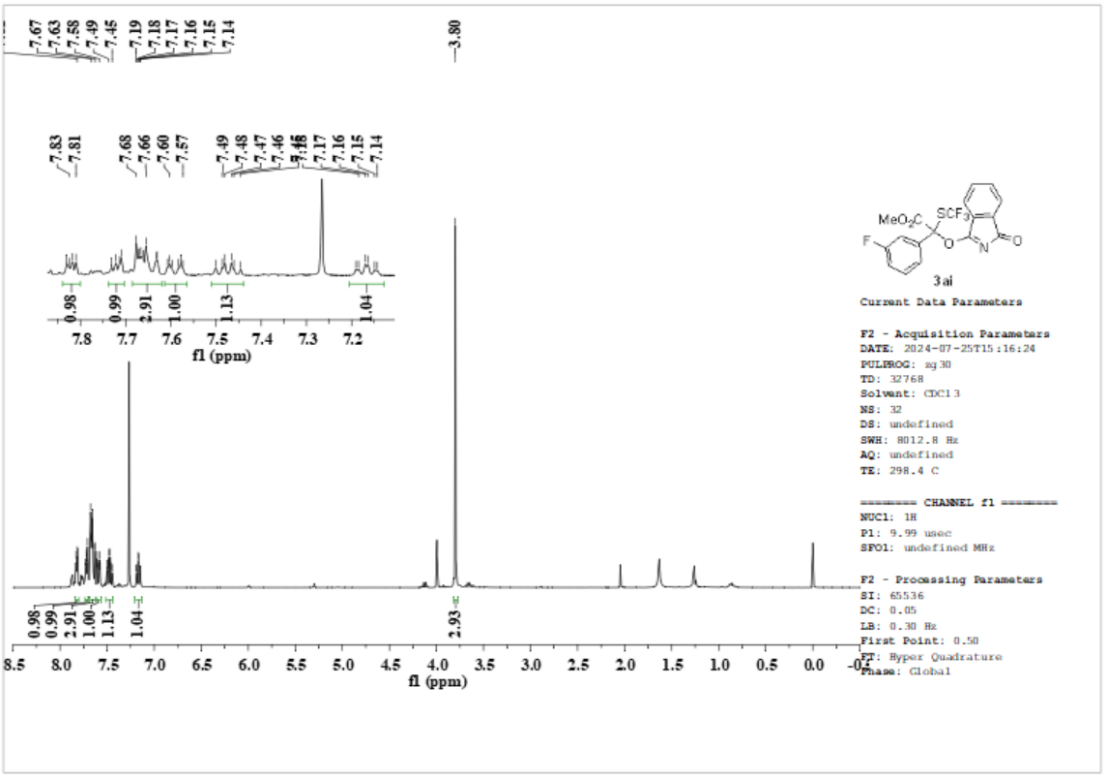

3ag
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-21T13:01:44
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 298 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
fl (ppm)

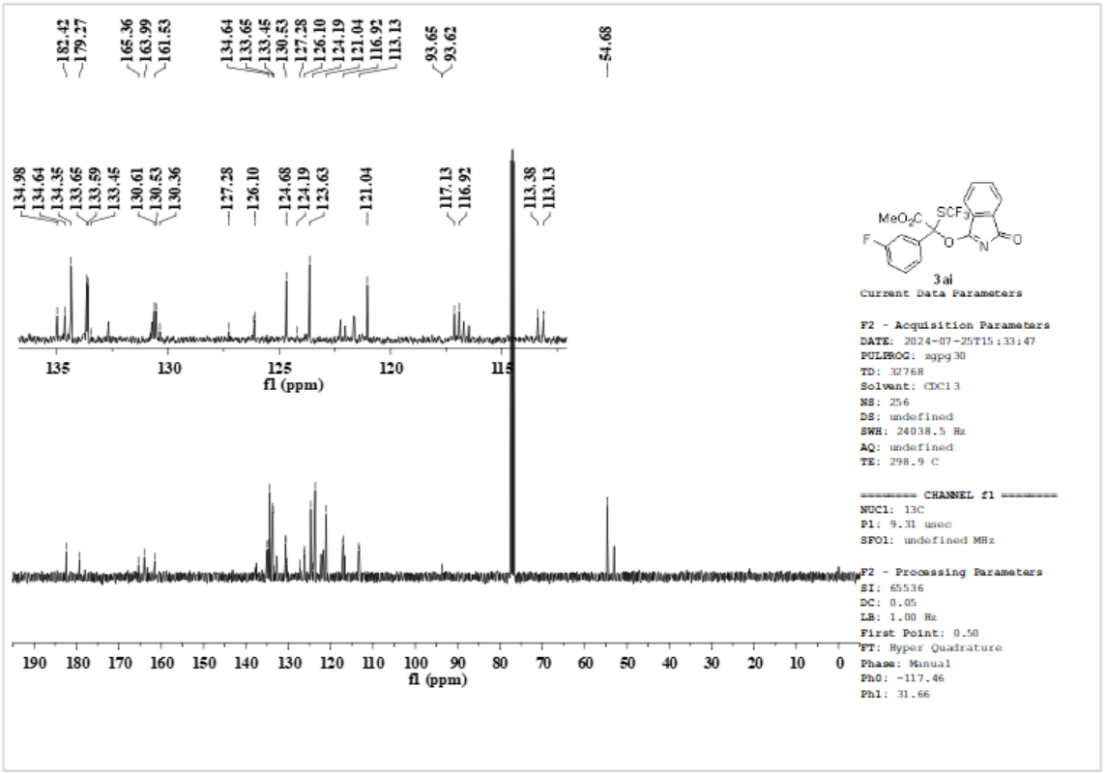

-37.26
-37.30
-37.15
-37.25
-37.35
f1 (ppm)
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75
f1 (ppm)
3ag
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-21T13:03:30
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.7 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

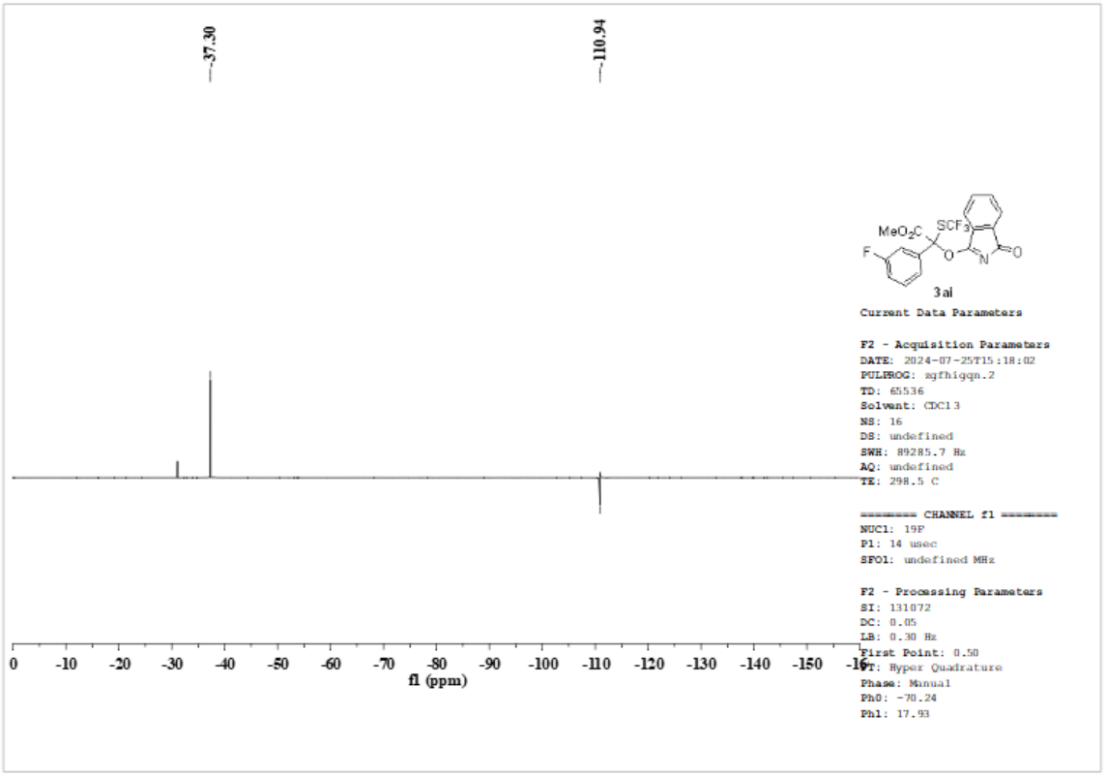

7.79
7.74
7.65
7.62
7.31
7.20
7.20
7.18
7.17
7.17
7.17
7.15
7.15
3.97
7.91
7.88
7.85
7.66
7.65
7.64
7.63
7.62
7.31
7.31
7.29
7.29
7.17
7.17
7.15
1.51
0.91
1.00
2.00
1.61
1.81
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1
f1 (ppm)
1.51
0.91
1.00
2.00
1.61
1.81
3.57
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
3ah
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-30T08:23:48
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.3 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -236.30
Ph1: 15.64

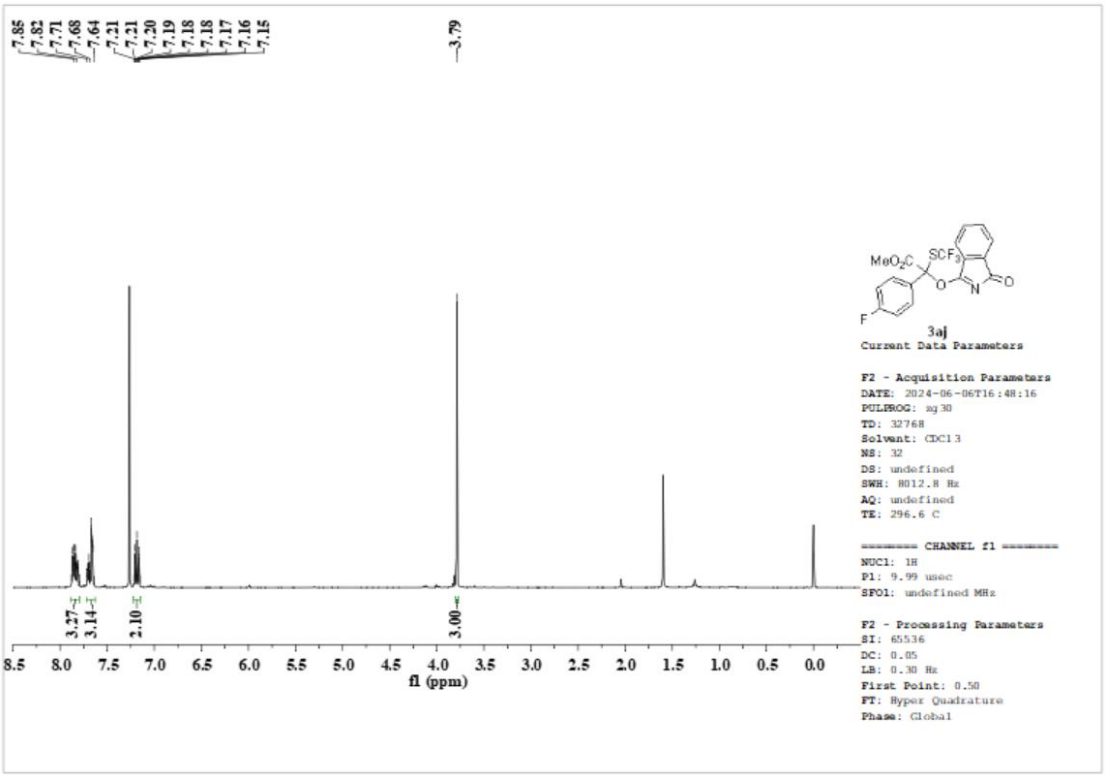

183.81
167.79
164.44
164.04
161.48
134.36
132.66
130.89
127.61
124.90
123.64
121.71
116.75
116.54
93.11
53.05
136.87
136.78
134.36
132.66
130.90
130.52
127.61
124.90
124.56
123.64
121.82
121.71
116.75
116.54
f1 (ppm)
MeO2C
SCF3
O
N
O
F
3ah
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-30T08:44:30
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 298 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -117.46
Ph1: 31.66

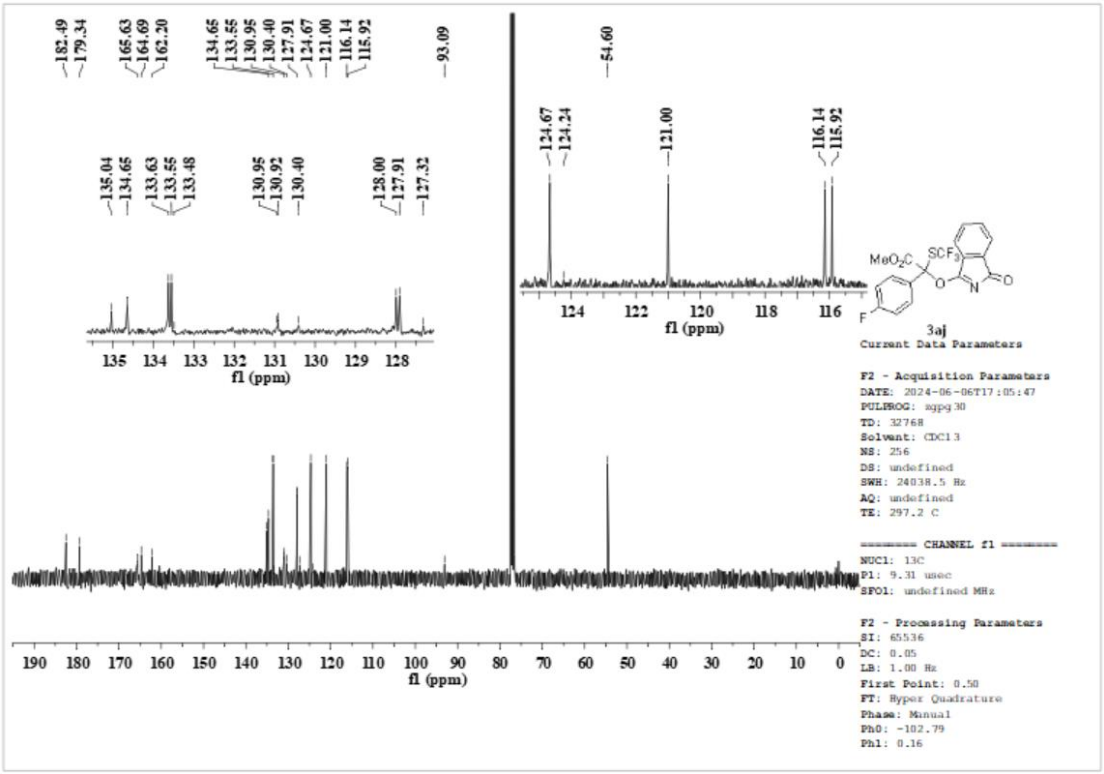

31.07
111.61
f1 (ppm)
MeO2C
SCF3
O
N
O
F
3ah
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-30T08:28:13
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.4 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -19.16
Ph1: -91.56

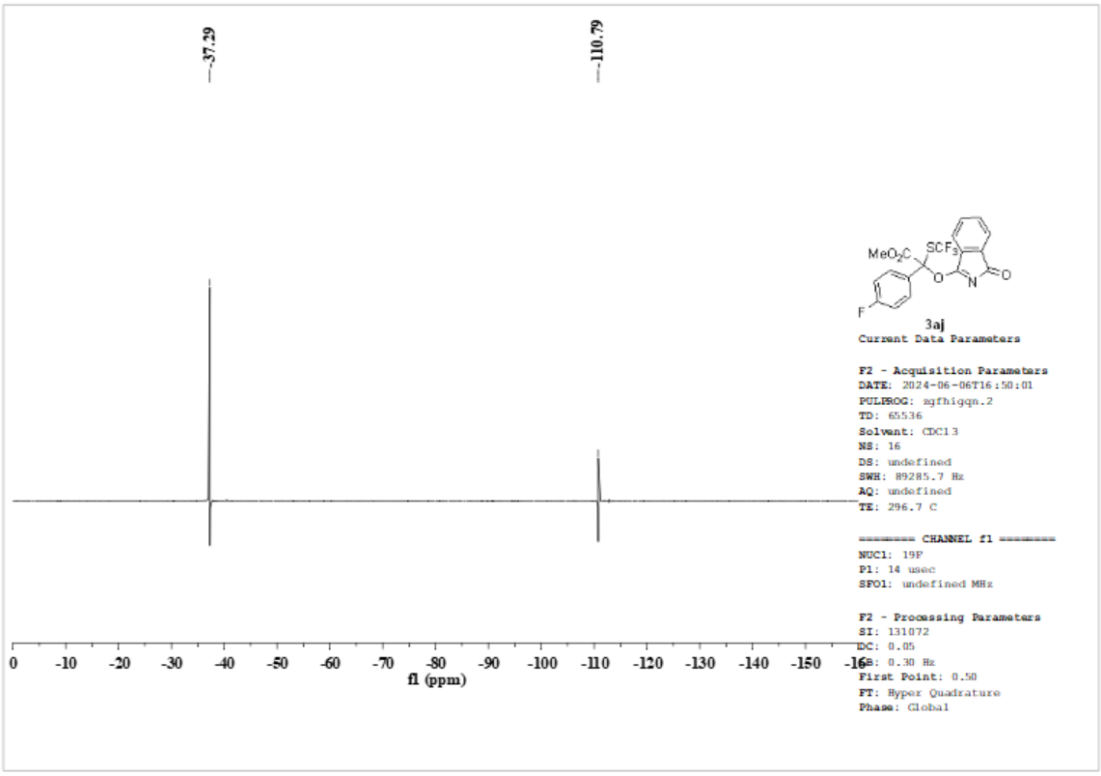

7.67
7.63
7.58
7.49
7.45
7.19
7.18
7.17
7.16
7.15
7.14
3.80
7.83
7.81
7.68
7.66
7.60
7.57
7.49
7.48
7.47
7.46
7.17
7.16
7.15
7.14
0.98
0.99
2.91
1.00
1.13
1.04
7.8
7.7
7.6
7.5
7.4
7.3
7.2
f1 (ppm)
0.98
0.99
2.91
1.00
1.13
1.04
2.93
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
MeO2C
SCF3
F
O
N
O
3ai
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-25T15:16:24
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 298.4 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

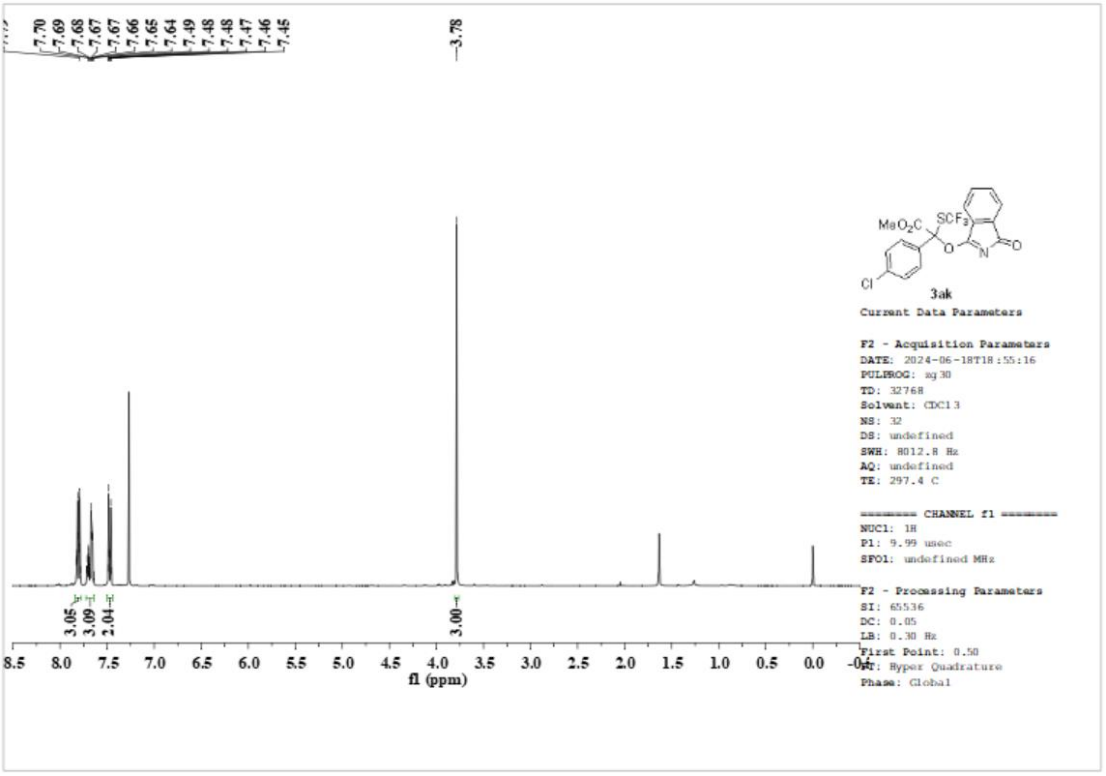

182.42
179.27
165.36
163.99
161.53
134.64
133.65
133.45
130.53
127.28
126.10
124.19
121.04
116.92
113.13
93.65
93.62
54.68
134.98
134.64
134.35
133.65
133.59
133.45
130.61
130.53
130.36
127.28
126.10
124.68
124.19
123.63
121.04
117.13
116.92
113.38
113.13
135
130
125
120
115
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
MeO2C
SCF3
F
O
N
O
3ai
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-25T15:33:47
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 298.9 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -117.46
Ph1: 31.66

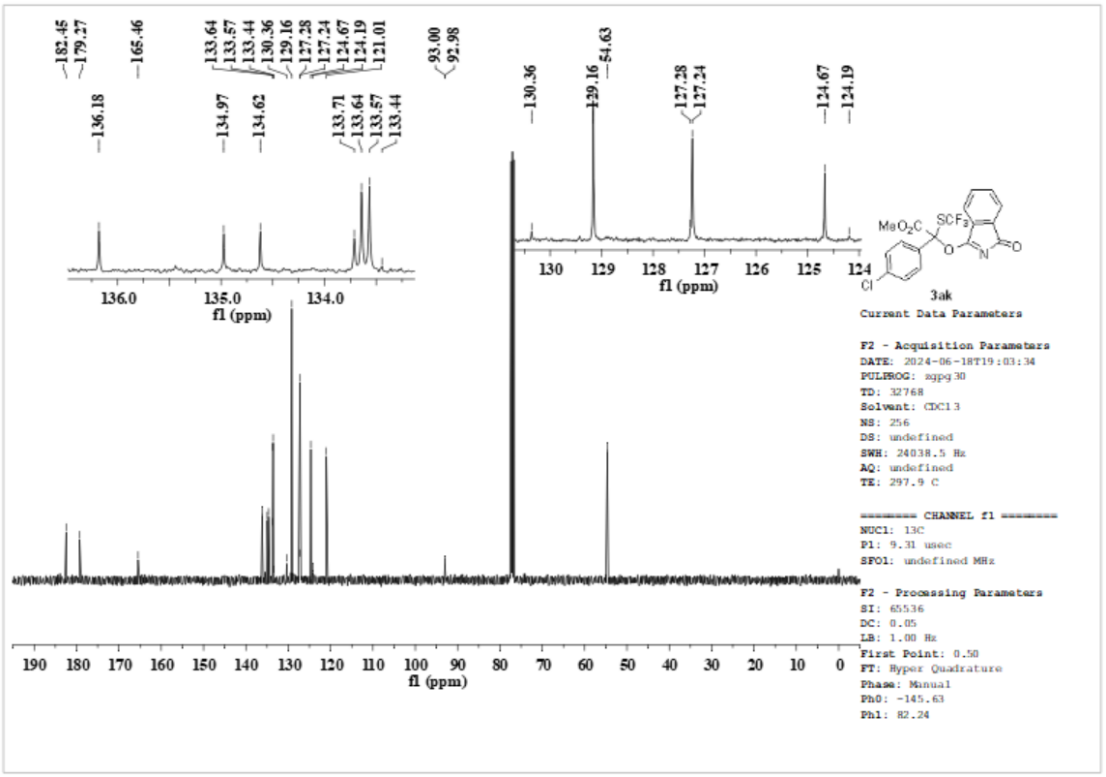

-37.30
-110.94
MeO2C
SCF3
F
O
N
O
3ai
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-25T15:18:02
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 298.5 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -70.24
Ph1: 17.93
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)

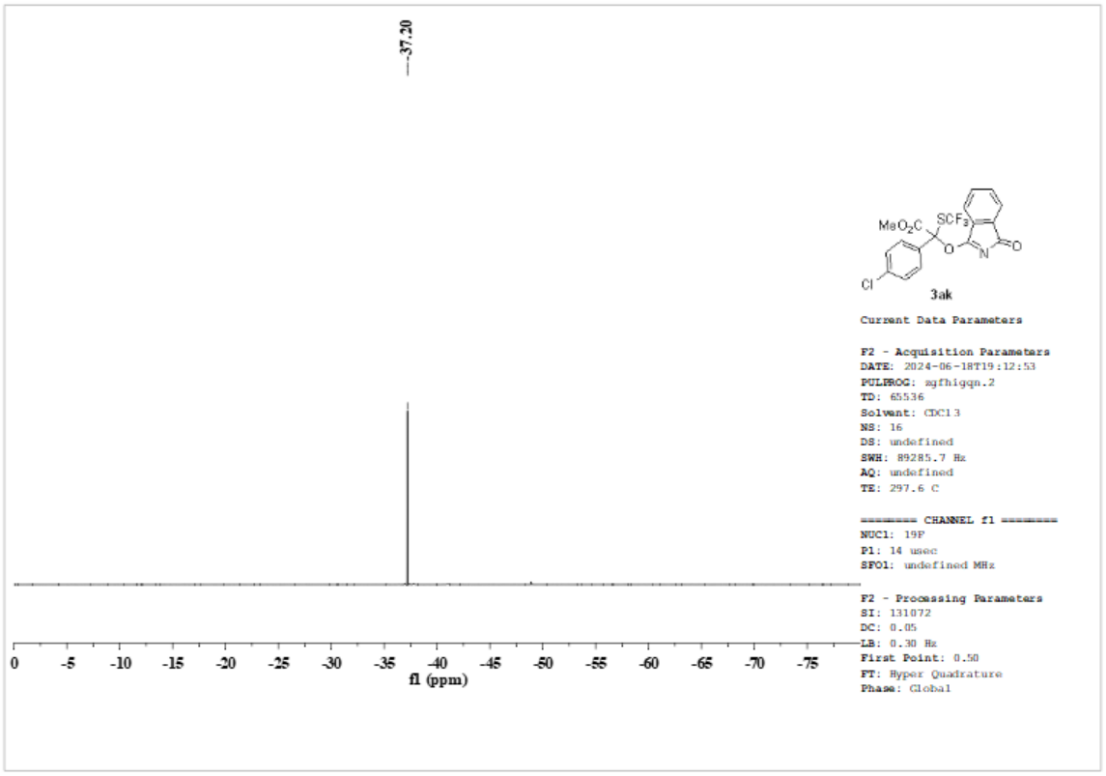

7.85
7.82
7.71
7.68
7.64
7.21
7.21
7.20
7.19
7.18
7.18
7.17
7.16
7.15
3.79
MeO2C
SCF3
F
O
N
O
3aj
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-06T16:48:16
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 296.6 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
3.27
3.14
2.10
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

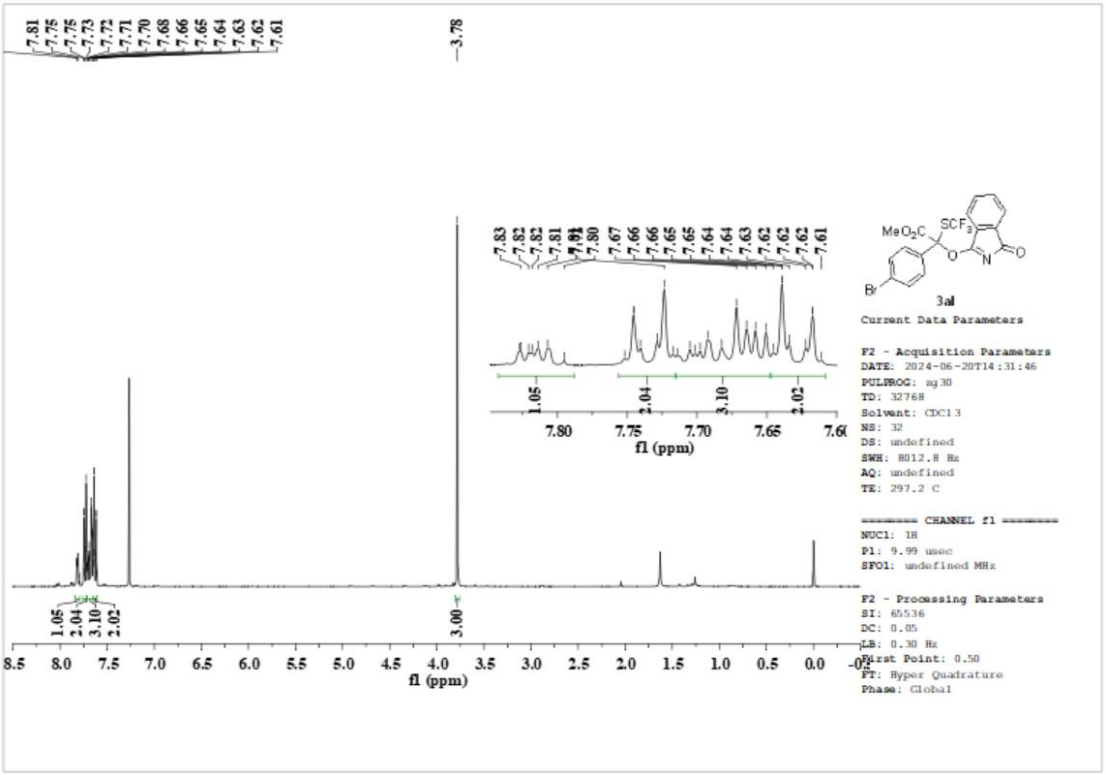

182.49
179.34
165.63
164.69
162.20
134.65
133.55
130.95
130.40
127.91
124.67
121.00
116.14
115.92
93.09
54.60
135.04
134.65
133.63
133.55
133.48
130.95
130.92
130.40
128.00
127.91
127.32
124.67
124.24
121.00
116.14
115.92
fl (ppm)
MeO2C
SCF3
O
N
O
F
3aj
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-06T17:05:47
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 297.2 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
GB: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -102.79
Ph1: 0.16

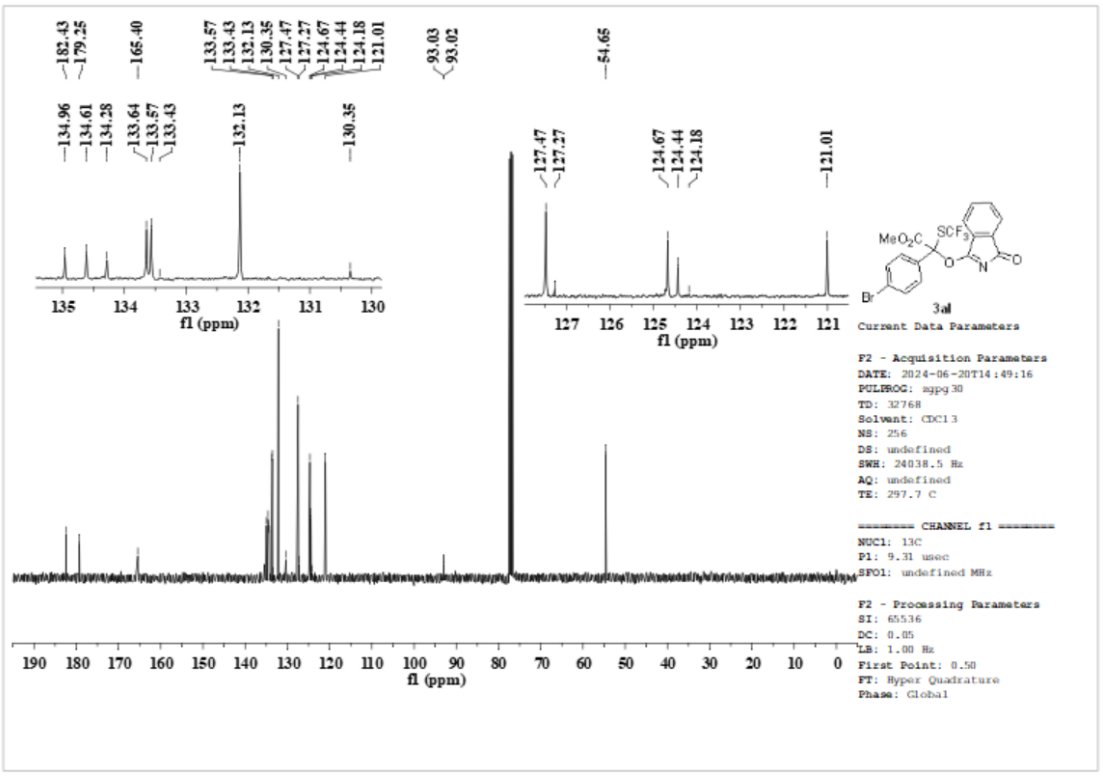

37.29
110.79
fl (ppm)
MeO2C
SCF3
O
N
O
F
3aj
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-06T16:50:01
PULPROG: zgfhiqqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 296.7 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
GB: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

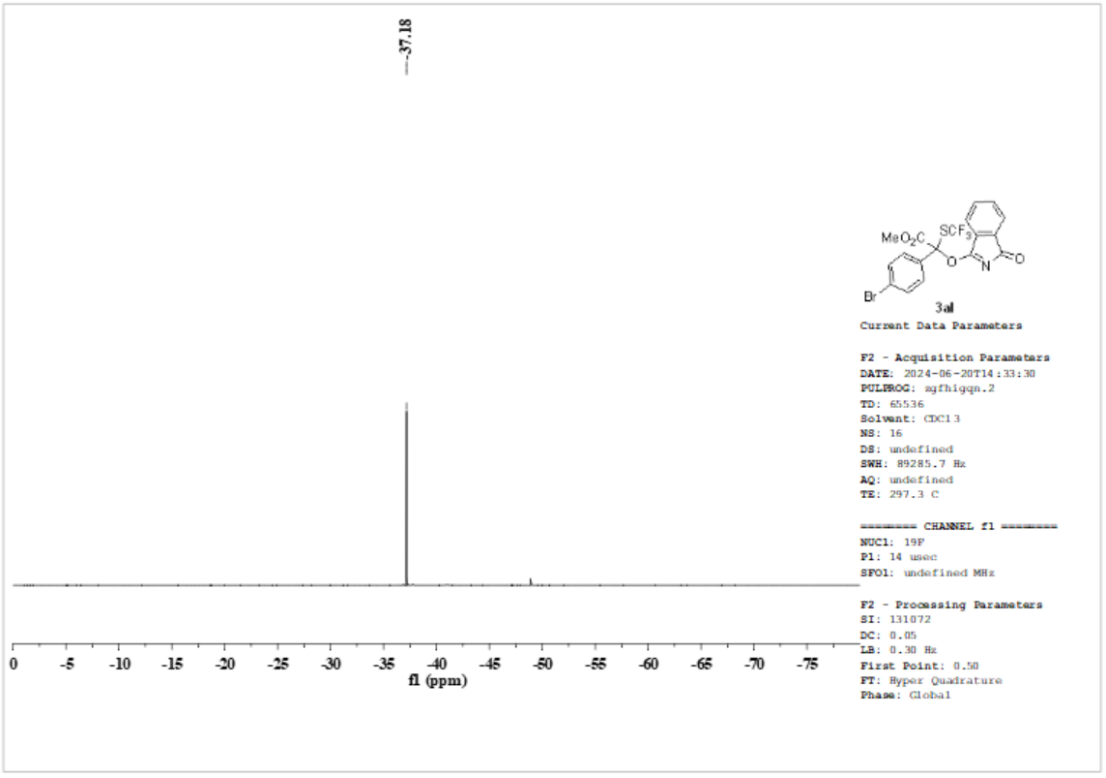

7.70
7.69
7.68
7.67
7.67
7.66
7.65
7.64
7.49
7.48
7.48
7.47
7.46
7.45
3.78
3.05
3.09
2.04
3.00
fl (ppm)
3ak
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-18T18:55:16
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.4 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

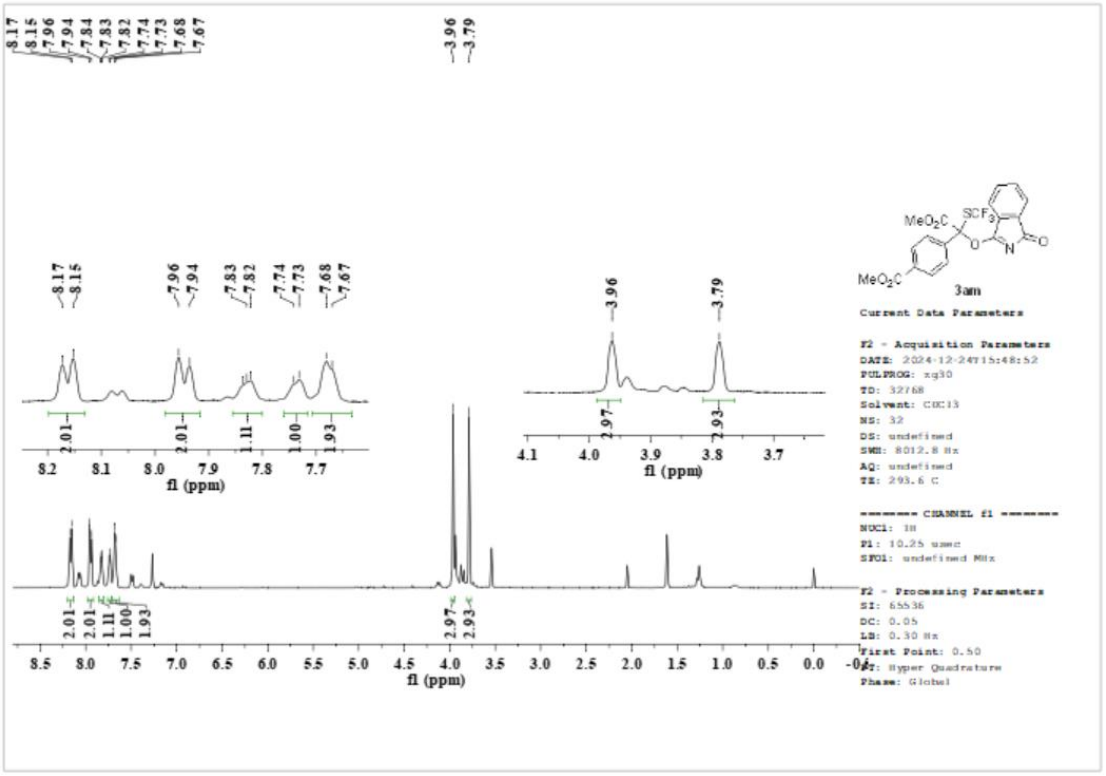

182.45
179.27
165.46
136.18
134.97
134.62
133.71
133.64
133.57
133.44
133.64
133.57
133.44
130.36
129.16
127.28
127.24
124.67
124.19
121.01
93.00
92.98
54.63
fl (ppm)
3ak
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-18T19:03:34
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 297.9 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
PH0: -145.63
PH1: 82.24

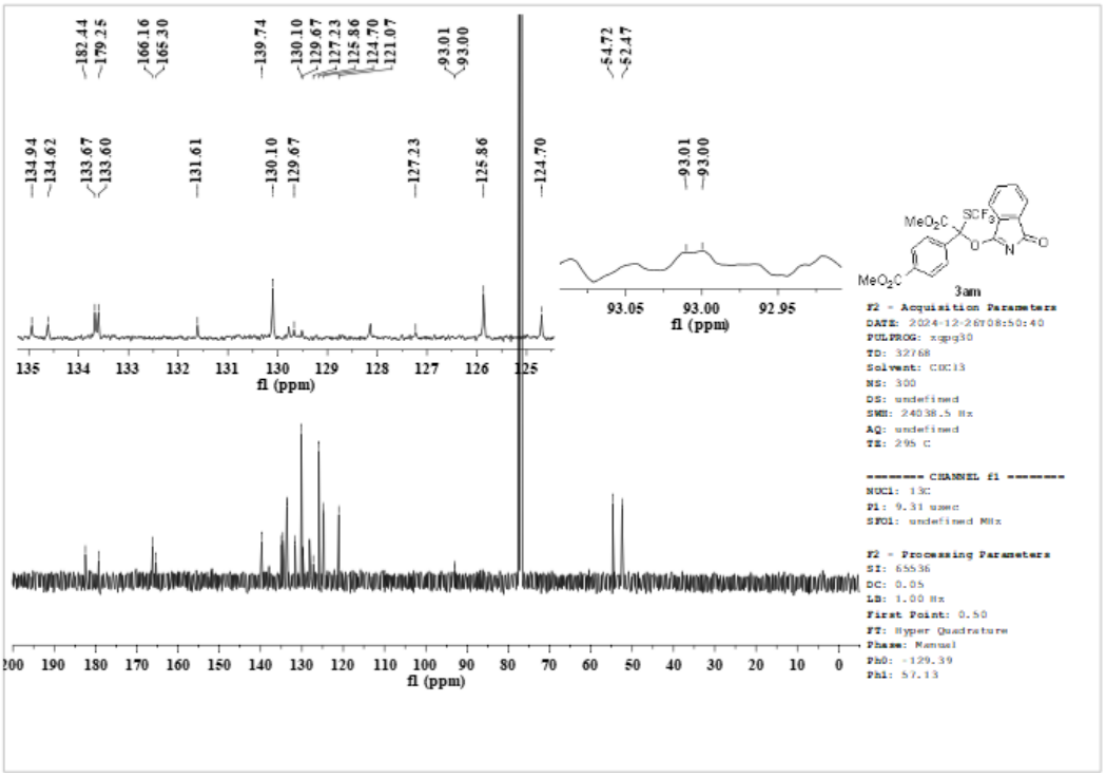

-37.20
3ak
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-18T19:12:53
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.6 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)

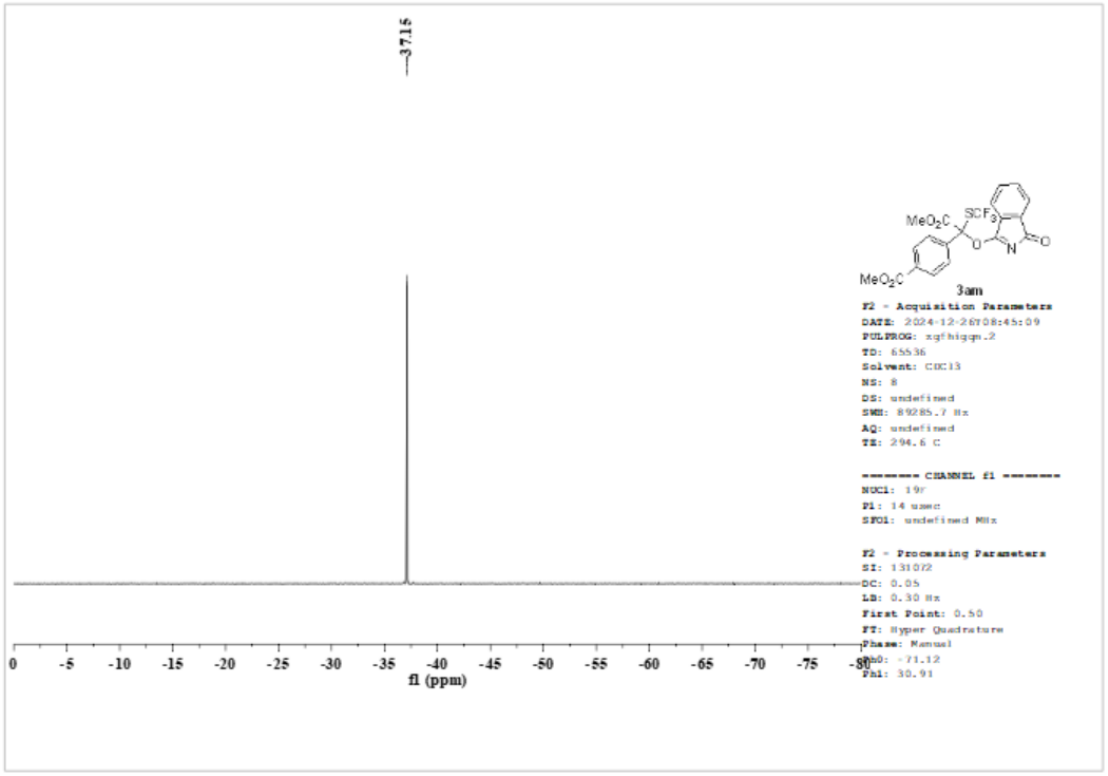

7.81
7.75
7.75
7.73
7.72
7.71
7.70
7.68
7.66
7.65
7.64
7.63
7.62
7.61
-3.78
3al
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-20T14:31:46
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.2 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
1.05
2.04
3.10
2.02
3.00
f1 (ppm)

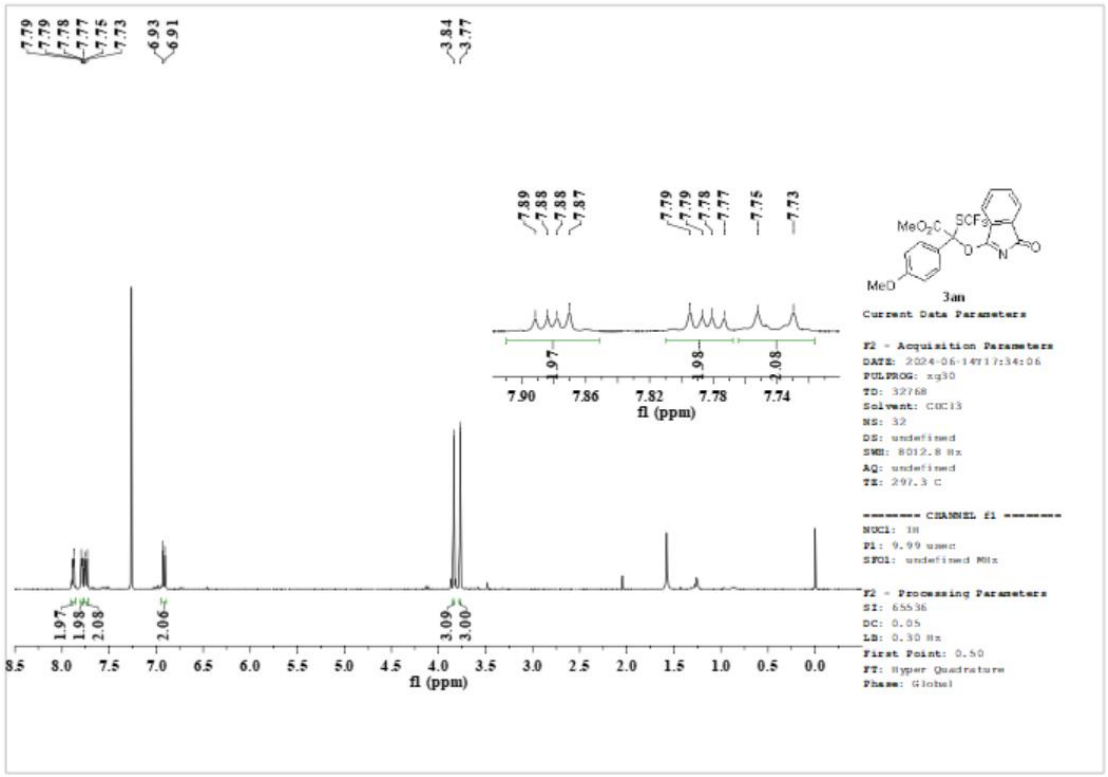

182.43
179.25
165.40
133.57
133.43
132.13
130.35
127.47
127.27
124.67
124.44
124.18
121.01
93.03
93.02
54.65
134.96
134.61
134.28
133.64
133.57
133.43
132.13
130.35
127.47
127.27
124.67
124.44
124.18
121.01
f1 (ppm)
f1 (ppm)
f1 (ppm)
3al
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-20T14:49:16
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 297.7 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

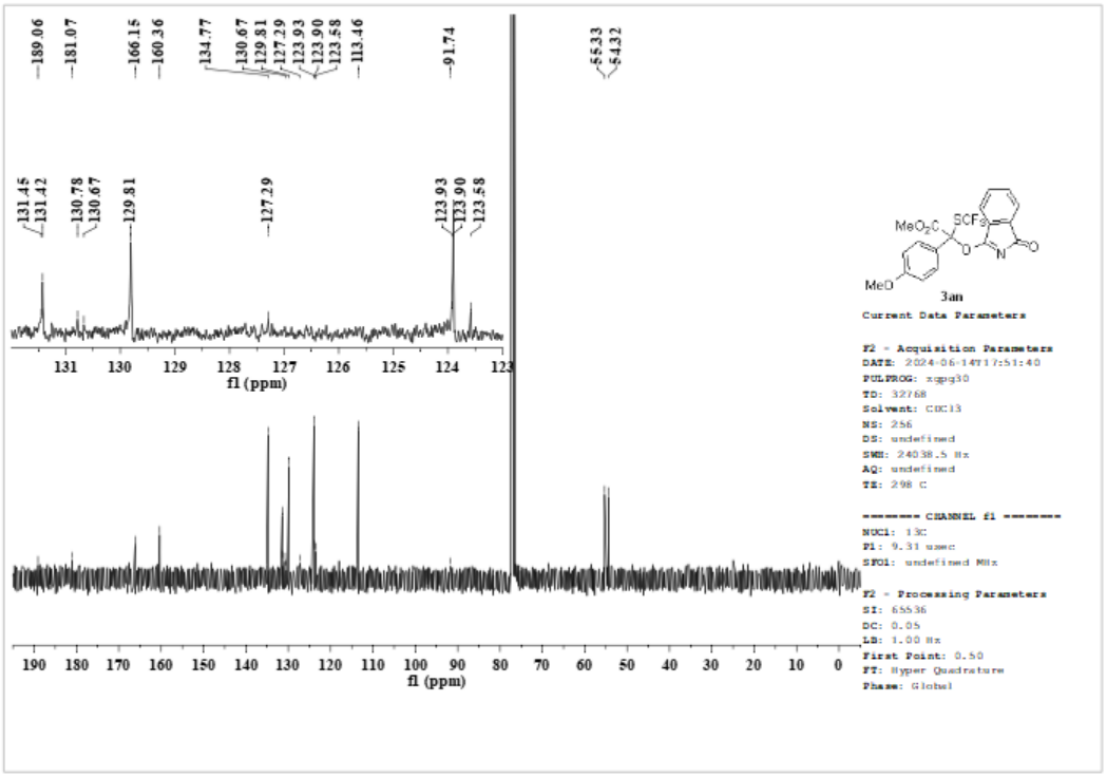

37.18
f1 (ppm)
3al
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-20T14:33:30
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.3 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

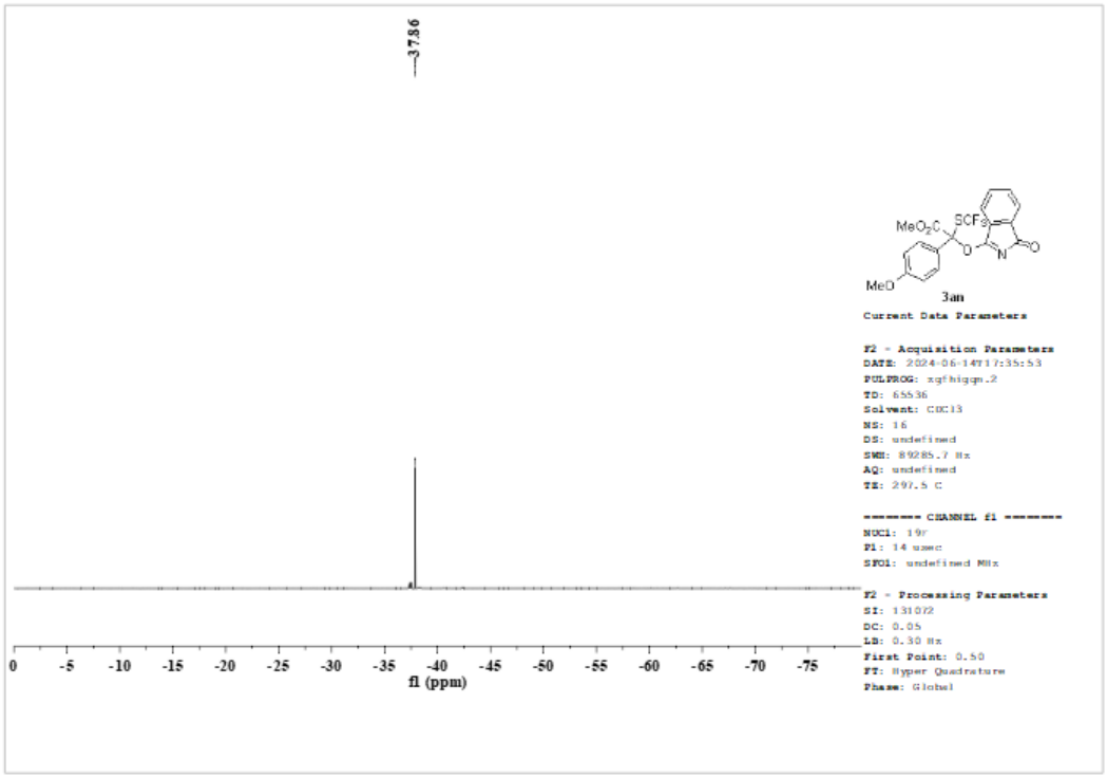

8.17
8.15
7.96
7.94
7.84
7.83
7.82
7.74
7.73
7.68
7.67
3.96
3.79
MeO2C
SCF3
O
N
O
MeO2C
3am
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-12-24T15:48:52
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 293.6 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 10.25 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
2.01
2.01
1.11
1.00
1.93
2.97
2.93
fl (ppm)

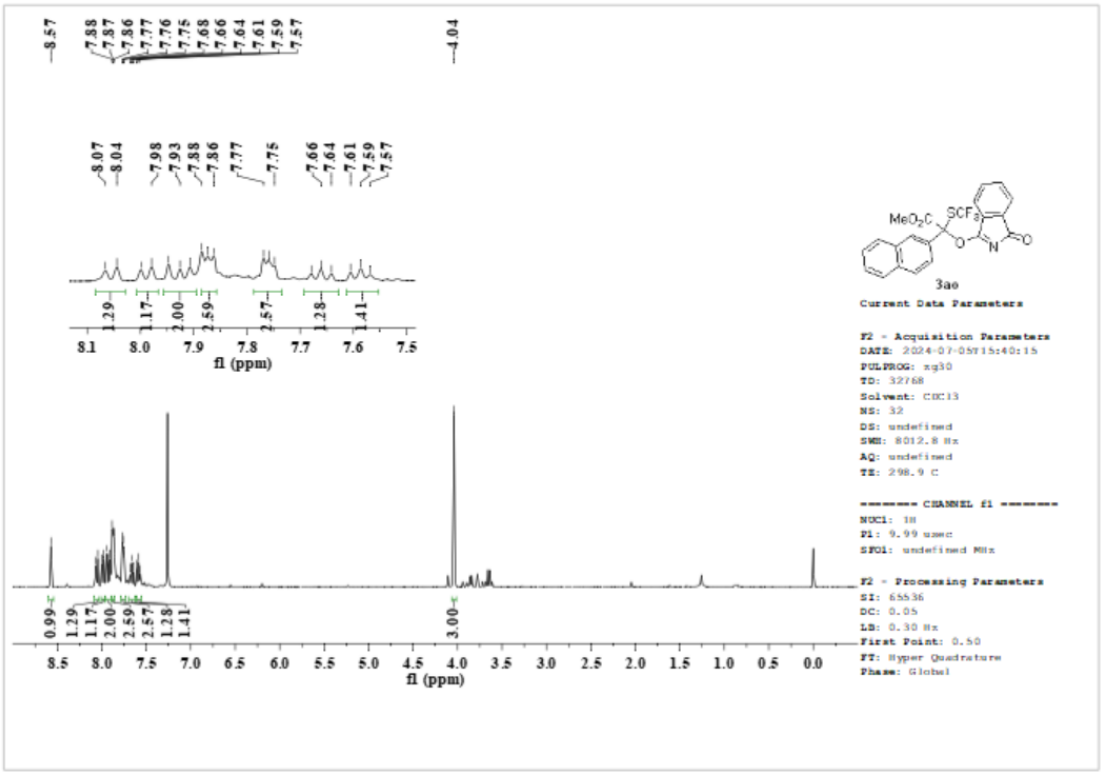

182.44
179.25
166.16
165.30
139.74
130.10
129.67
127.23
125.86
124.70
121.07
93.01
93.00
54.72
52.47
134.94
134.62
133.67
133.60
131.61
130.10
129.67
127.23
125.86
124.70
93.01
93.00
MeO2C
SCF3
O
N
O
MeO2C
3am
F2 - Acquisition Parameters
DATE: 2024-12-26T08:50:40
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 300
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 295 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -129.39
Ph1: 57.13
fl (ppm)

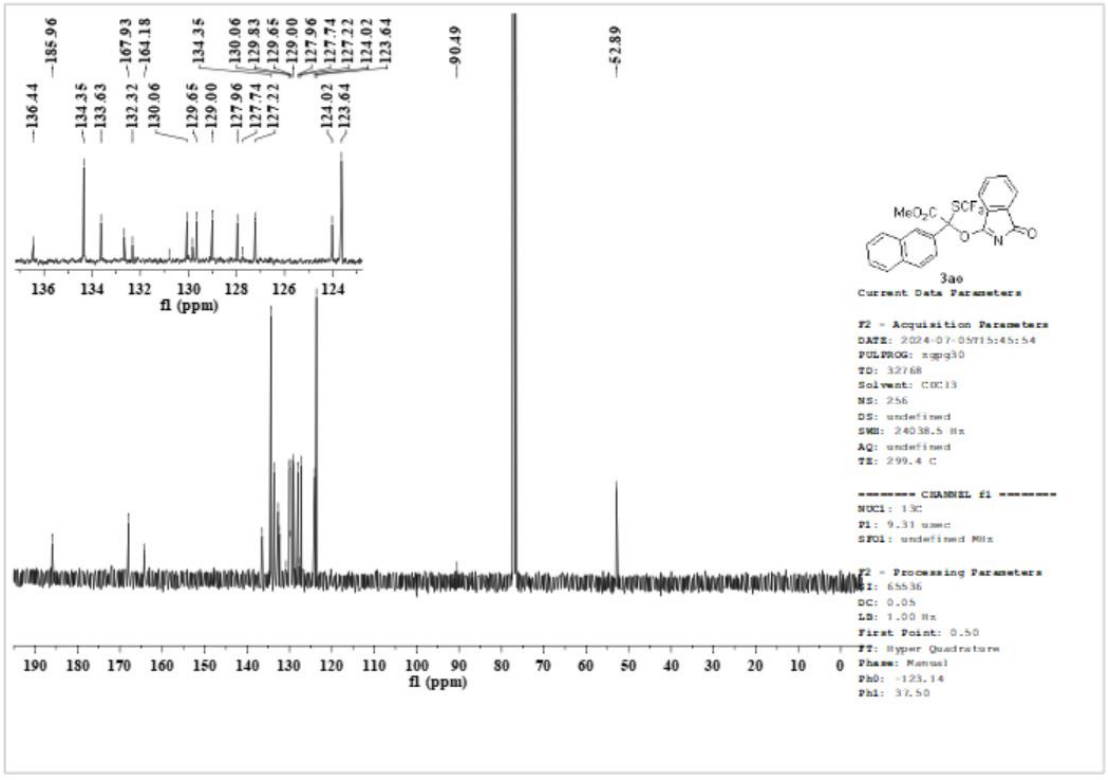

-37.15
3am
F2 - Acquisition Parameters
DATE: 2024-12-26T08:45:09
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 8
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 294.6 C
------- CHANNEL f1 -------
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
PH0: -71.12
PH1: 30.91
f1 (ppm)

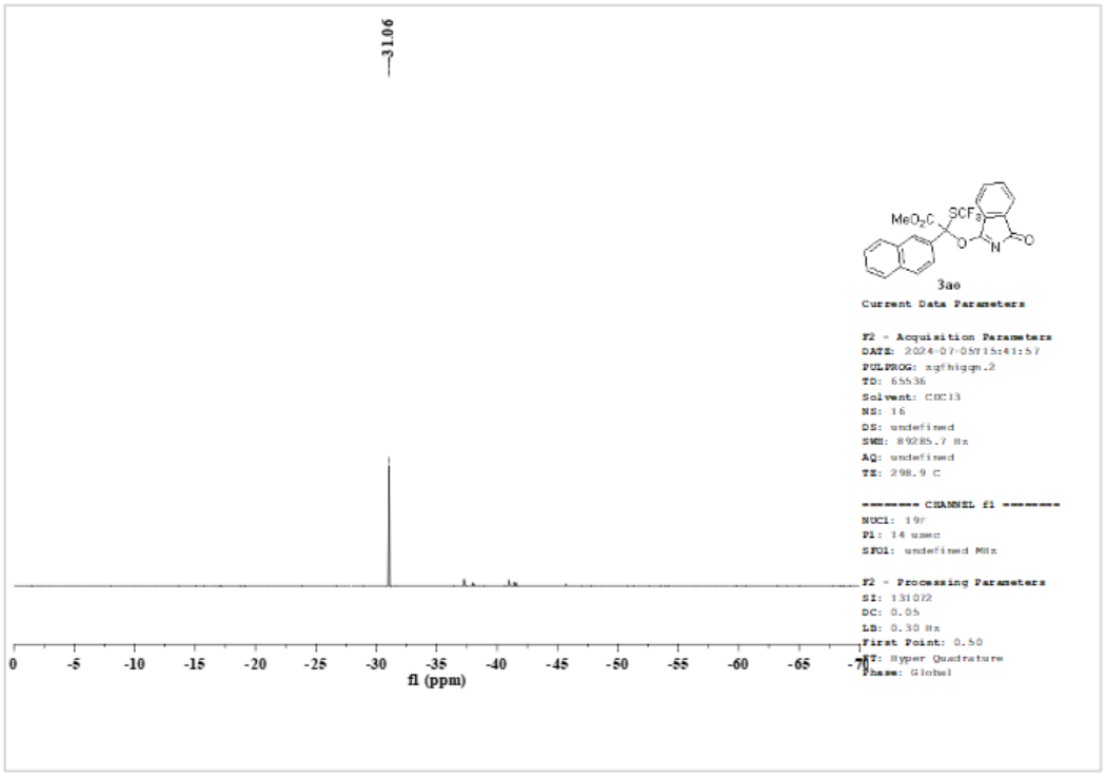

7.79
7.79
7.78
7.77
7.75
7.73
6.93
6.91
3.84
3.77
1.97
1.98
2.08
2.06
3.09
3.00
3am
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-14T17:34:06
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.3 C
------- CHANNEL f1 -------
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)

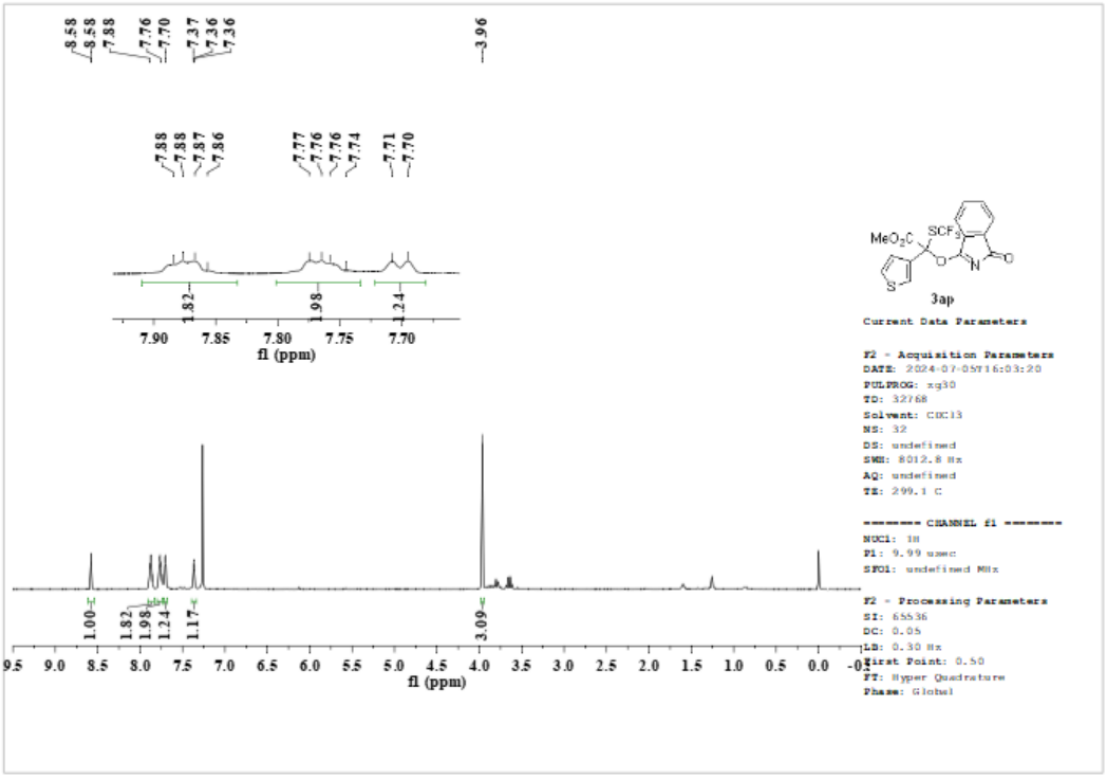

189.06
181.07
166.15
160.36
134.77
130.67
129.81
127.29
123.93
123.90
123.58
113.46
91.74
55.33
54.32
131.45
131.42
130.78
130.67
129.81
127.29
123.93
123.90
123.58
fl (ppm)
MeO2C
SCF3
MeO
3an
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-14T17:51:40
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 298 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
fl (ppm)

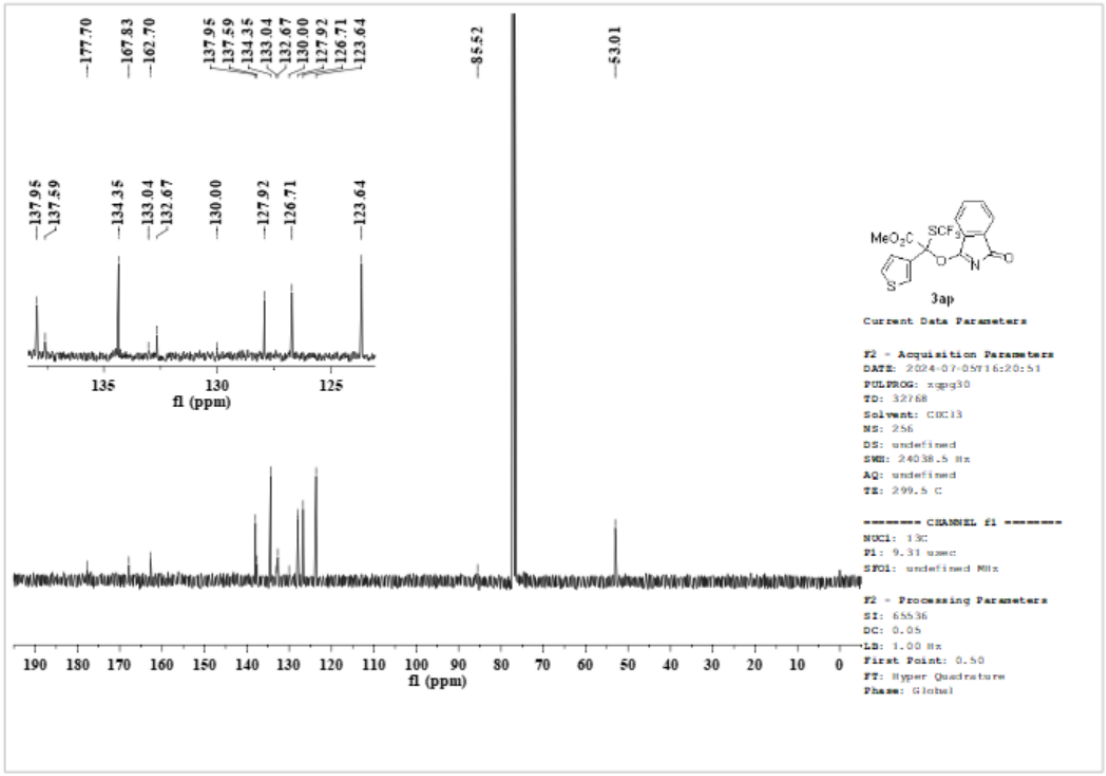

37.86
MeO2C
SCF3
MeO
3an
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-06-14T17:35:53
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.5 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
fl (ppm)

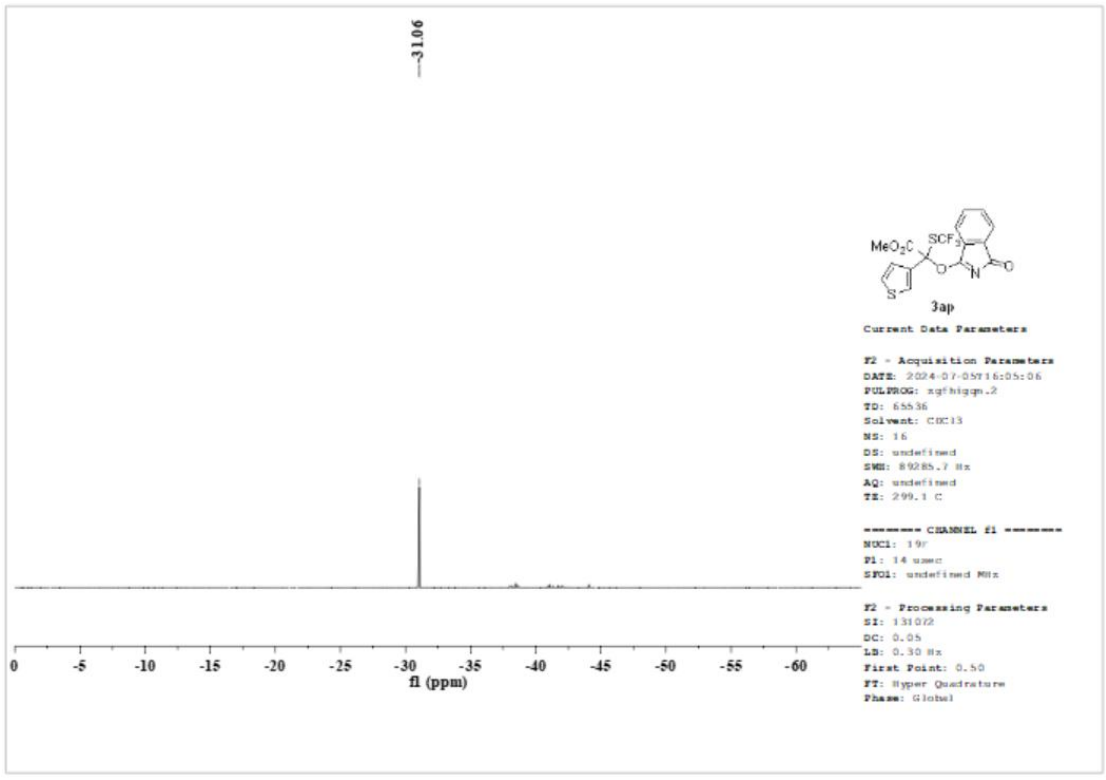

8.57
7.88
7.87
7.86
7.77
7.76
7.75
7.68
7.66
7.64
7.61
7.59
7.57
4.04
8.07
8.04
7.98
7.93
7.88
7.86
7.77
7.75
7.66
7.64
7.61
7.59
7.57
1.29
1.17
2.00
2.59
2.57
1.28
1.41
8.1 8.0 7.9 7.8 7.7 7.6 7.5
fl (ppm)
MeO2C
SCF3
O
N
O
3ae
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-05T15:40:15
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 298.9 C
======= CHANNEL f1 =======
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
0.99
1.29
1.17
2.00
2.59
2.57
1.28
1.41
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
fl (ppm)

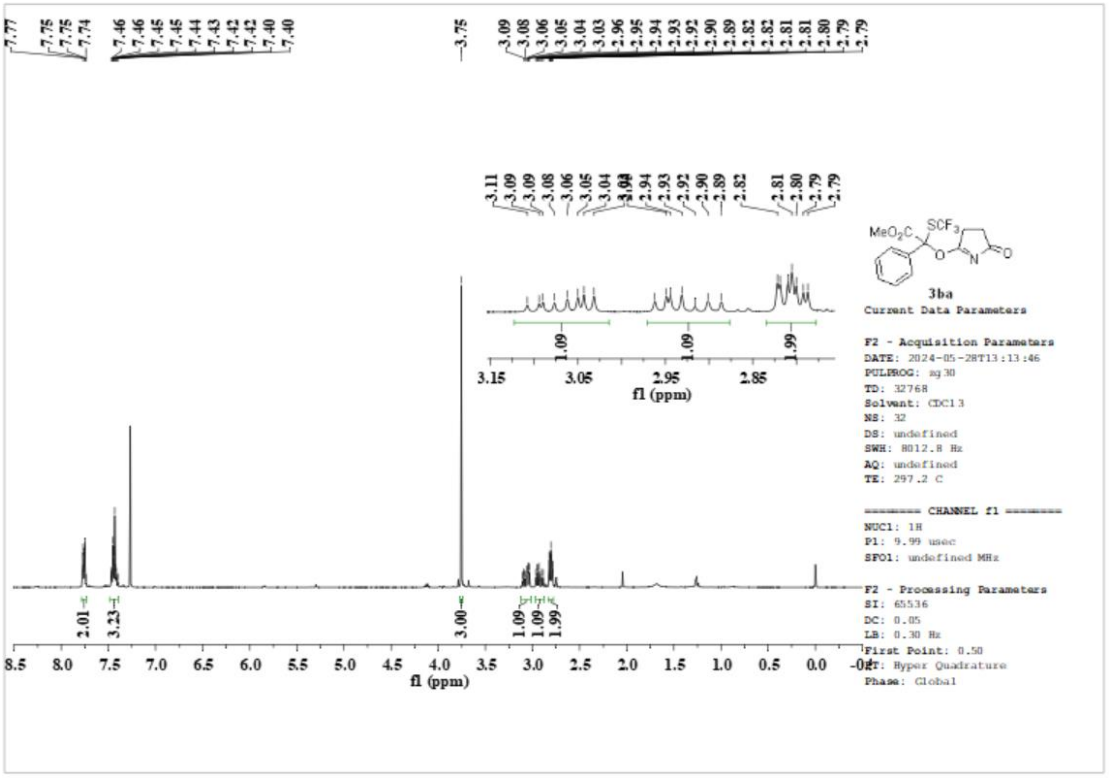

185.96
167.93
164.18
134.35
130.06
129.83
129.65
129.00
127.96
127.74
127.22
124.02
123.64
90.49
52.89
136.44
134.35
133.63
132.32
130.06
129.65
129.00
127.96
127.74
127.22
124.02
123.64
136 134 132 130 128 126 124
fl (ppm)
MeO2C
SCF3
O
N
O
3ae
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-05T15:45:54
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 299.4 C
======= CHANNEL f1 =======
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Manual
Ph0: -123.14
Ph1: 37.50
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
fl (ppm)

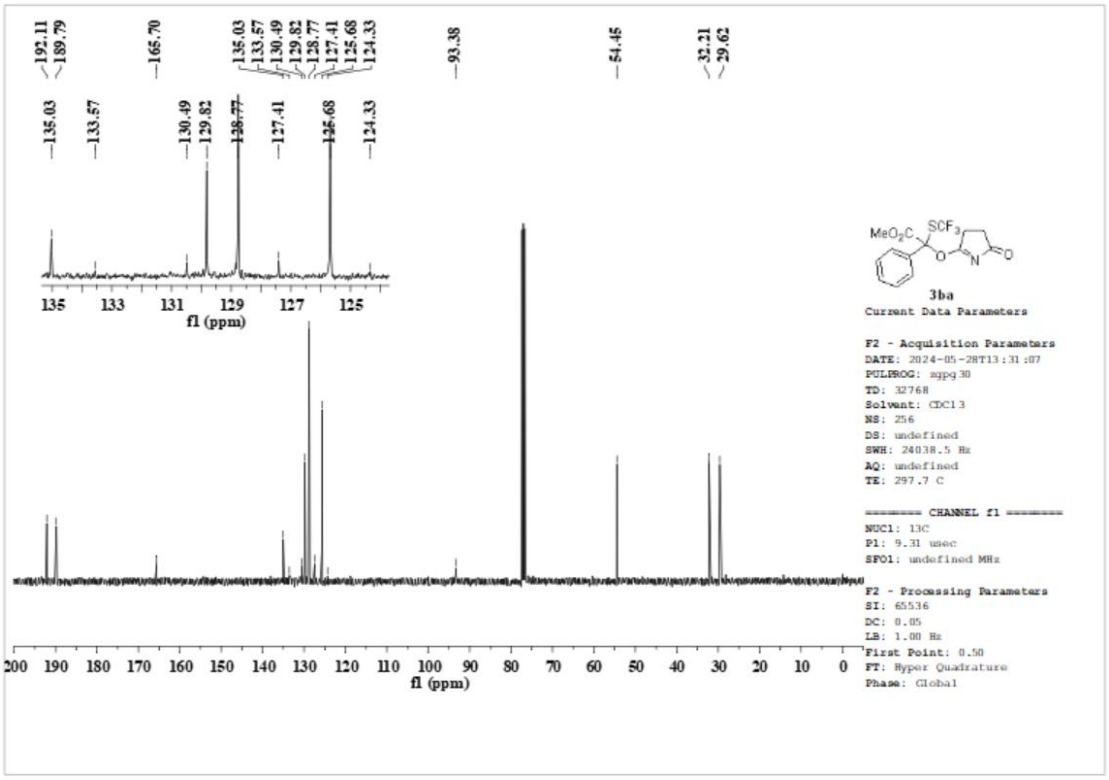

-31.06
MeO2C
SCF3
O
N
O
3ae
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-05T15:41:57
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 298.9 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65
f1 (ppm)

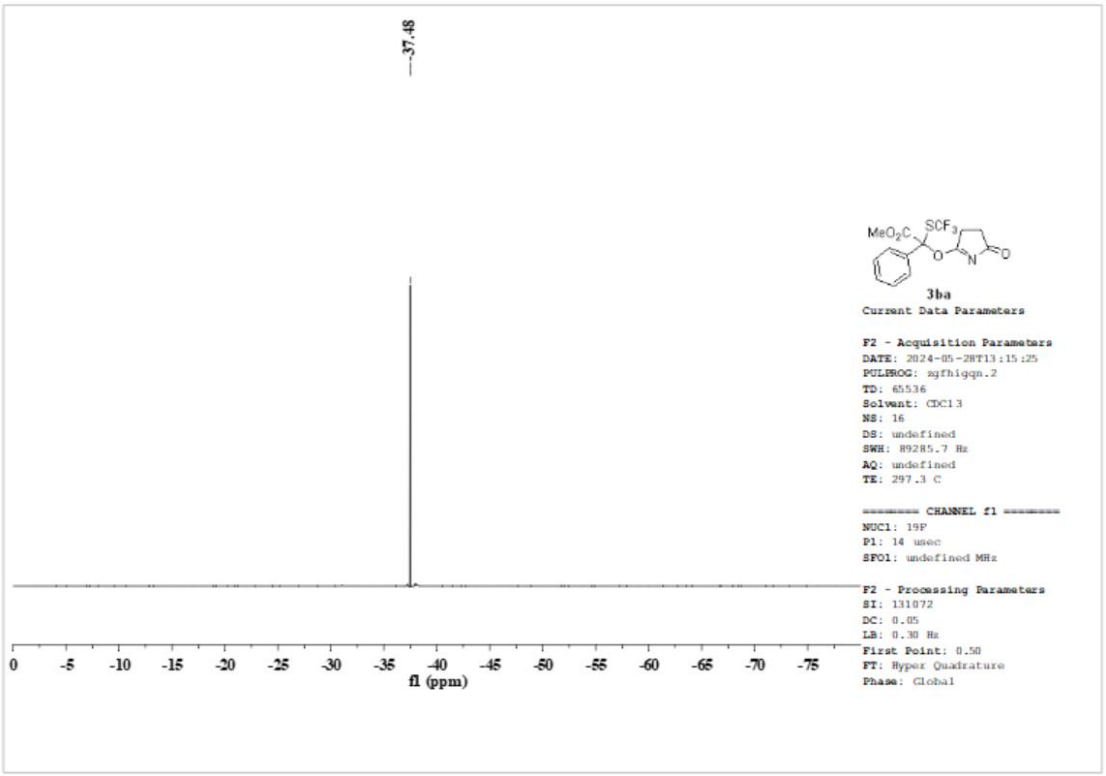

8.58
8.58
7.88
7.76
7.70
7.37
7.36
7.36
-3.96
7.88
7.88
7.87
7.86
7.77
7.76
7.76
7.74
7.71
7.70
1.82
1.98
1.24
7.90 7.85 7.80 7.75 7.70
f1 (ppm)
MeO2C
SCF3
O
N
O
S
3ap
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-05T16:03:20
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 299.1 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
1.00
1.82
1.98
1.24
1.17
3.09
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

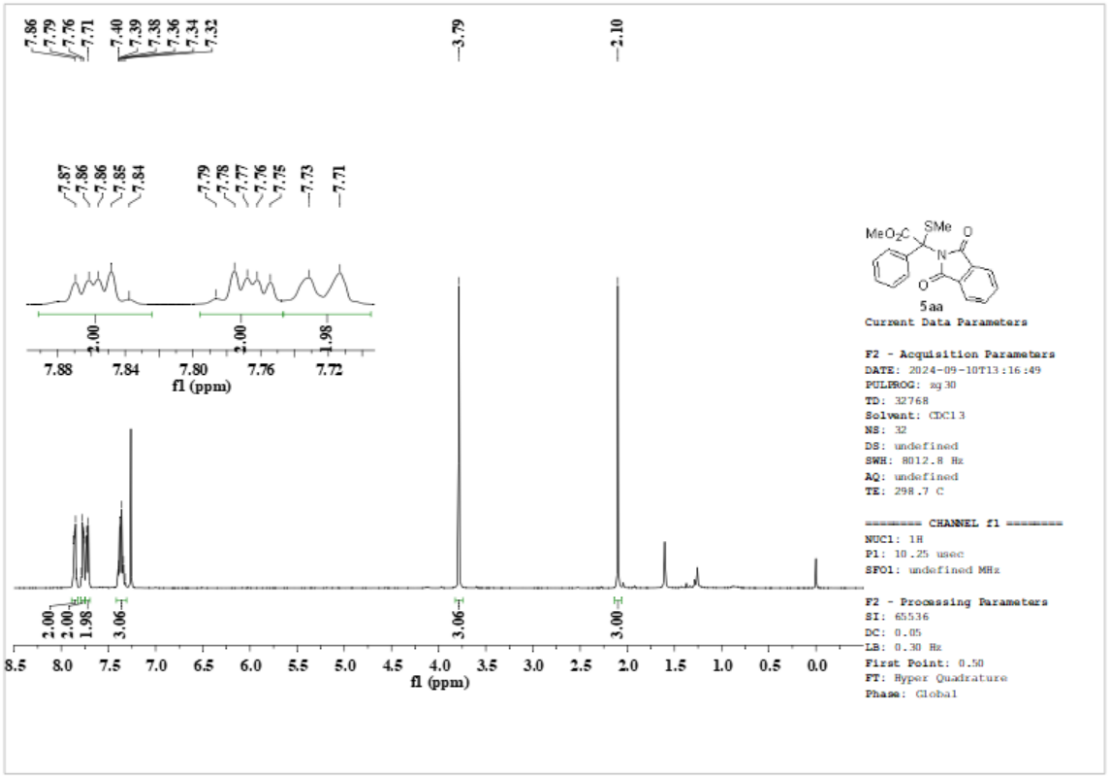

177.70
167.83
162.70
137.95
137.59
134.35
133.04
132.67
130.00
127.92
126.71
123.64
85.52
53.01
fl (ppm)
MeO2C
SCF3
3ap
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-05T16:20:51
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 299.5 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
GC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

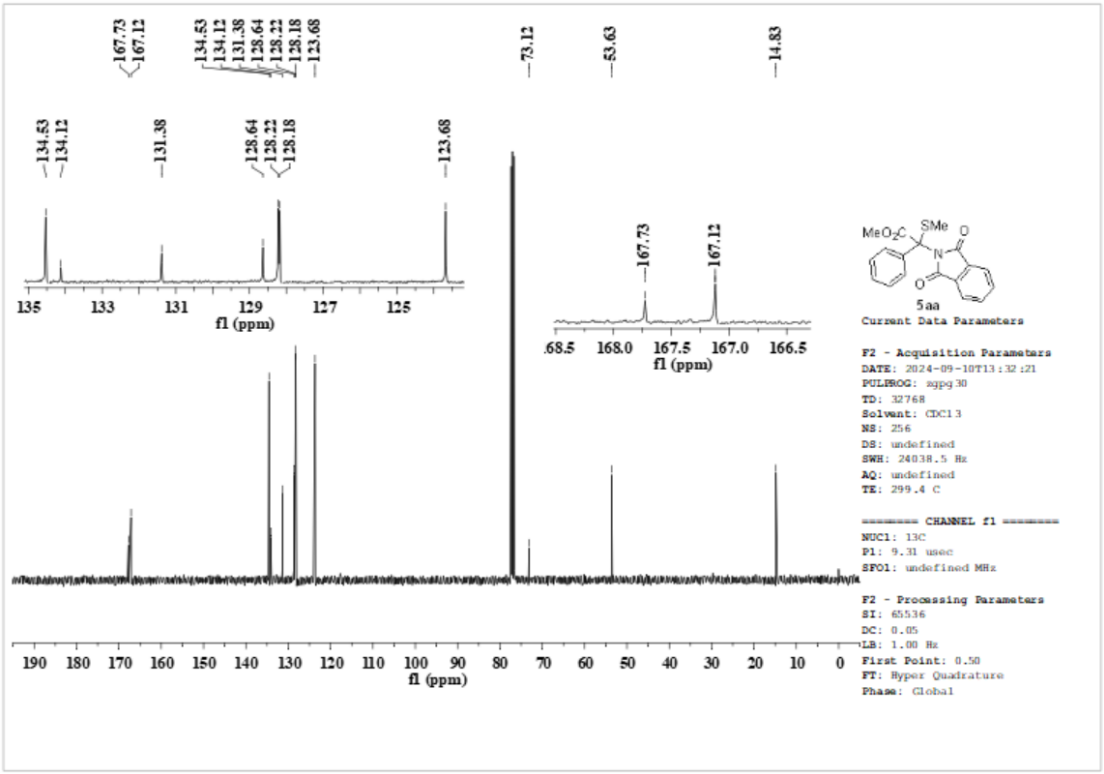

31.06
fl (ppm)
MeO2C
SCF3
3ap
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-07-05T16:05:06
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 299.1 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
GC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global

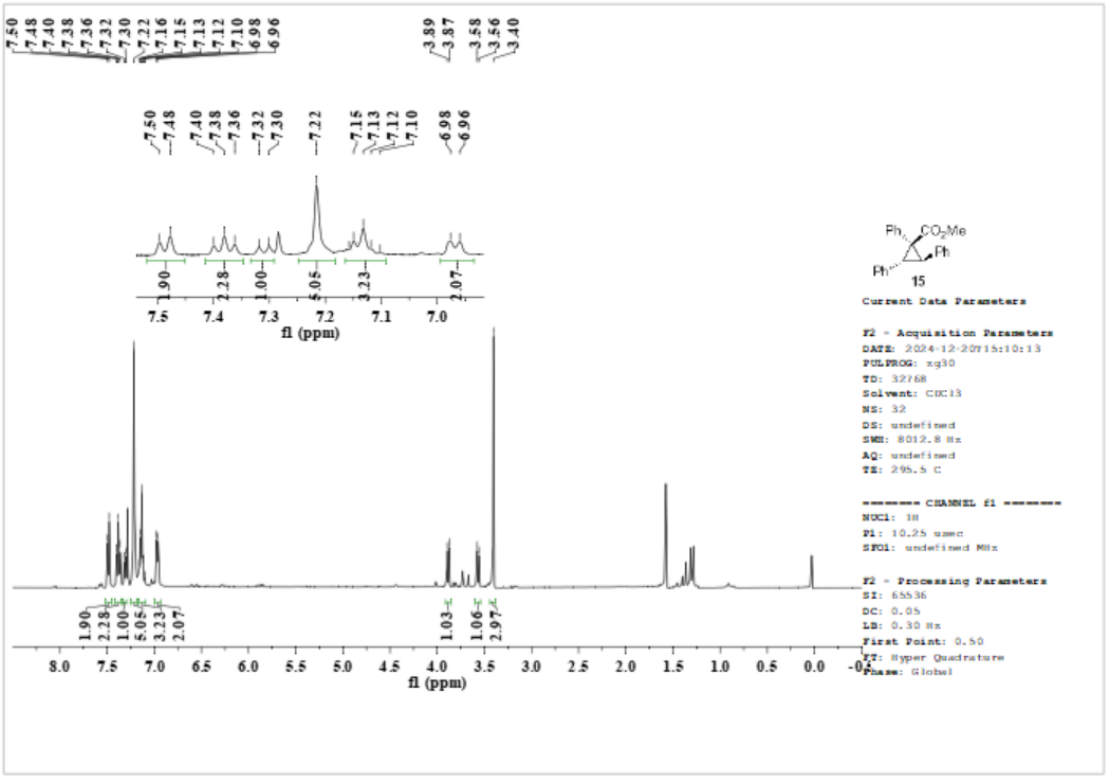

3ba
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-28T13:13:46
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 297.2 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 9.99 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)

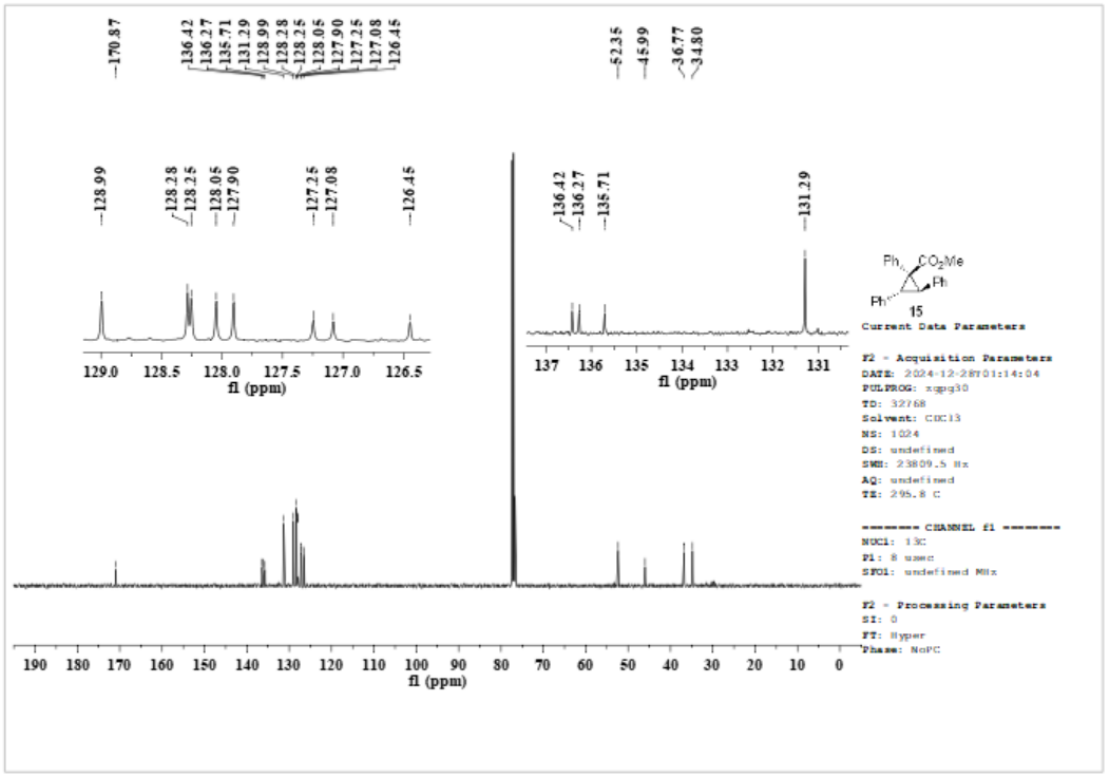

3ba
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-28T13:31:07
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 297.7 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)

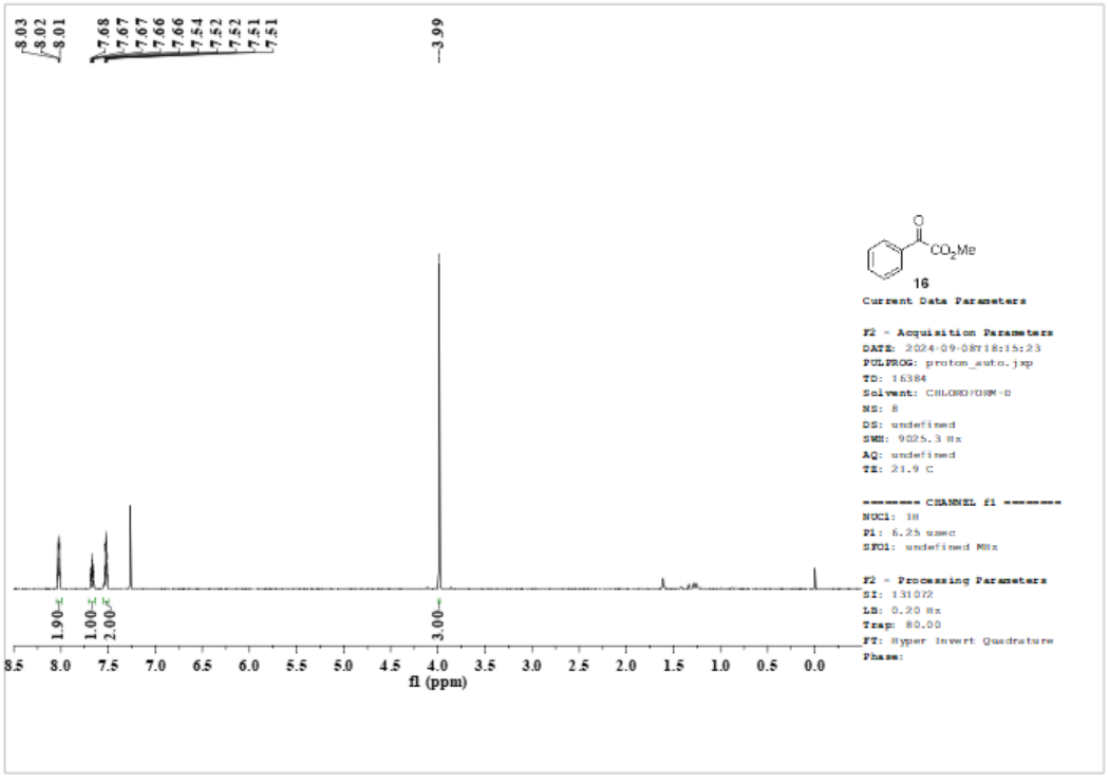

-37.48
MeO2C
SCF3
O
N
O
3ba
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-05-28T13:15:25
PULPROG: zgfhigqn.2
TD: 65536
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 89285.7 Hz
AQ: undefined
TE: 297.3 C
======== CHANNEL f1 ========
NUC1: 19F
P1: 14 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75
f1 (ppm)

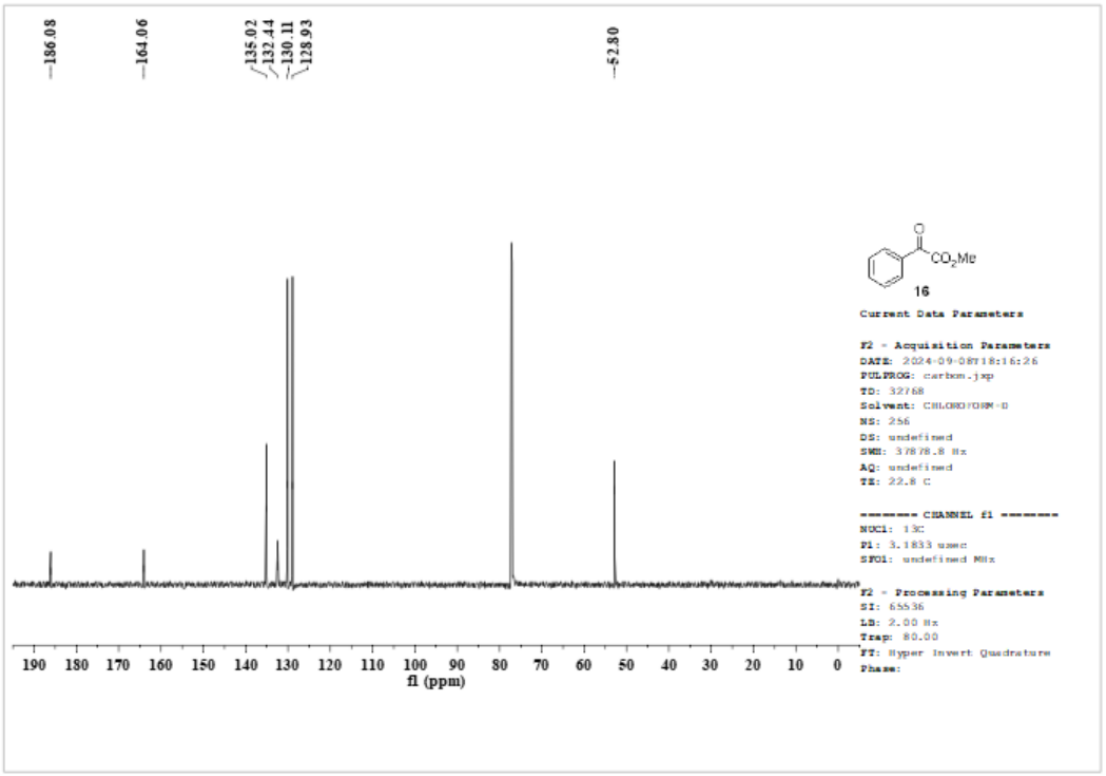

7.86
7.79
7.76
7.71
7.40
7.39
7.38
7.36
7.34
7.32
3.79
2.10
7.87
7.86
7.86
7.85
7.84
7.79
7.78
7.77
7.76
7.75
7.73
7.71
2.00
2.00
1.98
7.88
7.84
7.80
7.76
7.72
f1 (ppm)
MeO2C
SMe
O
N
O
5aa
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-09-10T13:16:49
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 298.7 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 10.25 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
2.00
2.00
1.98
3.06
3.06
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

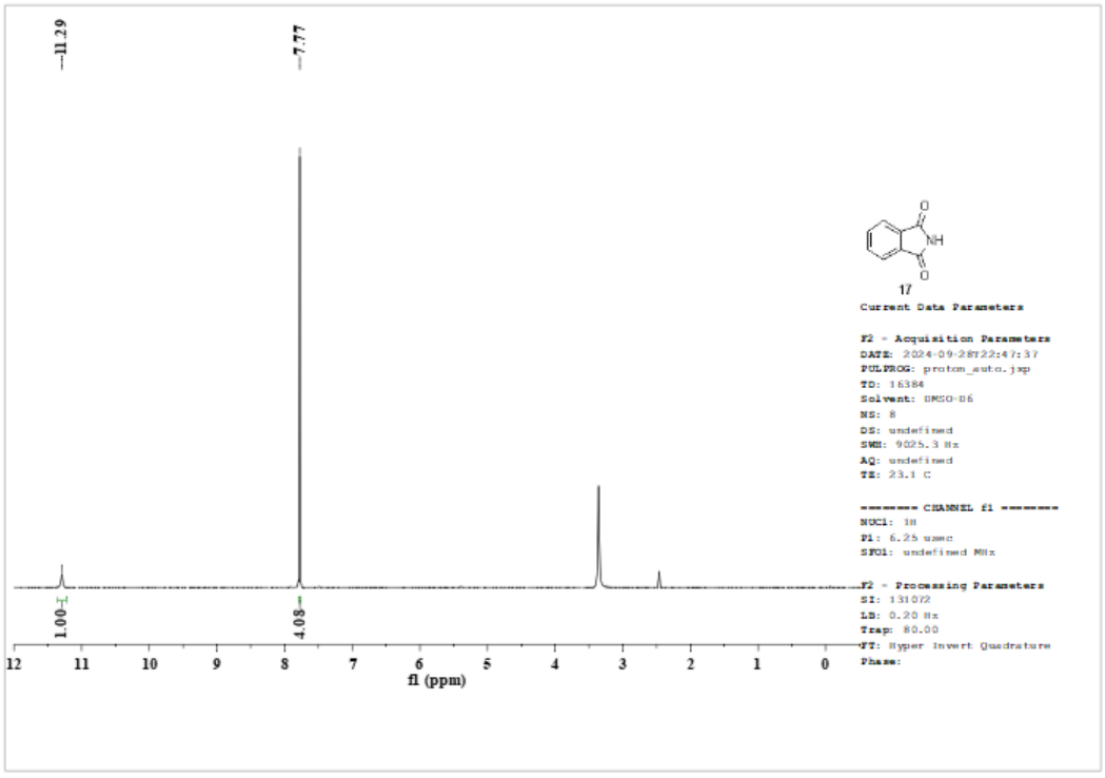

167.73
167.12
134.53
134.12
131.38
128.64
128.22
128.18
123.68
73.12
53.63
14.83
MeO2C
SMe
O
N
O
5aa
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-09-10T13:32:21
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 256
DS: undefined
SWH: 24038.5 Hz
AQ: undefined
TE: 299.4 C
======== CHANNEL f1 ========
NUC1: 13C
P1: 9.31 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 1.00 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)

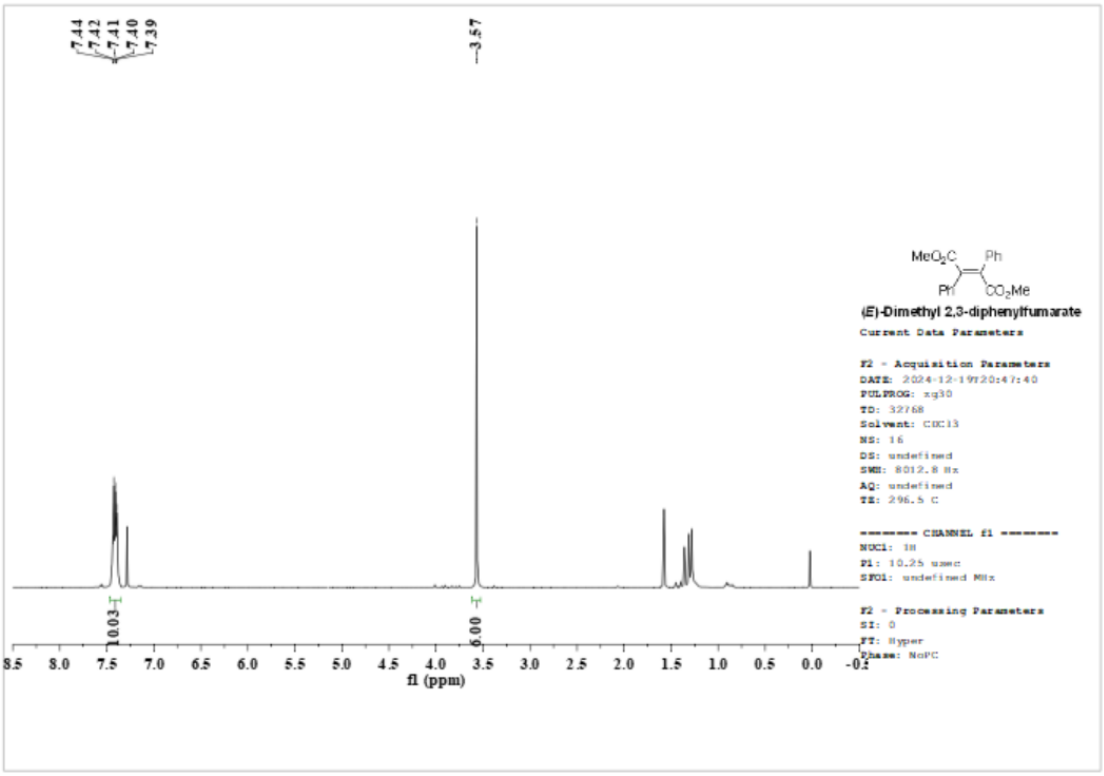

7.50
7.48
7.40
7.38
7.36
7.32
7.30
7.22
7.16
7.15
7.13
7.12
7.10
6.98
6.96
3.89
3.87
3.58
3.56
3.40
1.90
2.28
1.00
5.05
3.23
2.07
1.03
1.06
2.97
Ph
CO2Me
Ph
Ph
15
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-12-20T15:10:13
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 32
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 295.5 C
======== CHANNEL f1 ========
NUC1: 1H
P1: 10.25 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
DC: 0.05
LB: 0.30 Hz
First Point: 0.50
FT: Hyper Quadrature
Phase: Global
f1 (ppm)

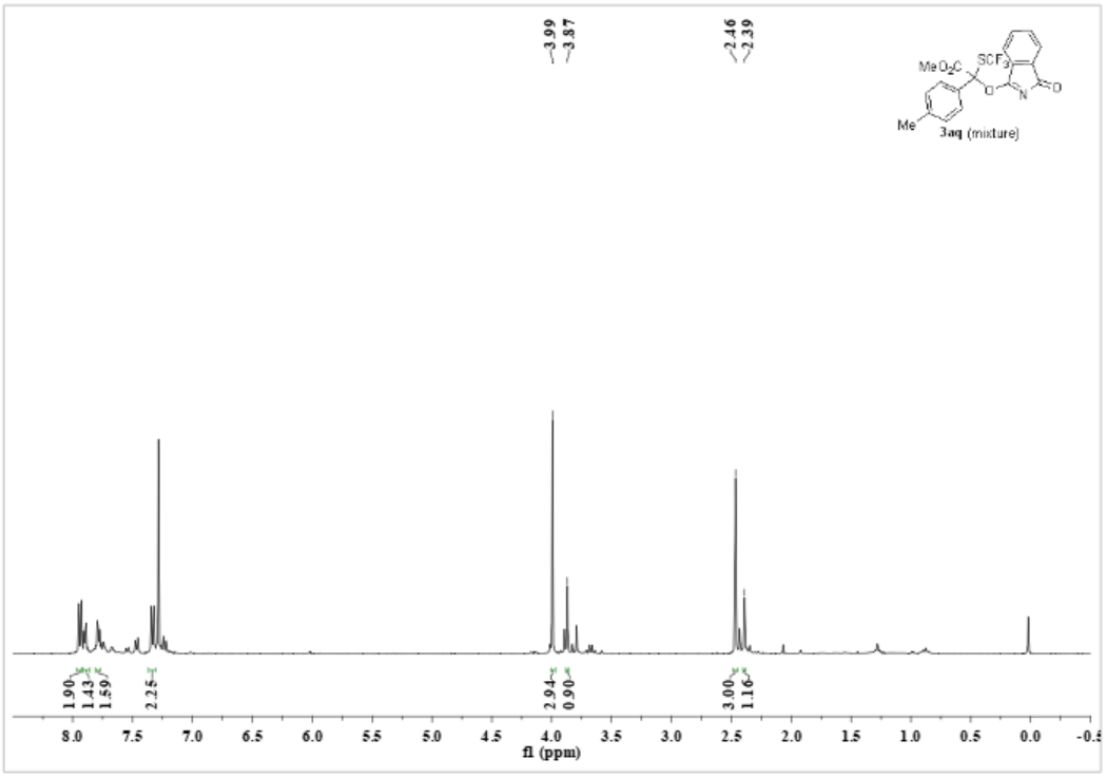

170.87
136.42
136.27
135.71
131.29
128.99
128.28
128.25
128.05
127.90
127.25
127.08
126.45
52.35
45.99
36.77
34.80
Ph
CO2Me
Ph
Ph
15
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-12-28T01:14:04
PULPROG: zgpg30
TD: 32768
Solvent: CDCl3
NS: 1024
DS: undefined
SWH: 23809.5 Hz
AQ: undefined
TE: 295.8 C
======= CHANNEL f1 =======
NUC1: 13C
P1: 8 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 0
FT: Hyper
Phase: NoPC
f1 (ppm)
f1 (ppm)
f1 (ppm)

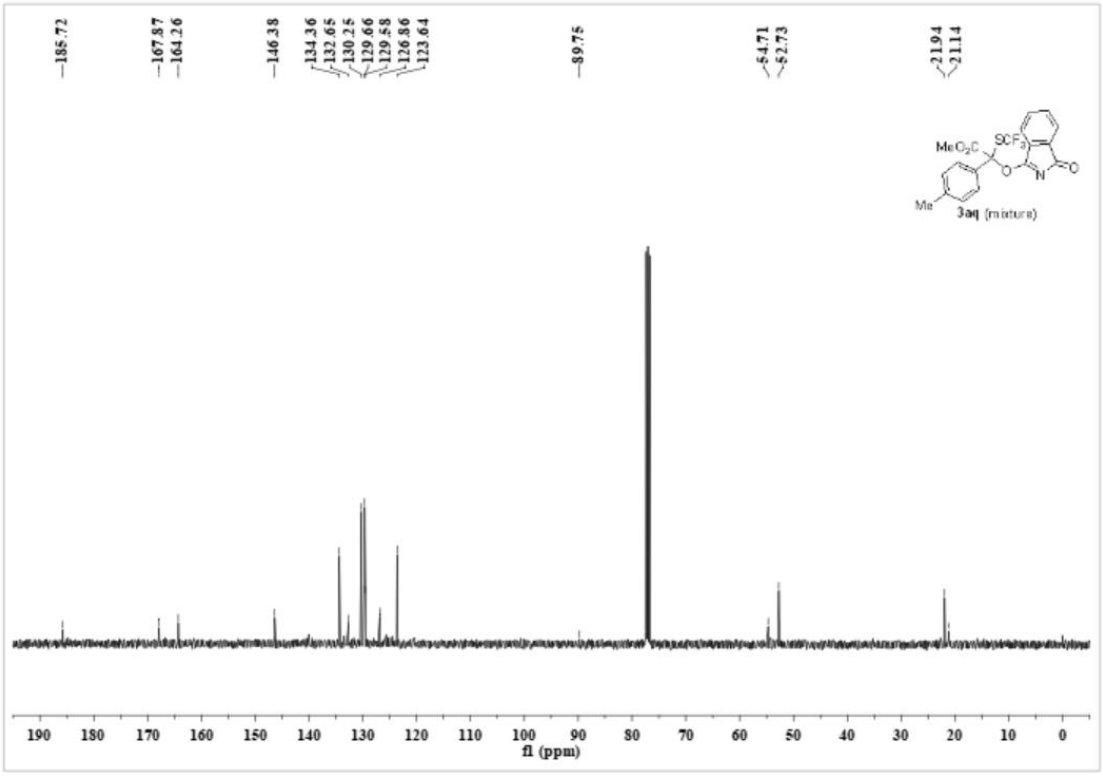

8.03
8.02
8.01
7.68
7.67
7.67
7.66
7.66
7.54
7.52
7.52
7.51
7.51
3.99
CO2Me
O
16
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-09-08T18:15:23
PULPROG: proton_auto.jxp
TD: 16384
Solvent: CHLOROFORM-D
NS: 8
DS: undefined
SWH: 9025.3 Hz
AQ: undefined
TE: 21.9 C
======= CHANNEL f1 =======
NUC1: 1H
P1: 6.25 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
LB: 0.20 Hz
Trap: 80.00
FT: Hyper Invert Quadrature
Phase:
1.90
1.00
2.00
3.00
f1 (ppm)

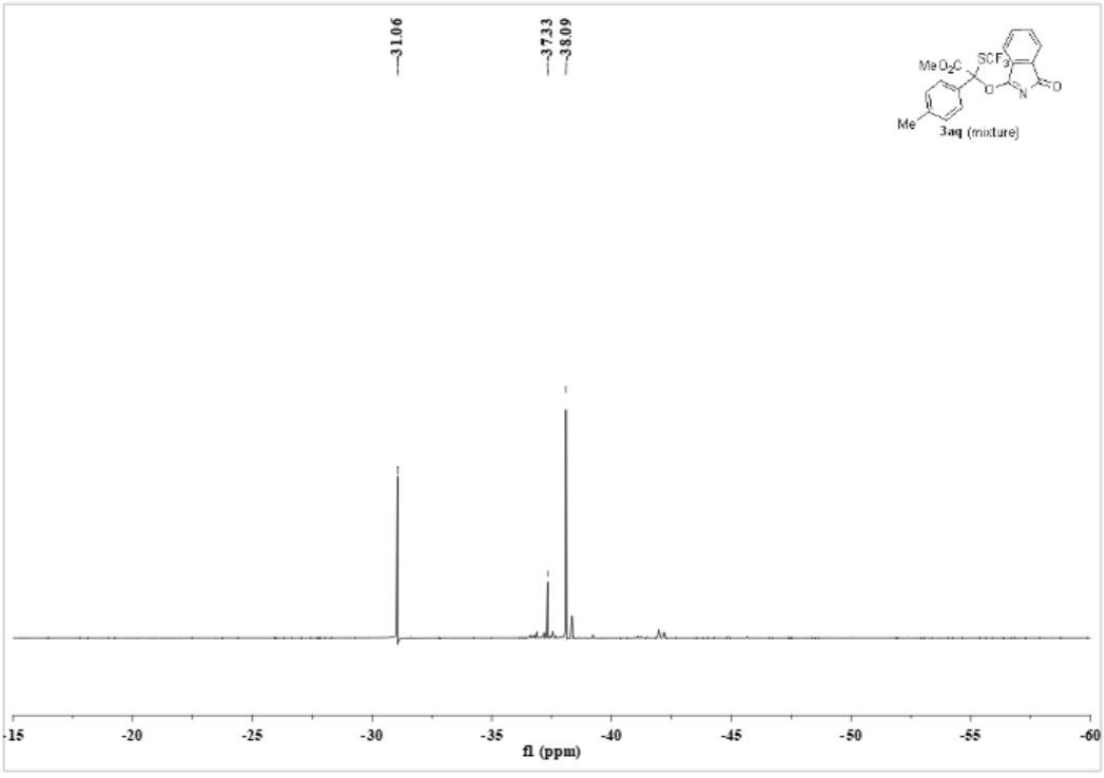

186.08
164.06
135.02
132.44
130.11
128.93
52.80
CO2Me
16
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-09-08T18:16:26
PULPROG: carbon.jxp
TD: 32768
Solvent: CHLOROFORM-D
NS: 256
DS: undefined
SWH: 37878.8 Hz
AQ: undefined
TE: 22.8 C
------- CHANNEL f1 -------
NUC1: 13C
P1: 3.1833 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 65536
LB: 2.00 Hz
Trap: 80.00
FT: Hyper Invert Quadrature
Phase:
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

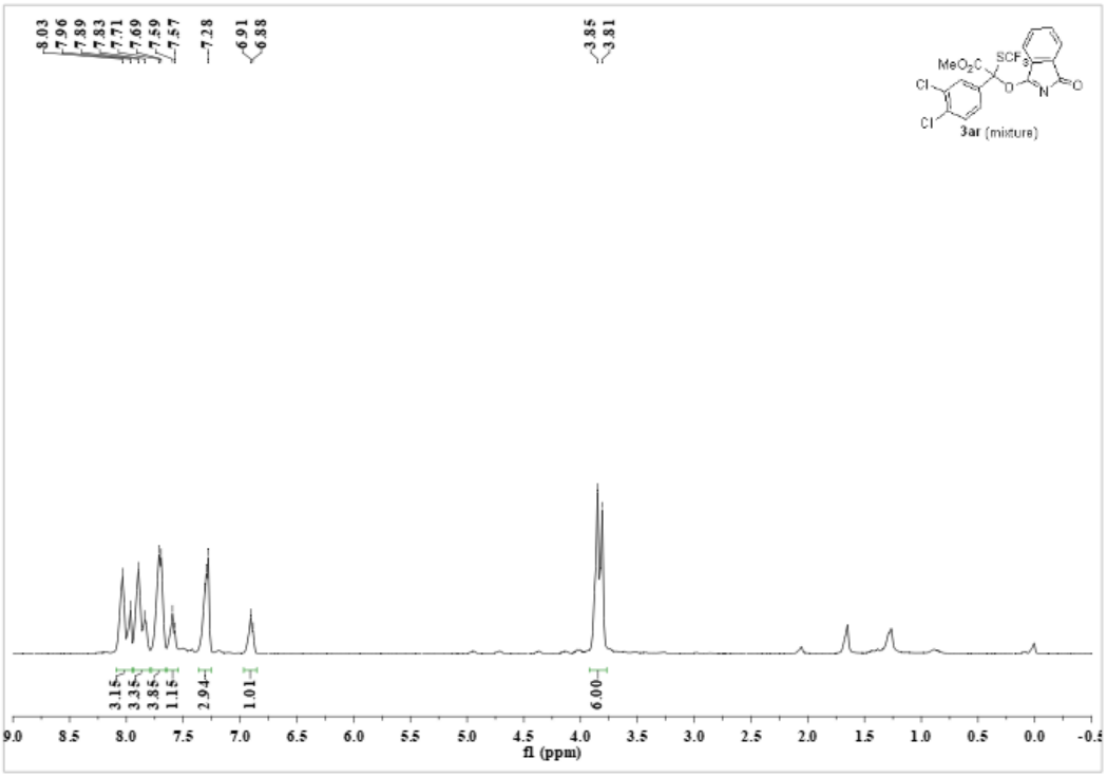

11.29
7.77
NH
17
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-09-28T22:47:37
PULPROG: proton_auto.jxp
TD: 16384
Solvent: DMSO-D6
NS: 8
DS: undefined
SWH: 9025.3 Hz
AQ: undefined
TE: 23.1 C
------- CHANNEL f1 -------
NUC1: 1H
P1: 6.25 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 131072
LB: 0.20 Hz
Trap: 80.00
FT: Hyper Invert Quadrature
Phase:
1.00
4.08
12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

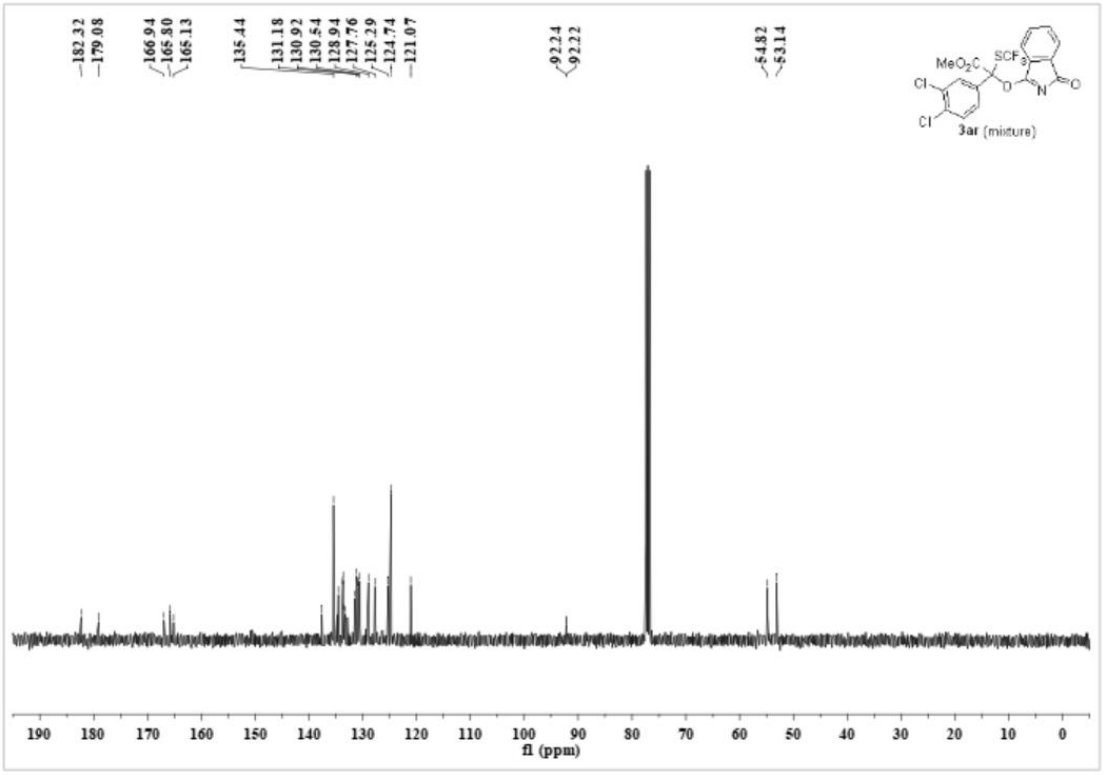

7.44
7.42
7.41
7.40
7.39
3.57
10.03
6.00
f1 (ppm)
(E)-Dimethyl 2,3-diphenylfumarate
Current Data Parameters
F2 - Acquisition Parameters
DATE: 2024-12-19T20:47:40
PULPROG: zg30
TD: 32768
Solvent: CDCl3
NS: 16
DS: undefined
SWH: 8012.8 Hz
AQ: undefined
TE: 296.5 C
======= CHANNEL f1 =======
NUC1: 1H
P1: 10.25 usec
SFO1: undefined MHz
F2 - Processing Parameters
SI: 0
FT: Hyper
Phase: ManPC

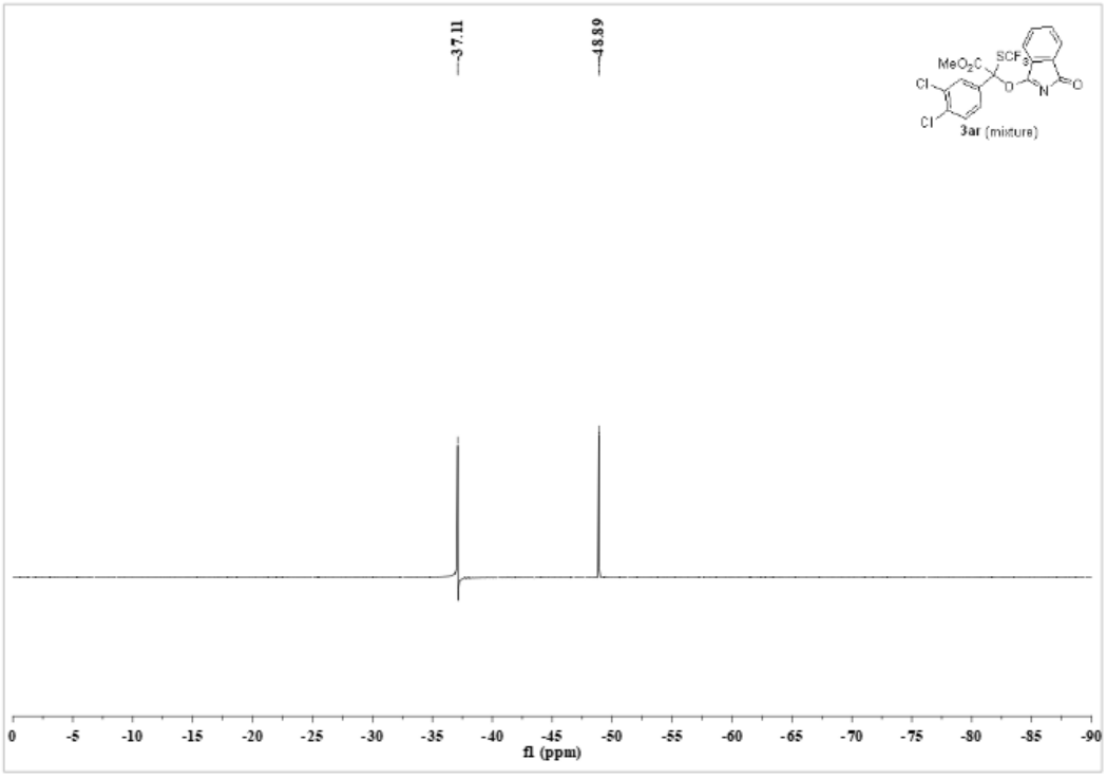

3.99
3.87
2.46
2.39
1.90
1.43
1.59
2.25
2.94
0.90
3.00
1.16
f1 (ppm)
3aq (mixture)

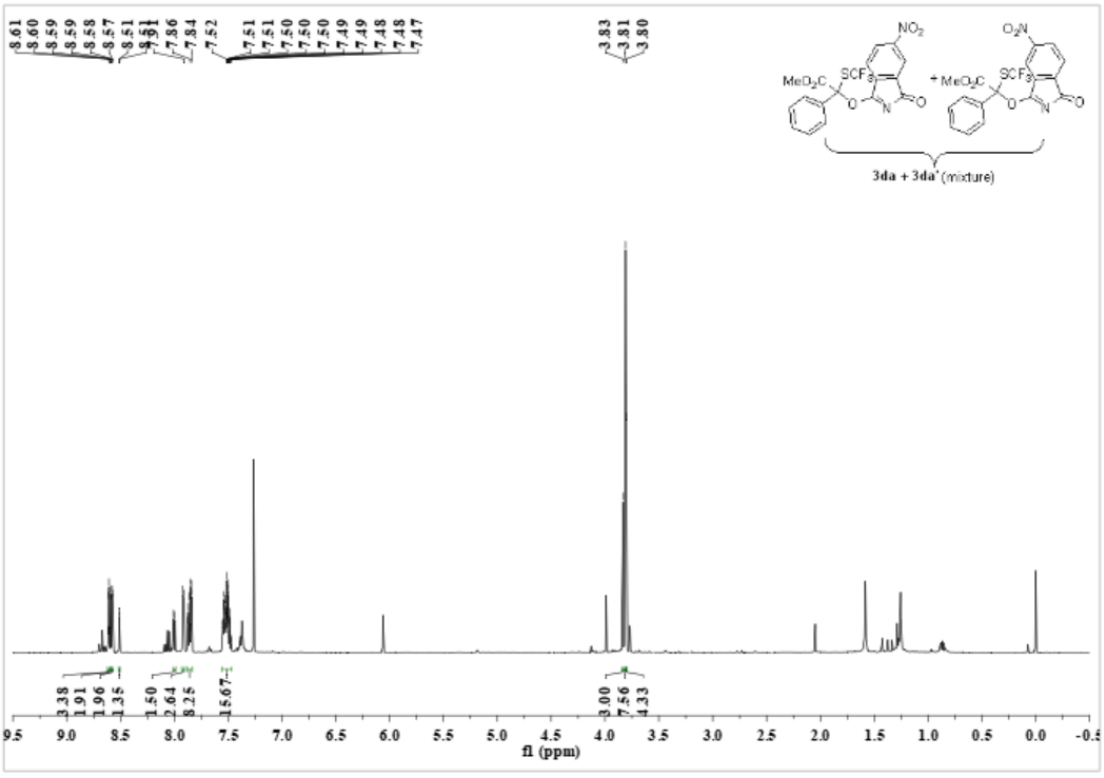

185.72
167.87
164.26
146.38
134.36
132.65
130.25
129.66
129.58
126.86
123.64
89.75
54.71
52.73
21.94
21.14
MeO2C
SCF3
O
N
O
Me
3aq (mixture)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

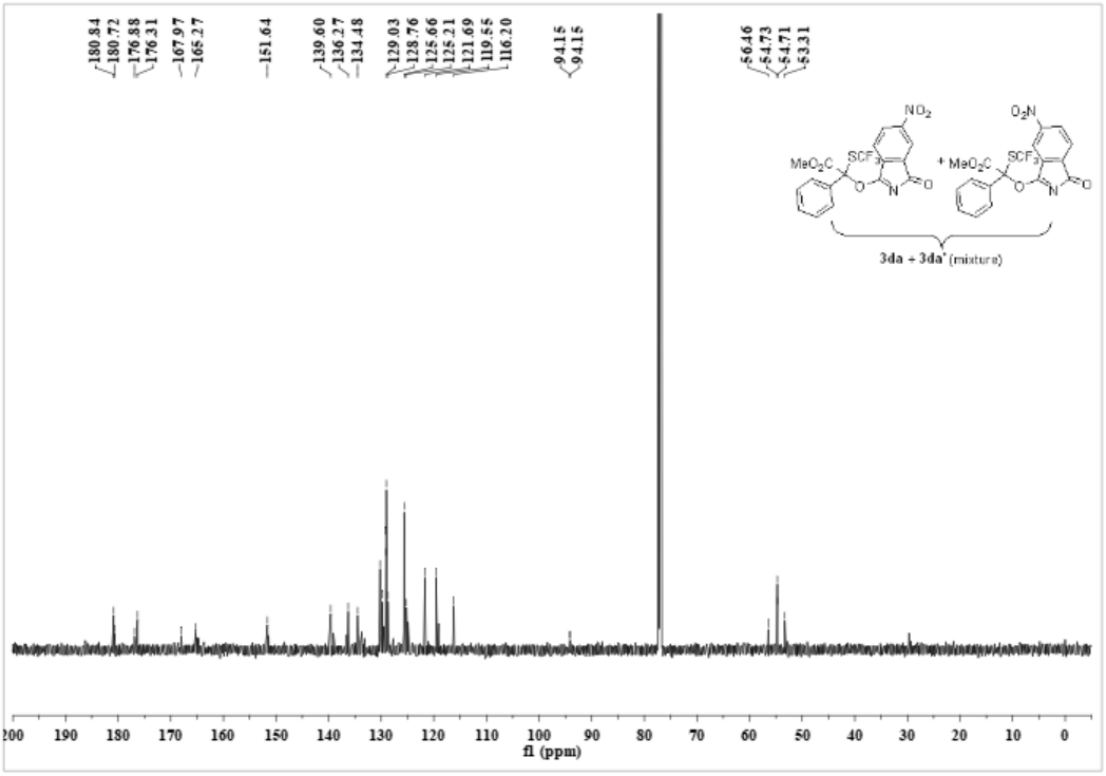

31.06
37.33
38.09
MeO2C
SCF3
O
N
O
Me
3aq (mixture)
-15 -20 -25 -30 -35 -40 -45 -50 -55 -60
f1 (ppm)

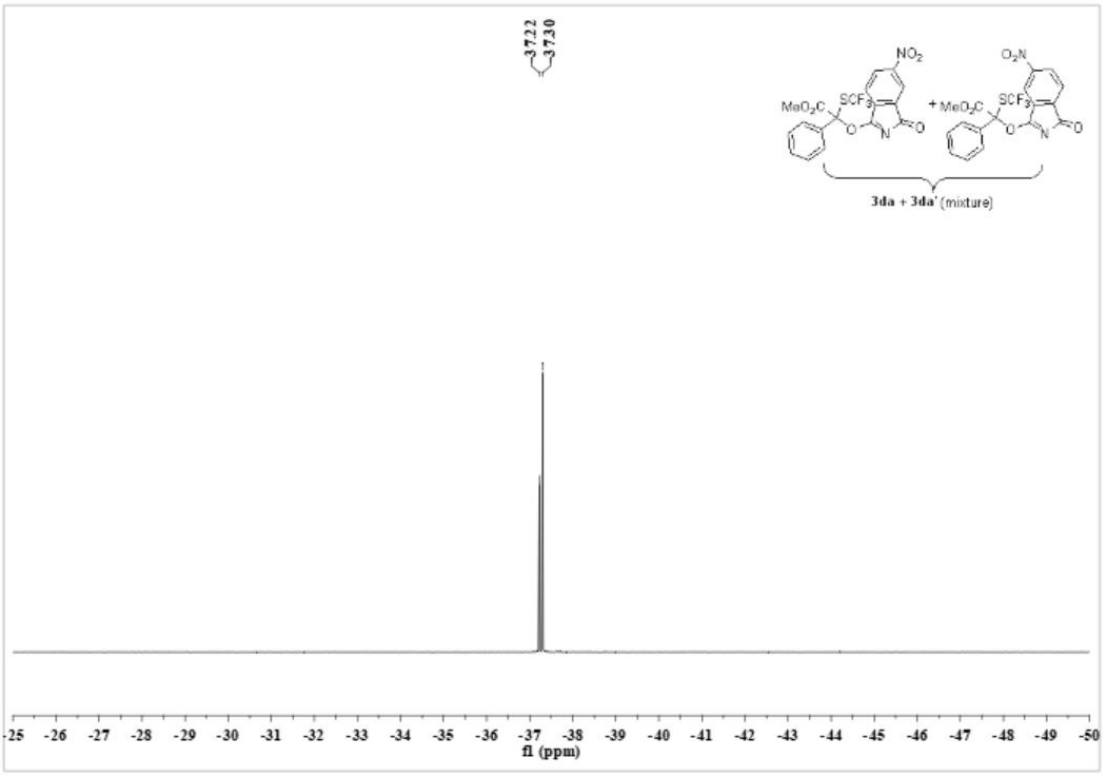

8.03
7.96
7.89
7.83
7.71
7.69
7.59
7.57
7.28
6.91
6.88
3.85
3.81
3ar (mixture)
3.15
3.35
3.85
1.15
2.94
1.01
6.00
fl (ppm)


182.32
179.08
166.94
165.80
165.13
135.44
131.18
130.92
130.54
128.94
127.76
125.29
124.74
121.07
92.24
92.22
54.82
53.14
3ar (mixture)
fl (ppm)


-37.11
-48.89
3ar (mixture)
fl (ppm)


3da + 3da' (mixture)
fl (ppm)


180.84
180.72
176.88
176.31
167.97
165.27
151.64
139.60
136.27
134.48
129.03
128.76
125.66
125.21
121.69
119.55
116.20
94.15
94.15
56.46
54.73
54.71
53.31
NO2
O2N
MeO2C
SCF3
O
N
O
+ MeO2C
SCF3
O
N
O
3da + 3da' (mixture)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-37.22
-37.30
NO2
O2N
MeO2C
SCF3
O
N
O
+ MeO2C
SCF3
O
N
O
3da + 3da' (mixture)
-25 -26 -27 -28 -29 -30 -31 -32 -33 -34 -35 -36 -37 -38 -39 -40 -41 -42 -43 -44 -45 -46 -47 -48 -49 -50
f1 (ppm)