Supporting Information

$B(C_6F_5)_3$-catalyzed Wolff rearrangement/[2 + 2] cascade cyclization of α-diazoketones with diazenes to access aza-β-lactams

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1. General Information

Solvents: Unless otherwise indicated, solvents were obtained from commercial suppliers. Ether (Et_2O), Dibutyl ether (DBE), Dichloromethane (DCM), N,N-dimethylformamide (DMF), Acetonitrile (CH_3CN), and Toluene were stored over activated 4Å molecular sieves following drying procedures.

Reagents: All reagents were used as purchased from commercial suppliers. Unless noted otherwise, commercially available chemicals were used without further purification.

Nuclear Magnetic Resonance Spectroscopy: All NMR spectra were collected at 298 K on Bruker 400 MHz spectrometer in 5 mm diameter NMR tubes. ^{1}H chemical shifts are reported relative to proteo-solvent signals ($CDCl_3$, δ = 7.26 ppm). Data are reported as: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, td = triplet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets), coupling constants (Hz), integration and assignment. $^{13}C\{^{1}H\}$ chemical shifts are reported relative to proteo-solvent signals ($CDCl_3$, δ = 77.00 ppm). ^{19}F NMR spectra were measured at 376 MHz and $CFCl_3$ (-63.2 ppm) was used as an external standard.

Mass Spectrometry: High resolution mass spectra (HRMS) were recorded with an Thermo Scientific™ Q Exactive™ Plus Orbitrap LC-MS/MS System by ESI on a quadrupole mass analyzer.

X-ray Crystallography: Single crystal X-ray crystallographic data were collected on a Bruker D8 QUEST diffractometer using Cu (60W, Diamond, $\mu K\alpha$ = 12.894 mm^{-1}) micro-focus X-ray sources at 161 K. The structure was solved and refined using Full-matrix least-squares based on F^2 with program SHELXS and SHELXL[4] within OLEX2.

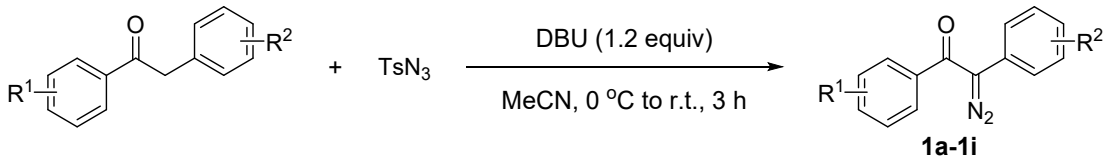
Thin Layer Chromatography: Thin layer chromatography (TLC) was performed on aluminum-backed pre-coated plates with silica gel 60 F254 with suitable solvent system and was visualized using UV fluorescence.

Column Chromatography: Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040-0.063 mm).

Heating: Oil bath served as the heat source for all conventionally heating reactions.

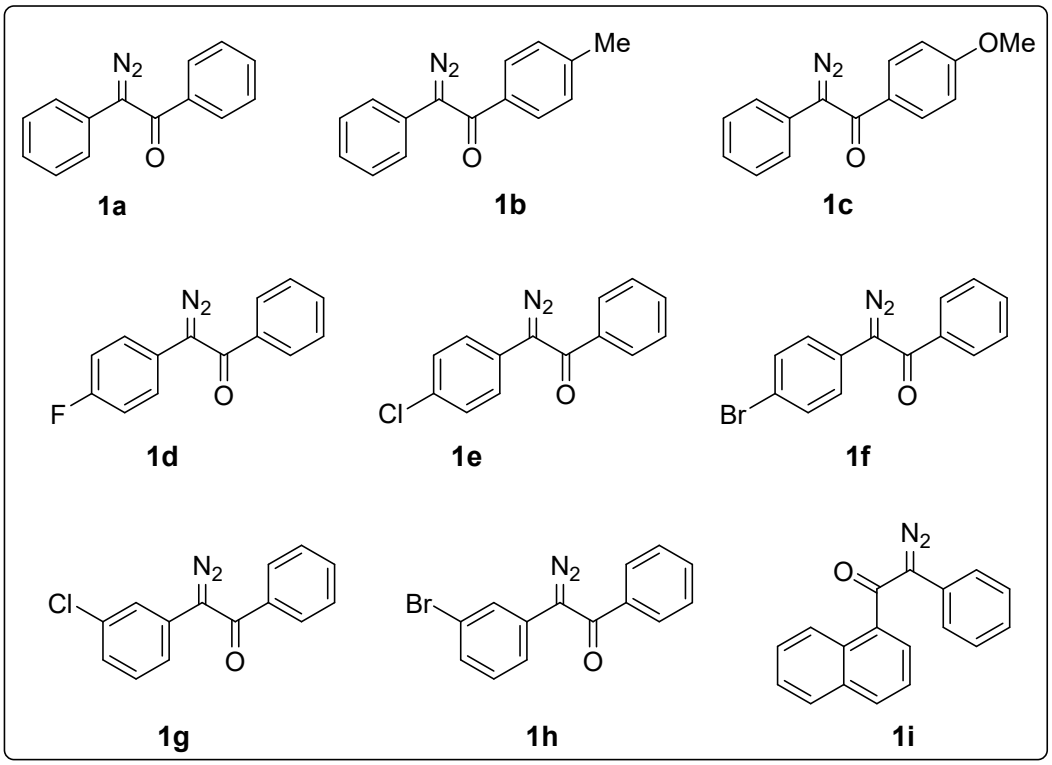
2. General Procedure for the Synthesis of Substrates α-aryldiazoketones 1a-1i

1a-1i are known compounds and were synthesized according to procedures reported in the relevant literatures.[1-4]

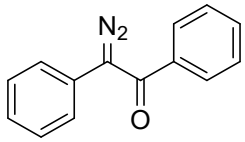


1a-1i

To a solution of β-ketone (10 mmol, 1.0 equiv) and 4-methylbenzenesulfonyl azide (12 mmol, 2.37 g, 3.0 mL, 1.2 equiv) in CH_3CN at 0 °C was added DBU (12 mmol, 1.83 g, 1.8 mL, 1.2 equiv) dropwise under nitrogen. The resulting solution was stirred at 0 °C for 3 h and slowly brought to room temperature. Upon completion as indicated by thin layer chromatography (TLC), the reaction was quenched with water, extracted with ethyl acetate, and dried over anhydrous Na_2SO_4. The reaction mixture was concentrated under reduced pressure, and the crude material was purified by column chromatography to give pure products **1a-1i**.



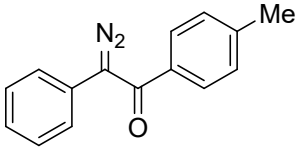
2-diazo-1,2-diphenylethan-1-one (**1a**)



1a

Following the general procedure, **1a** was obtained as orange solid in (8.6 mmol, 1.9 g) 87% yield. R_f = 0.32 (silica gel, PE:EA = 25:1). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.71-7.59 (m, 2H), 7.57-7.47 (m, 3H), 7.47-7.40 (m, 4H), 7.32-7.27 (m, 1H). The **^{1}H NMR** data are consistent with those previously reported.

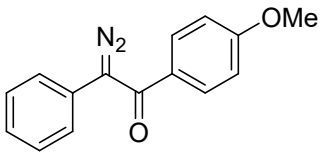
2-diazo-2-phenyl-1-(*p*-tolyl)ethan-1-one (**1b**)



1b

Following the general procedure, **1b** was obtained as orange solid in (8.2 mmol, 1.9 g) 82% yield. R_f = 0.32 (silica gel, PE:EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 1.6 Hz, 1H), 7.51 (d, *J* = 2.0 Hz, 1H), 7.48-7.43 (m, 2H), 7.41-7.37 (m, 2H), 7.27-7.23 (m, 1H), 7.21 (d, *J* = 7.6 Hz, 2H), 2.39 (s, 3H). The **^{1}H NMR** data are consistent with those previously reported.

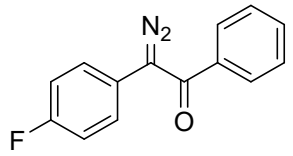
2-diazo-1-(4-methoxyphenyl)-2-phenylethan-1-one (**1c**)



1c

Following the general procedure, **1c** was obtained as orange solid in (7.9 mmol, 2.0 g) 79% yield. R_f = 0.32 (silica gel, PE:EA = 15:1). **1b** was obtained in 81% yield as orange solid. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68-7.54 (m, 2H), 7.46-7.36 (m, 4H), 7.28-7.21 (m, 1H), 6.91-6.87 (m, 2H), 3.84 (s, 3H). The **^{1}H NMR** data are consistent with those previously reported.

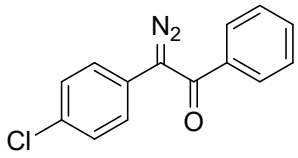
2-diazo-2-(4-fluorophenyl)-1-phenylethan-1-one (**1d**)



1d

Following the general procedure, **1d** was obtained as orange solid in (8.5 mmol, 2.0 g) 85% yield. R_f = 0.32 (silica gel, PE:EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57-7.51 (m, 2H), 7.48-7.44 (m, 2H), 7.43-7.38 (m, 4H), 7.30-7.24 (m, 1H). The **^{1}H NMR** data are consistent with those previously reported.

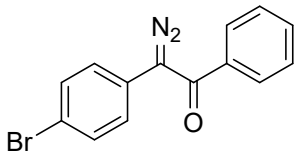
2-(4-chlorophenyl)-2-diazo-1-phenylethan-1-one (**1e**)



1e

Following the general procedure, **1e** was obtained as orange solid in (8.5 mmol, 2.1 g) 85% yield. R_f = 0.32 (silica gel, PE:EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68-7.56 (m, 2H), 7.55-7.48 (m, 1H), 7.48-7.35 (m, 6H). The **^{1}H NMR** data are consistent with those previously reported.

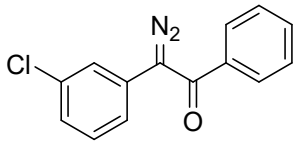
2-(4-bromophenyl)-2-diazo-1-phenylethan-1-one (**1f**)



1f

Following the general procedure, **1f** was obtained as orange solid in (8.0 mmol, 2.4 g) 80% yield. R_f = 0.32 (silica gel, PE:EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.64-7.59 (m, 2H), 7.55-7.50 (m, 3H), 7.46-7.42 (m, 2H), 7.40-7.36 (m, 2H). The **^{1}H NMR** data are consistent with those previously reported.

2-(3-chlorophenyl)-2-diazo-1-phenylethan-1-one (**1g**)

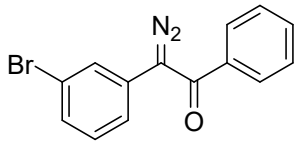


1g

Following the general procedure, **1g** was obtained as yellow solid in (7.8 mmol, 2.0 g)

78% yield. R_f= 0.32 (silica gel, PE:EA = 20:1). The **^{1}H NMR** data are consistent with those previously reported.

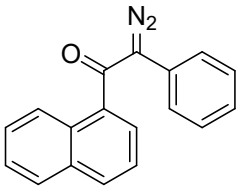
2-(3-bromophenyl)-2-diazo-1-phenylethan-1-one (**1h**)



1h

Following the general procedure, **1h** was obtained as yellow solid in (7.3 mmol, 2.2 g) 73% yield. R_f= 0.32 (silica gel, PE:EA = 20:1). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71 (t, *J* = 2.0 Hz, 1H), 7.64-7.61 (m, 2H), 7.55-7.51 (m, 1H), 7.47-7.37 (m, 5H). The **^{1}H NMR** data are consistent with those previously reported.

2-diazo-1-(naphthalen-1-yl)-2-phenylethan-1-one (**1i**)

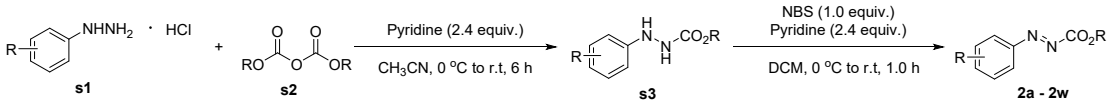


1i

Following the general procedure, **1i** was obtained as orange solid in (6.9 mmol, 1.9 g) 69% yield. R_f= 0.32 (silica gel, PE:EA = 20:1). **^{1}H NMR** (400 MHz, Chloroform-d) δ 8.30-8.10 (m, 1H), 7.98-7.91 (m, 2H), 7.66-7.55 (m, 5H), 7.52-7.43 (m, 3H), 7.29 (t, *J* = 7.2 Hz, 1H). The **^{1}H NMR** data are consistent with those previously reported.

3. Preparation of aryldiazenes 2a-2w

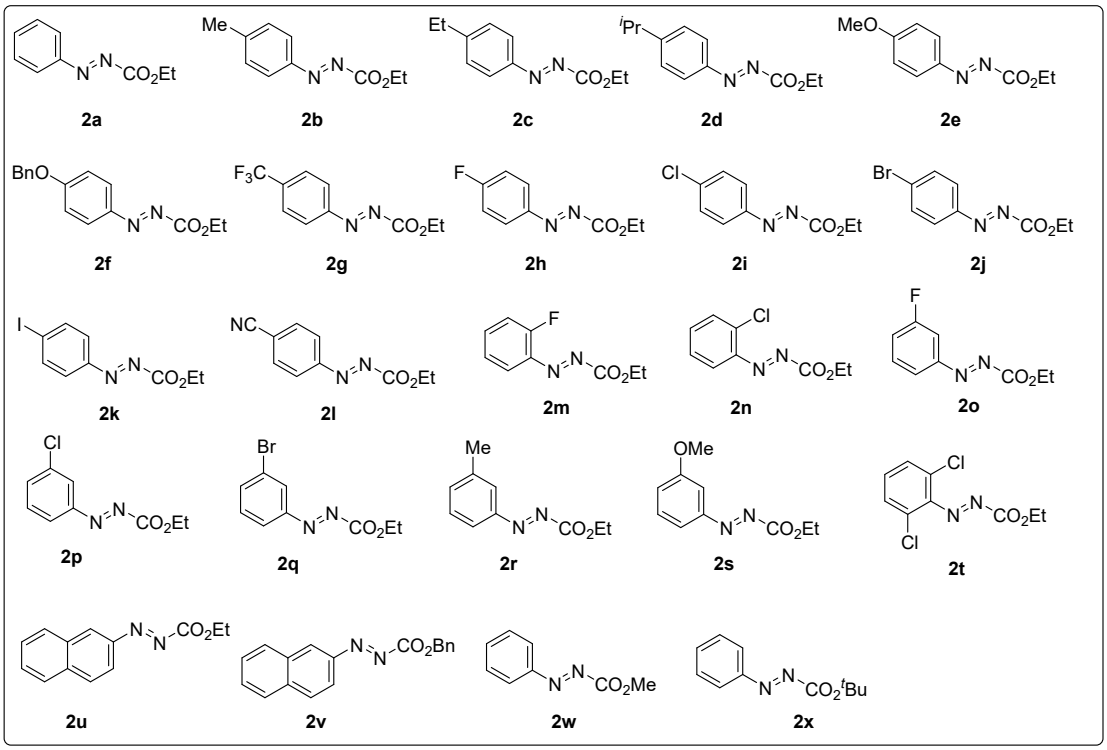
2a-**2w** are known compounds and were synthesized according to procedures reported in the relevant literatures.[5-8]



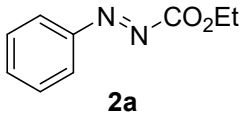
To a 10 mL round-bottom flask equipped with a magnetic stir bar, substituted phenylhydrazine hydrochloride **s1** (5.0 mmol), pyridine (0.97 mL, 12.0 mmol, 2.4 equiv.), and CH_3CN (5.0 mL) were added. The solution was cooled to 0 °C, and dialkyl pyrocarbonate **s2** (1.1 equiv, 5.5 mmol) was added dropwise. The reaction mixture was stirred for 1 h at room temperature. The reaction was diluted by adding CH_2Cl_2 and

washed with 4 M HCl aqueous solution. Two layers were separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated on rotary evaporator. The residue was purified by recrystallization using CH_2Cl_2 and hexane to give alkyl 2-phenylhydrazinecarboxylates **s3**.

A round bottom flask equipped with a magnetic stir bar was charged with above 2-phenylhydrazinecarboxylates **s3** (4.0 mmol, 1.0 equiv.) in DCM (5 mL), pyridine (0.65 mL, 8 mmol, 2.0 equiv.) was added. The solution was cooled to 0 °C and NBS (0.71 g, 4.0 mmol, 1.0 equiv.) was added under stirring. The reaction mixture was stirred for 25 min at 0 °C, and then for 30 min at room temperature. The reaction mixture was then concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel eluted with petroleum ether/EtOAc to afford the corresponding aryldiazenes **2a-2w**.



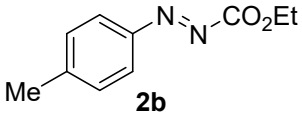
ethyl (*E*)-2-phenyldiazene-1-carboxylate (**2a**)



2a

Following the general procedure, **2a** was obtained as wine-red liquid in 97% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98-7.87 (m, 2H), 7.59-7.48 (m, 3H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

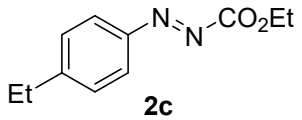
ethyl (E)-2-(p-tolyl)diazene-1-carboxylate (**2b**)



2b

Following the general procedure, **2b** was obtained as wine-red liquid in 95% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86-7.82 (m, 2H), 7.31 (dd, *J* = 8.0 Hz, 2H), 4.50 (q, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

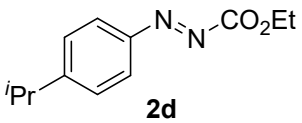
ethyl (*E*)-2-(4-ethylphenyl)diazene-1-carboxylate (**2c**)



2c

Following the general procedure, **2c** was obtained as wine-red liquid in 96% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89-7.85 (m, 2H), 7.36 - 7.32 (m, 2H), 4.51 (q, *J* = 6.8 Hz, 2H), 2.73 (q, *J* = 7.6 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H), 1.27 (t, *J* = 7.6 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

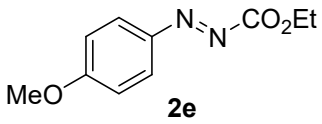
ethyl (*E*)-2-(4-isopropylphenyl)diazene-1-carboxylate (**2d**)



2d

Following the general procedure, **2d** was obtained as wine-red liquid in 94% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89-7.86 (m, 2H), 7.38-7.35 (m, 2H), 4.50 (q, *J* = 7.2 Hz, 2H), 2.98 (p, *J* = 6.8 Hz, 1H), 1.45 (t, *J* = 7.2 Hz, 3H), 1.27 (d, *J* = 7.2 Hz, 6H).

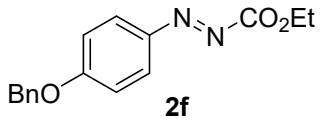
ethyl (*E*)-2-(4-methoxyphenyl)diazene-1-carboxylate (**2e**)



2e

Following the general procedure, **2e** was obtained as wine-red liquid in 90% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.23-8.19 (m, 2H), 7.27-7.23 (m, 2H), 4.75 (q, *J* = 7.2 Hz, 2H), 4.14 (s, 3H), 1.71 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

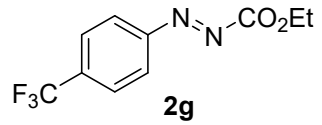
ethyl (*E*)-2-(4-(benzyloxy)phenyl)diazene-1-carboxylate (**2f**)



2f

Following the general procedure, **2f** was obtained as orange solid in 89% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00-7.92 (m, 2H), 7.46-7.34 (m, 5H), 7.10-7.06 (m, 2H), 5.17 (s, 2H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.47 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

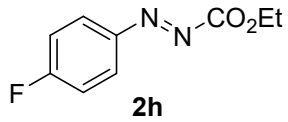
ethyl (*E*)-2-(4-(trifluoromethyl)phenyl)diazene-1-carboxylate (**2g**)



2g

Following the general procedure, **2g** was obtained as wine-red liquid in 83% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06-7.96 (m, 2H), 7.82-7.77 (m, 2H), 4.53 (q, *J* = 6.8 Hz, 2H), 1.47 (t, *J* = 7.6 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

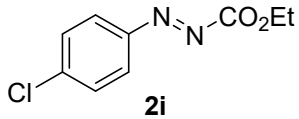
ethyl (E)-2-(4-fluorophenyl)diazene-1-carboxylate (**2h**)



2h

Following the general procedure, **2h** was obtained as wine-red liquid in 85% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02-7.91 (m, 2H), 7.23-7.16 (m, 2H), 4.50 (q, *J* = 7.2 Hz, 2H), 1.45 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

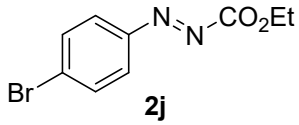
ethyl (*E*)-2-(4-chlorophenyl)diazene-1-carboxylate (**2i**)



2i

Following the general procedure, **2i** was obtained as dark brown solid in 87% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90-7.84 (m, 2H), 7.52-7.47 (m, 2H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

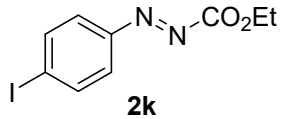
ethyl (*E*)-2-(4-bromophenyl)diazene-1-carboxylate (**2j**)



2j

Following the general procedure, **2j** was obtained as dark brown solid in 88% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 6.8 Hz, 2H), 7.66 (d, *J* = 2.0 Hz ,2H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 72 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported. The **^{1}H NMR** data are consistent with those previously reported.

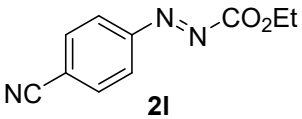
ethyl (*E*)-2-(4-iodophenyl)diazene-1-carboxylate (**2k**)



2k

Following the general procedure, **2k** was obtained as dark brown solid in 90% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93-7.84 (m, 2H), 7.68-7.59 (m, 2H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

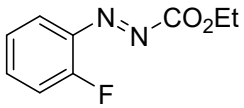
ethyl (*E*)-2-(4-cyanophenyl)diazene-1-carboxylate (**2l**)



2l

Following the general procedure, **2l** was obtained as wine-red solid in 77% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.86-7.81 (m, 2H), 4.53 (q, *J* = 7.2 Hz, 2H), 1.47 (t, *J* = 7.2 Hz, 3H).

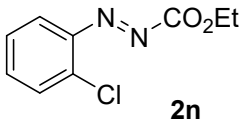
ethyl (E)-2-(2-fluorophenyl)diazene-1-carboxylate (**2m**)



2m

Following the general procedure, **2m** was obtained as wine-red liquid in 79% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71-7.64 (m, 1H), 7.60-7.54 (m, 1H), 7.31-7.25 (m, 1H), 7.21 (ddt, *J* = 1.2 Hz, 1H), 4.50 (q, *J* = 7.2 Hz, 2H), 1.45 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

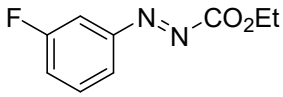
ethyl (*E*)-2-(2-chlorophenyl)diazene-1-carboxylate (**2n**)



2n

Following the general procedure, **2n** was obtained as wine-red liquid in 78% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (ddd, *J* = 1.4 Hz, 2H), 7.51-7.46 (m, 1H), 7.32 (ddd, *J* = 0.8 Hz, 1H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

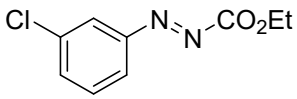
ethyl (*E*)-2-(3-fluorophenyl)diazene-1-carboxylate (**2o**)



2o

Following the general procedure, **2o** was obtained as wine-red liquid in 89% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.79 (ddd, *J* = 5.2 Hz, 1H), 7.59-7.48 (m, 2H), 7.31-7.26 (m, 1H), 4.52 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H).

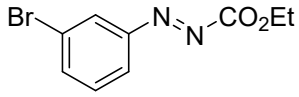
ethyl (*E*)-2-(3-chlorophenyl)diazene-1-carboxylate (**2p**)



2p

Following the general procedure, **2p** was obtained as wine-red liquid in 91% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.88-7.83 (m, 2H), 7.54 (ddd, *J* = 1.2 Hz, 1H), 7.48 (dd, *J* = 7.2 Hz, 1H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

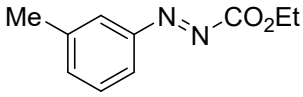
ethyl (*E*)-2-(3-bromophenyl)diazene-1-carboxylate (**2q**)



2q

Following the general procedure, **2q** was obtained as wine-red liquid in 92% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (t, *J* = 1.6 Hz, 1H), 7.90 (ddd, *J* = 0.8 Hz, 1H), 7.70 (ddd, *J* = 0.8 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 4.51 (q, *J* = 6.8 Hz, 2H), 1.46 (t, *J* = 7.2 Hz ,3H). The **^{1}H NMR** data are consistent with those previously reported.

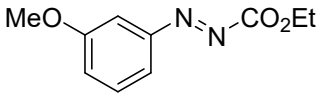
ethyl (*E*)-2-(m-tolyl)diazene-1-carboxylate (**2r**)



2r

Following the general procedure, **2r** was obtained as wine-red liquid in 94% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77-7.72 (m, 2H), 7.40 (dd, *J* = 5.6 Hz, 2H), 4.51 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

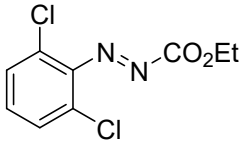
ethyl (*E*)-2-(3-methoxyphenyl)diazene-1-carboxylate (**2s**)



2s

Following the general procedure, **2s** was obtained as wine-red liquid in 92% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (ddd, *J* = 0.8 Hz, 1H), 7.46-7.38 (m, 2H), 7.14 (ddd, *J* = 0.8 Hz, 1H), 4.51 (q, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 1.46 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

ethyl (*E*)-2-(2,6-dichlorophenyl)diazene-1-carboxylate (**2t**)

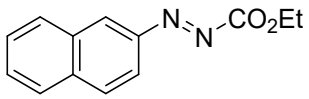


2t

Following the general procedure, **2t** was obtained as wine-red liquid in 51% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47-7.40 (m, 2H), 7.30-7.28 (m, 1H), 4.57 (q, *J* = 6.8 Hz,

2H), 1.50 (t, *J* = 7.2 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

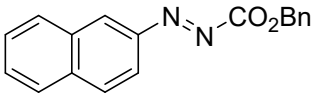
benzyl (*E*)-2-(naphthalen-2-yl)diazene-1-carboxylate (**2u**)



2u

Following the general procedure, **2u** was obtained as orange solid in 93% yield. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.67-8.55 (m, 1H), 8.04 (d, *J* = 8.0 Hz, 1H), 7.96-7.85 (m, 3H), 7.61 (dddd, *J* = 7.5 Hz, 2H), 4.55 (q, *J* = 7.0 Hz, 2H), 1.50 (t, *J* = 7.0 Hz, 3H). The **^{1}H NMR** data are consistent with those previously reported.

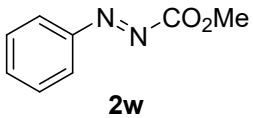
benzyl (*E*)-2-(naphthalen-2-yl)diazene-1-carboxylate (**2v**)



2v

Following the general procedure, **2v** was obtained as orange solid in 91% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.60 (s, 1H), 8.03 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.93-7.87 (m, 3H), 7.65-7.57 (m, 2H), 7.52 (dd, *J* = 8.0, 1.6 Hz, 2H), 7.45-7.37 (m, 3H), 5.50 (s, 2H). The **^{1}H NMR** data are consistent with those previously reported.

methyl (*E*)-2-phenyldiazene-1-carboxylate (**2w**)

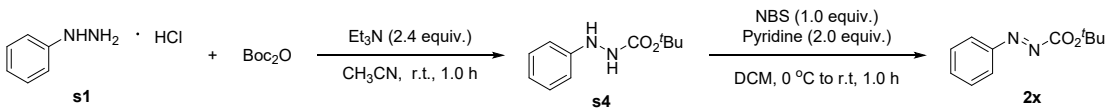


2w

Following the general procedure, **2w** was obtained as wine-red liquid in 94% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.95-7.91 (m, 2H), 7.61-7.50 (m, 3H), 4.07 (s, 3H). The **^{1}H NMR** data are consistent with those previously reported.

4. Preparation of 2x

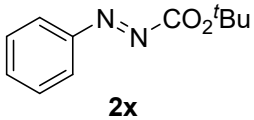
2x is known compound and was synthesized according to procedures reported in the relevant literatures.[8]



A 10 mL round-bottom flask, which was equipped with a magnetic stir bar and charged with phenylhydrazine hydrochloride (0.72 g, 5.0 mmol, 1.0 equiv.). After 5.0 mL of CH_3CN was added, Et_3N (1.6 mL, 12.0 mmol, 2.4 equiv.), di-tert-butyl dicarbonate (1.32 g, 6.0 mmol, 1.2 equiv.) were added in sequence. The reaction mixture was stirred for 1 h at room temperature. The reaction was diluted by adding CH_2Cl_2 and washed with a saturated aqueous solution of $NaHCO_3$. Two layers were separated, and the aqueous layer was extracted with CH_2Cl_2. The combined organic layer was dried over $MgSO_4$, filtered, and concentrated on rotary evaporator. The residue was purified by recrystallization using CH_2Cl_2 and hexane to give tert-butyl 2-phenylhydrazine-1-carboxylate **s4** as a white solid (1.02 g, 98% yield).

To a stirred solution of compound **s4** (1.02 g, 4.9 mmol, 1.0 equiv.) in DCM (10 mL), pyridine (0.8 mL, 9.8 mmol, 2.0 equiv.) was added. The solution was cooled to 0 ^oC and NBS (0.87 g, 4.9 mmol, 1.0 equiv.) was added under stirring. The reaction mixture was stirred for 25 min at 0 ^oC, and then for 30 mim at room temperature. The reaction mixture was then concentrated under reduced pressure. The crude product was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 50/1 to afford the compound **2x** as a wine-red oil (0.99 g, 98% yield).

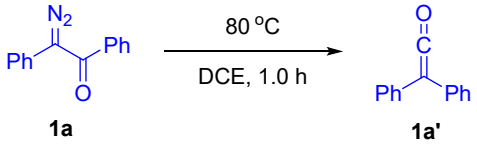
tert-butyl (*E*)-2-phenyldiazene-1-carboxylate (**2x**)



2x

Following the general procedure, **2x** was obtained as wine-red liquid in 98% yield. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 (dt, *J* = 1.2 Hz, 2H), 7.32-7.24 (m, 3H), 1.41 (s, 9H). The **^{1}H NMR** data are consistent with those previously reported.

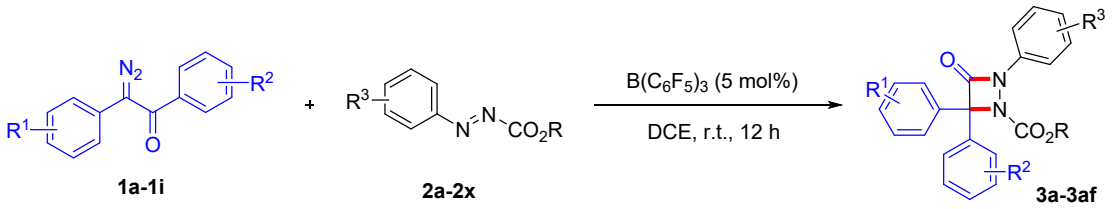
4. Preparation of 1a'



1a **1a'**

In an inert atmosphere glovebox, a solution of **1a** (0.3 mmol, 1.0 equiv) in 1,2-dichloroethane (DCE, 2 mL) was stirred at 80 °C for 1 h. The reaction mixture was then concentrated in vacuo to afford the desired product **1a'**. ^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.42-7.37 (m, 2H),7.34-7.14 (m, 3H). ^{13}**C NMR** (100 Hz, $CDCl_3$) δ 201.07, 130.76, 129.22, 127.67, 126.18.

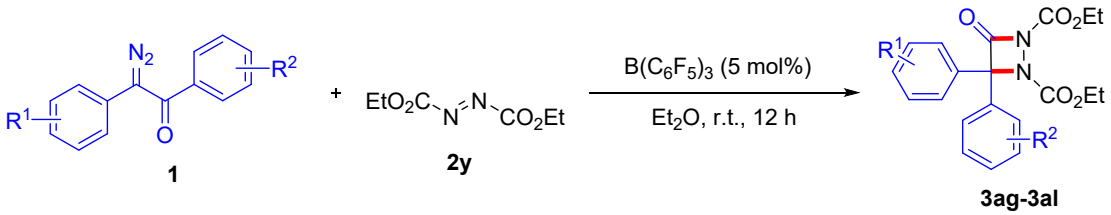
5. General procedure for the for [2+2] cycloadditions of α-aryldiazoketones with aryldiazenes.



1a-1i **2a-2x** **3a-3af**

In an inert atmosphere glovebox, to a solution of aryldiazenes **2** (0.15 mmol, 1.0 equiv.), α-aryldiazoketones **1** (0.18 mmol, 1.2 equiv.) in DCE (0.5 mL) were added a solution of $B(C_6F_5)_3$ (5 mol%) in DCE (0.5 mL). Finally, the reaction mixture was stirred at room temperature for 12 h. Afterwards, the reaction mixture was then concentrated under reduced pressure and the residue was purified by flash chromatography on silica gel eluted with petroleum ether/EtOAc to give the desired products aza-β-lactams **3a-3af**.

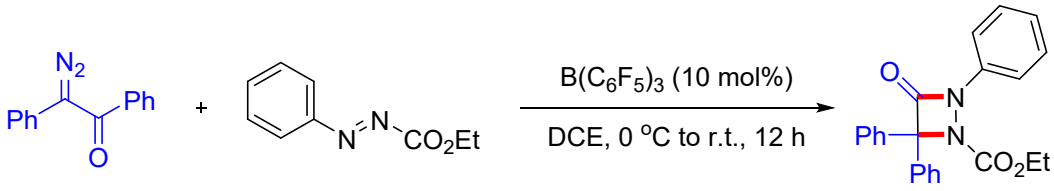
6. General procedure for [2+2] cycloadditions of α-aryldiazoketones with dialkyl azodicarboxylate 2y.



In an inert atmosphere glovebox, to a solution of dialkyl azodicarboxylate **2y** (0.15 mmol, 1.0 equiv.), α-aryldiazoketones **1** (0.18 mmol, 1.2 equiv.) in Et_2O (0.5 mL) were added a solution of $B(C_6F_5)_3$ (5 mol%) in Et_2O (0.5 mL). Afterwards, the reaction mixture was stirred at room temperature for 12 h. Then a white solid precipitated, which was then filtered and washed three times with *n*-hexane to afford the desired products aza-β-lactams **3ag-3al**.

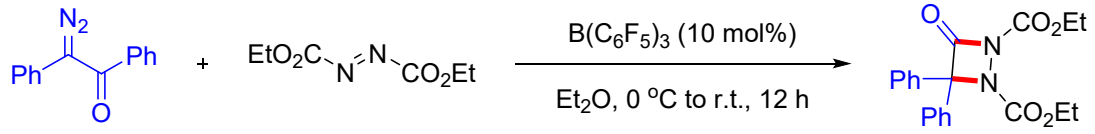
7. Gram-scale version of Wolff rearrangement/[2+2] cascade cyclization and synthetic transformations

7.1 Gram-scale synthesis of 3a.



In an inert atmosphere glovebox, a Schlenk flask (100 mL) was charged with **1a** (1.87 g, 8.4 mmol) and **2a** (1.25 g, 7.0 mmol) and DCE (7 mL) was added. The resulting mixture was cooled to 0 ºC. Finally, a solution of $B(C_6F_5)_3$ (0.35 g, 0.7 mmol) in DCE (7 mL) was added slowly to the mixture under stirring at 0 ºC. Then, the reaction was warmed to room temperature slowly and stirred for 12 hours. Afterwards, the reaction mixture was then concentrated under reduced pressure and the residue was purified by flash chromatography (eluent: petroleum ether/ethyl acetate = 30/1) on silica gel to afford the product **3a** as a white solid (2.16 g, 83% yield).

7.2 Gram-scale synthesis of 3ag.



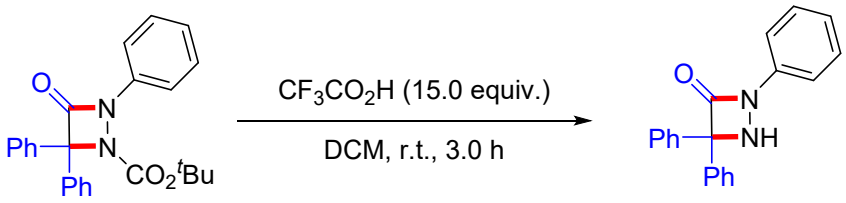
1a
1.87 g, 8.4 mmol

2y
1.22 g, 7 mmol

3ag
2.19 g, 85% yield

In an inert atmosphere glovebox, a Schlenk flask (100 mL) was charged with **1a** (1.87 g, 8.4 mmol) and **2y** (1.22 g, 7.0 mmol) and Et_2O (7 mL) was added. The resulting mixture was cooled to 0 ºC. Finally, a solution of $B(C_6F_5)_3$ (0.35 g, 0.7 mmol) in Et_2O (7 mL) was added slowly to the mixture under stirring at 0 ºC. Then, the reaction was warmed to room temperature slowly and stirred for 12 hours. Afterwards, a white solid precipitated, which was then filtered and washed three times with n-hexane to afford the desired products aza-β-lactams **3ag** as a white solid (2.19 g, 85% yield).

7.3 Transformations of 3x.



3x

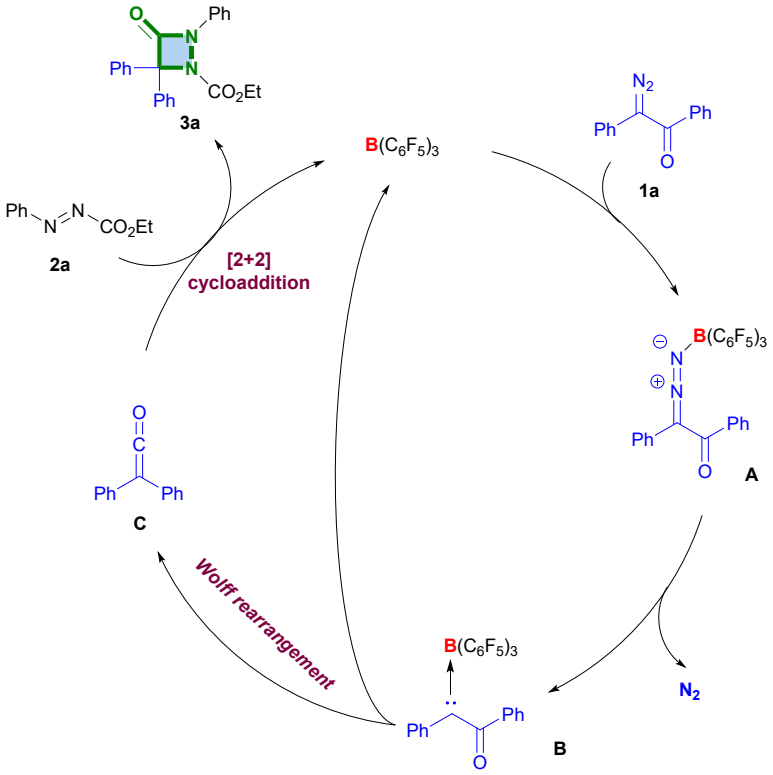
3x-DG, 95% yield

To the vials containing **3x** (0.15 mmol, 60.0 mg, 1.0 equiv) and charged with a stir bar was added DCM (1.0 mL) followed by TFA (0.17 mL, 15.0 equiv) and the reactions was stirred at room temperature for 3 hours, then aqueous $NaHCO_3$ solution was added and extracted with DCM (3 ×1.5 mL) and the phases were separated. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel, petroleum ether/EtOAc =30:1 to 10:1) to give **3x-DG** (42.8 mg, 95%) as a white solid.

8. Plausible reaction mechanism

On the basis of previous work[9,10], a plausible mechanism for the current transformation is proposed. First，the reaction is initiated by the coordination of

$B(C_6F_5)_3$ with **1a** led to the N→B adduct **A**. This Lewis acid activation facilitates the extrusion of N_2, generating intermediate **B**. Subsequently, **B** undergoes a Wolff rearrangement, leading to intermediate **C** with concurrent release of $B(C_6F_5)_3$ for further catalysis. Finally, the electrophilic center in intermediate **C** is attacked by the nucleophilic **2a**, subsequent cyclization delivers the final product **3a**.



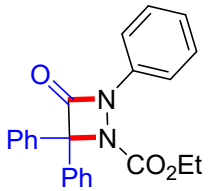
Proposed reaction mechanism.

8. Single crystal X-ray crystallography

X-ray crystallographic data were collected on a Bruker D8 QUEST diffractometer using Cu (60W, Diamond, μKα = 12.894 mm^{-1}) micro-focus X-ray sources at 161 K. The structure was solved and refined using Full-matrix least-squares based on F^2 with program SHELXS and SHELXL[6] within OLEX2. Products **3a** and **3aa** were crystallized by slow vapor diffusion of DCM and hexane layered on top at room temperature. CCDC 2494209 and CCDC 2528549 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre (CCDC).

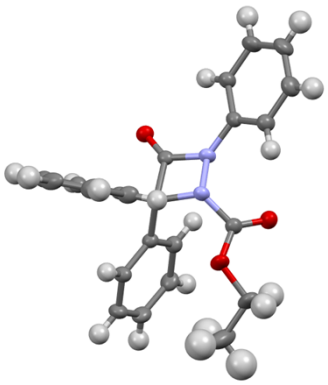
Table S1 Crystal data and structure refinement details for compound **3a**

Identification code	TT2
Empirical formula	$C_{23}H_{20}N_2O_3$
Formula weight	372.41
Temperature/K	150.0
Crystal system	triclinic
Space group	P-1
a/Å	8.3482(6)
b/Å	9.8084(7)
c/Å	13.6240(10)
α/°	98.448(4)
β/°	107.812(4)
γ/°	107.751(4)
Volume/Å^3	975.40(13)
Z	2
ρ_{calc}g/cm^3	1.268
μ/mm^{-1}	0.685
F(000)	392.0
Crystal size/mm^3	0.014 × 0.01 × 0.008
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	7.068 to 136.762
Index ranges	-10 ≤ h ≤ 10, -11 ≤ k ≤ 11, -16 ≤ l ≤ 16
Reflections collected	11454
Independent reflections	3519 [R_{int} = 0.0543, R_{sigma} = 0.0482]
Data/restraints/parameters	3519/0/254
Goodness-of-fit on F^2	1.066
Final R indexes [I>=2σ (I)]	R_1 = 0.0391, wR_2 = 0.0928
Final R indexes [all data]	R_1 = 0.0502, wR_2 = 0.0995
Largest diff. peak/hole / e Å^{-3}	0.22/-0.26



3a

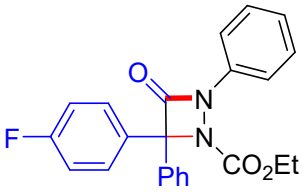
=



CCDC-2494209

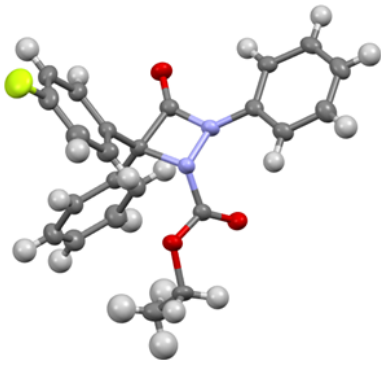
Table S2 Crystal data and structure refinement details for compound **3aa**

Identification code	T1
Empirical formula	$C_{23}H_{19}FN_2O_3$
Formula weight	390.40
Temperature/K	149.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	15.864(5)
b/Å	11.530(3)
c/Å	10.602(3)
α/°	90
β/°	95.905(10)
γ/°	90
Volume/Å^3	1928.9(9)
Z	4
ρ_{calc}g/cm^3	1.344
μ/mm^{-1}	0.795
F(000)	816.0
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	5.6 to 132.984
Index ranges	$-18 \le h \le 18$, $-13 \le k \le 13$, $-12 \le l \le 12$
Reflections collected	16717
Independent reflections	3371 [R_{int} = 0.0729, R_{sigma} = 0.0495]
Data/restraints/parameters	3371/0/263
Goodness-of-fit on F^2	1.062
Final R indexes [I>=2σ (I)]	R_1 = 0.0475, wR_2 = 0.1239
Final R indexes [all data]	R_1 = 0.0619, wR_2 = 0.1355
Largest diff. peak/hole / e Å^{-3}	0.87/-0.30



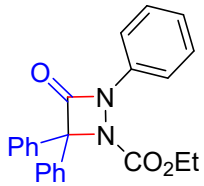
3aa

=



CCDC-2528549

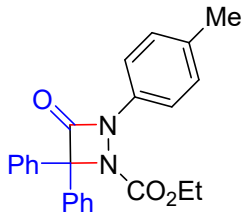
9. Characterization data of products 3a-3al



3a

ethyl 3-oxo-2,4,4-triphenyl-1,2-diazetidine-1-carboxylate (**3a**)

The compound **3a** as obtained as a white solid in 94% yield (53.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57-7.52 (m, 4H), 7.47-7.36 (m, 10H), 7.19-7.13 (m, 1H), 3.84 (q, *J* = 7.2 Hz, 2H), 0.82 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.49, 158.32, 135.02, 129.16, 128.75 (two peaks were incorporated), 128.53 (two peaks were incorporated), 128.52 (two peaks were incorporated), 128.22 (two peaks were incorporated), 124.81, 116.50, 91.66, 62.89, 13.57. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{21}N_2O_3^+$, ($[M+H]^+$): 373.1547, Found: 373.1549.

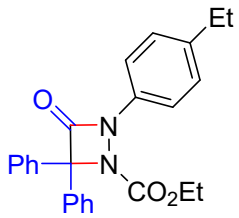


3b

ethyl 3-oxo-4,4-diphenyl-2-(p-tolyl)-1,2-diazetidine-1-carboxylate (**3b**)

The compound **3b** as obtained as a white solid in 95% yield (55.1 mg).

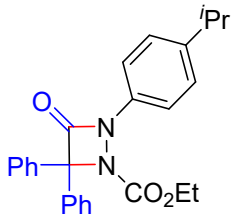
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54-7.49 (m, 4H), 7.40 (dd, *J* = 6.0, 4.0 Hz, 6H), 7.35-7.31 (m, 2H), 7.17 (dd, *J* = 8.4, 0.8 Hz, 2H), 3.82 (q, *J* = 7.2 Hz, 2H), 2.32 (s, 3H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.44, 158.41, 135.70, 135.08, 134.65, 129.11, 128.50, 128.24, 116.79, 116.67, 91.59, 62.84, 20.92, 13.60. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_3^+$, ($[M+H]^+$): 387.1703, Found: 387.1700.



3c

ethyl 2-(4-ethylphenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3c**)

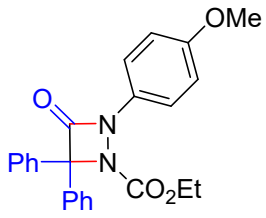
The compound **3c** as obtained as a white solid in 92% yield (55.3 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.49 (m, 4H), 7.43-7.33 (m, 6H), 7.37-7.33 (m, 2H), 7.22-7.16 (m, 2H), 3.82 (q, *J* = 7.2 Hz, 2H), 2.62 (q, *J* = 7.6 Hz, 2H), 1.20 (t, *J* = 7.6 Hz, 3H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.45, 158.43, 141.08, 135.86, 135.09, 129.13, 128.53, 128.25, 128.15, 116.78, 91.60, 62.85, 28.35, 15.67, 13.61. **HRMS (ESI, m/z)**: Calcd. for $C_{25}H_{25}N_2O_3^+$, ($[M+H]^+$): 401.1860, Found: 401.1863.



3d

ethyl 2-(4-isopropylphenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3d**)

The compound **3d** as obtained as a white solid in 93% yield (57.8 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.49 (m, 4H), 7.42-7.38 (m, 6H), 7.36 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 8.4 Hz, 2H), 3.82 (q, *J* = 7.2 Hz, 2H), 2.88 (s, *J* = 7.2 Hz, 1H), 1.22 (d, *J* = 7.2 Hz, 6H), 0.79 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.44, 158.45, 145.66, 135.91, 135.08, 129.13, 128.53, 128.24, 126.74, 116.71, 91.60, 62.86, 33.65, 23.96, 13.60. **HRMS (ESI, m/z)**: Calcd. for $C_{26}H_{27}N_2O_3^+$, ($[M+H]^+$): 415.2016, Found: 415.2018.

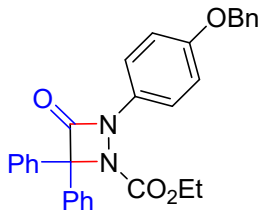


3e

ethyl 2-(4-methoxyphenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3e**)

The compound **3e** as obtained as a white solid in 93% yield (56.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52-7.50 (m, 4H), 7.44-7.35 (m, 8H), 6.89 (d, *J* = 9.2 Hz, 2H), 3.81 (q, *J* = 7.2 Hz, 2H), 3.78 (s, 3H), 0.79 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz,

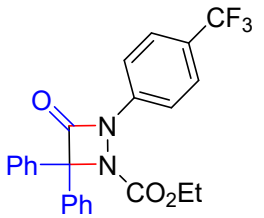
$CDCl_3$) δ 165.64, 157.18, 135.09, 131.51, 129.12, 128.53, 128.52, 128.23, 119.21, 114.09, 91.60, 62.82, 55.50, 13.59. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_4^+$, ($[M+H]^+$): 403.1652, Found: 403.1656.



3f

ethyl 2-(4-(benzyloxy)phenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3f**)

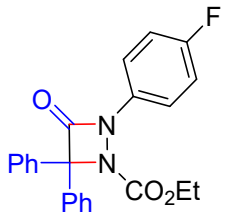
The compound **3f** as obtained as a white solid in 86% yield (61.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54-7.49 (m, 4H), 7.41-7.33 (m, 11H), 6.97 (dd, J = 9.2, 2.0 Hz, 2H), 5.05 (s, 2H), 3.82 (q, J = 7.2 Hz, 2H), 0.80 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.59, 158.55, 156.29, 136.75, 135.09, 131.74, 129.12, 128.56, 128.53, 128.23, 127.96, 127.38, 119.06, 115.13, 91.62, 70.27, 62.82, 13.59. **HRMS (ESI, m/z)**: Calcd. for $C_{30}H_{27}N_2O_4^+$, ($[M+H]^+$): 479.1965, Found: 479.1967.



3g

ethyl 3-oxo-4,4-diphenyl-2-(4-(trifluoromethyl)phenyl)-1,2-diazetidine-1-carboxylate (**3g**)

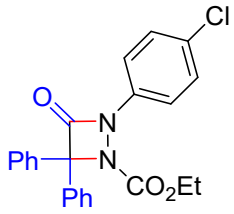
The compound **3g** as obtained as a white solid in 88% yield (58.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.74-7.57 (m, 3H), 7.56-7.46 (m, 6H), 7.45-7.38 (m, 5H), 3.84 (q, J = 7.2 Hz, 2H), 0.81 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.56, 158.17, 140.56 (q, $J_{C\text{-}F}$ = 1.4 Hz), 134.67, 129.41, 128.67, 128.17, 126.77, 126.44, 126.16 (q, $J_{C\text{-}F}$ = 3.8 Hz), 124.68 (q, $J_{C\text{-}F}$ = 273.1 Hz), 116.20, 92.24, 63.24, 13.57. **^{19}F{^{1}H} NMR** (471 MHz, $CDCl_3$), δ: -62.05. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{20}F_3N_2O_3^+$, ($[M+H]^+$): 441.1421, Found: 441.1425.



3h

ethyl 2-(4-fluorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3h**)

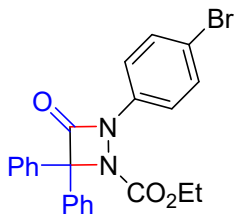
The compound **3h** as obtained as a white solid in 90% yield (52.7 mg). **1H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.48 (m, 4H), 7.44-7.39 (m, 8H), 7.09-7.03 (m, 2H), 3.82 (q, J = 7.2 Hz, 2H), 0.79 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.53, 159.85 (d, $^{1}J_{C\text{-}F}$ = 245.3 Hz), 158.41, 134.89, 134.24 (d, $^{4}J_{C\text{-}F}$ = 2.7 Hz), 129.26, 128.60, 128.19, 118.52 (d, $^{3}J_{C\text{-}F}$ = 8.3 Hz), 115.64 (d, $^{2}J_{C\text{-}F}$ = 23.2 Hz), 91.92, 63.01, 13.57. **^{19}F{^{1}H} NMR** (471 MHz, $CDCl_3$), δ: -116.92. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}FN_2O_3^+$, ($[M+H]^+$): 391.1452, Found: 391.1450.



3i

ethyl 2-(4-chlorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3i**)

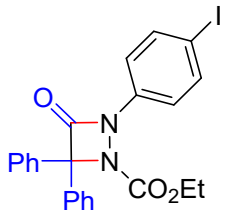
The compound **3i** as obtained as a white solid in 91% yield (55.5 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50-7.47 (m, 4H), 7.43-7.31 (m, 10H), 3.82 (q, J = 7.2 Hz, 2H), 0.80 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.43, 158.28, 136.59, 134.82，130.06, 129.31, 128.86, 128.62 (three peaks were incorporated), 128.19 (three peaks were incorporated), 117.79, 92.03, 63.09, 13.58. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}Cl^{34.9689}N_2O_3^+$, ($[M+H]^+$): 407.1157, Found: 407.1155; $C_{23}H_{20}Cl^{35.4500}N_2O_3^+$, ($[M+H]^+$): 409.1128, Found: 409.1126.



3j

ethyl 2-(4-bromophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3j**)

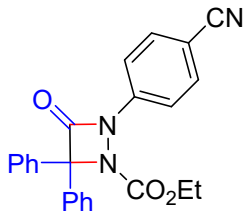
The compound **3j** as obtained as a white solid in 88% yield (59.6 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50-7.46 (m, 6H), 7.43-7.39 (m, 6H), 7.33-7.29 (m, 2H), 3.82 (q, *J* = 7.2 Hz, 2H), 0.79 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.42, 158.25, 137.07, 134.80, 131.78, 129.32, 128.63 (three peaks were incorporated), 128.19 (three peaks were incorporated), 118.07, 117.69, 92.06, 63.10, 13.59. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}Br^{79.9183}N_2O_3^+$, ([M+H]$^+$): 451.0652, Found: 451.0650; $C_{23}H_{20}Br^{80.9163}N_2O_3^+$, ([M+H]$^+$): 453.0632, Found: 453.0634.



3k

ethyl 2-(4-iodophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3k**)

The compound **3k** as obtained as a white solid in 92% yield (68.8 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.4 Hz, 2H), 7.50-7.46 (m, 4H), 7.44-7.38 (m, 6H), 7.19 (d, *J* = 8.4 Hz, 2H), 3.82 (q, *J* = 7.2 Hz, 2H), 0.79 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.39, 158.20, 137.74, 137.66 (two peaks were incorporated), 134.77, 129.31, 128.62 (two peaks were incorporated), 128.61 (two peaks were incorporated), 128.18, 118.29, 92.05, 88.30, 63.09, 13.58. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}IN_2O_3^+$, ([M+H]$^+$): 499.0513, Found: 499.0516.

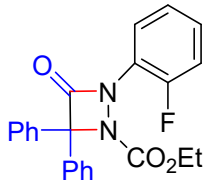


3l

ethyl 2-(4-cyanophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3l**)

The compound **3l** as obtained as a white solid in 77% yield (45.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68-7.61 (m, 2H), 7.53-7.44 (m, 6H), 7.44-7.39 (m, 6H), 3.83 (q, *J* = 7.2 Hz, 2H), 0.79 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.46, 158.04,

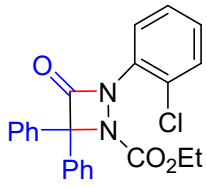
141.11, 134.46, 133.03, 129.52, 128.70, 128.12, 118.59, 116.53, 107.83, 92.48, 63.38, 13.54. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{20}N_3O_3^+$, ($[M+H]^+$): 398.1499, Found: 398.1496.



3m

ethyl 2-(2-fluorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3m**)

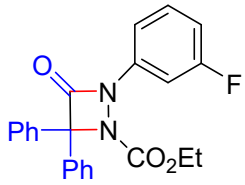
The compound **3m** as obtained as a white solid in 86% yield (47.4 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87-7.26 (m, 5H), 7.20-6.96 (m, 5H), 6.93-6.82 (m, 2H), 6.72 (ddd, J = 11.2, 8.0, 1.6 Hz, 1H), 4.37 (q, J = 7.2 Hz, 2H), 1.36 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (125 Hz, $CDCl_3$) δ 165.18, 154.11 (d, $^1J_{C\text{-}F}$ = 244.0 Hz), 148.56, 135.66, 133.46 (d, $^3J_{C\text{-}F}$ = 10.3 Hz), 129.69, 129.26, 128.66, 128.34, 128.04, 125.60 (d, $^3J_{C\text{-}F}$ = 7.3 Hz), 125.57, 123.66 (d, $^4J_{C\text{-}F}$ = 3.5 Hz), 120.03, 115.46 (d, $^2J_{C\text{-}F}$ = 18.9 Hz), 94.43, 77.25, 77.00, 76.75, 64.00, 14.18. **^{19}F{^{1}H} NMR** (471 MHz, $CDCl_3$), δ: -118.82. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}FN_2O_3^+$, ($[M+H]^+$): 391.1452, Found: 391.1449.



3n

ethyl 2-(2-chlorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3n**)

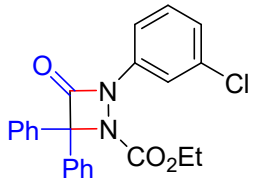
The compound **3n** as obtained as a white solid in 79% yield (48.2 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 8.2, 2.0 Hz, 2H), 7.47-7.38 (m, 3H), 7.21-7.08 (m, 7H), 7.04-6.99 (m, 1H), 6.86 (td, *J* = 7.6, 2.0 Hz, 1H), 4.36 (qd, *J* = 7.2, 2.8 Hz, 2H), 1.34 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.21, 148.28, 142.53, 135.77, 132.75, 129.97, 129.91, 129.35, 128.68, 128.41, 128.38, 128.01, 127.95, 126.64, 126.19, 121.35, 95.13, 63.98, 14.20. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}ClN_2O_3^+$, ($[M+H]^+$): 407.1157, Found: 407.1159.



3o

ethyl 2-(3-fluorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3o**)

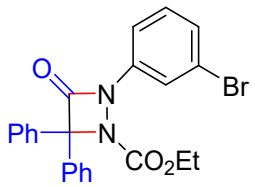
The compound **3o** as obtained as a white solid in 91% yield (53.3 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52-7.47 (m, 4H), 7.42-7.39 (m, 6H), 7.32 (td, *J* = 8.4, 6.0 Hz, 1H), 7.23 (ddd, *J* = 8.4, 2.0, 1.2 Hz, 1H), 7.14 (dt, *J* = 10, 2.4 Hz, 1H), 6.85 (tdd, *J* = 8.4, 2.4, 1.2 Hz, 1H), 3.83 (q, *J* = 7.2 Hz, 2H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.55, 162.74 (d, $^{1}J_{C\text{-}F}$ = 247.5 Hz), 158.24, 139.30 (d, $^{3}J_{C\text{-}F}$ = 10.6 Hz), 134.78, 130.17 (d, $^{3}J_{C\text{-}F}$ = 9.2 Hz), 129.32, 128.62, 128.19, 112.09 (d, $^{4}J_{C\text{-}F}$ = 3.0 Hz), 111.64 (d, $^{2}J_{C\text{-}F}$ = 21.3 Hz), 104.15 (d, $^{2}J_{C\text{-}F}$ = 27.1 Hz), 92.01, 63.12, 13.57. (d, $^{4}J_{C\text{-}F}$ = 2.7 Hz). **^{19}F{^{1}H} NMR** (471 MHz, $CDCl_3$), δ: -111.13. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}FN_2O_3^+$, ($[M+H]^+$): 391.1452, Found: 391.1450.



3p

ethyl 2-(3-chlorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3p**)

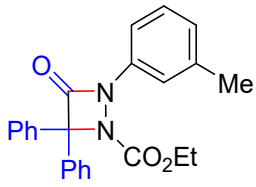
The compound **3p** as obtained as a white solid in 92% yield (56.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52-7.47 (m, 4H), 7.43-7.39 (m, 7H), 7.34 (ddd, J = 8.2, 2.0, 1.2 Hz, 1H), 7.28 (t, *J* = 8.0 Hz, 1H), 7.12 (ddd, *J* = 7.6, 2.0, 1.2 Hz, 1H), 3.83 (q, *J* = 7.2 Hz, 2H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.56, 158.21, 138.97, 134.77, 134.64, 129.88, 129.34, 128.64, 128.20, 124.91, 116.52, 114.66, 92.03, 63.14, 13.59. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}ClN_2O_3^+$, ($[M+H]^+$): 407.1157, Found: 407.1160.



3q

ethyl 2-(3-bromophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3q**)

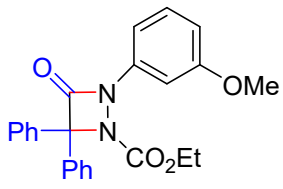
The compound **3q** as obtained as a white solid in 90% yield (61.0 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 (t, *J* = 2.0 Hz, 1H), 7.51-7.45 (m, 4H), 7.43-7.36 (m, 7H), 7.29-7.26 (m, 1H), 7.22 (t, *J* = 8.0 Hz, 1H), 3.83 (q, *J* = 7.2 Hz, 2H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.55, 158.19, 139.05, 134.76, 130.13, 129.33, 128.64, 128.19, 127.83, 122.52, 119.29, 115.12, 92.03, 63.15, 13.58. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}BrN_2O_3^+$, ($[M+H]^+$): 451.0652, Found: 451.0654.



3r

ethyl 3-oxo-4,4-diphenyl-2-(m-tolyl)-1,2-diazetidine-1-carboxylate (**3r**)

The compound **3r** as obtained as a white solid in 93% yield (53.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54-7.49 (m, 4H), 7.43-7.39 (m, 6H), 7.27-7.19 (m, 3H), 6.96 (d, *J* = 7.2 Hz, 1H), 3.82 (q, *J* = 13.2, 7.2 Hz, 2H), 2.37 (s, 3H), 0.80 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.52, 158.39, 138.84, 137.98, 135.07, 129.16, 128.62, 128.55, 128.26, 125.72, 117.11, 113.68, 91.58, 62.89, 21.50, 13.60. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_3^+$, ($[M+H]^+$): 387.1703, Found: 387.1700.

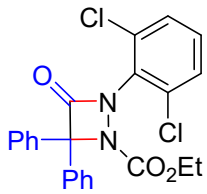


3s

ethyl 2-(3-methoxyphenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3s**)

The compound **3s** as obtained as a white solid in 94% yield (56.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.48 (m, 4H), 7.42-7.38 (m, 6H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.02 (t, *J* = 2.4 Hz, 1H), 6.99 (ddd, J = 8.0, 2.0, 1.2 Hz, 1H), 6.69 (ddd, J = 8.4, 2.4, 0.8 Hz,

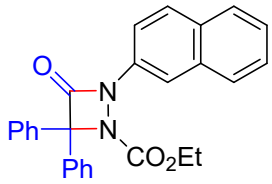
1H), 3.82 (q, J = 7.2 Hz, 2H), 3.81 (s, 3H), 0.80 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.59, 159.92, 158.33, 139.11, 134.98, 129.65, 129.20, 128.56, 128.24, 110.64, 108.83, 102.57, 91.69, 62.94, 55.38, 13.60. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_4^+$, ($[M+H]^+$): 403.1652, Found: 403.1654.



3t

ethyl 2-(2,6-dichlorophenyl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3t**)

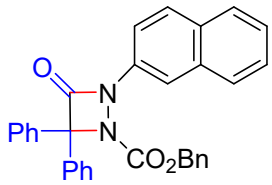
The compound **3t** as obtained as a white solid in 35% yield (43.8 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80-7.03 (m, 12H), 6.87 (t, J = 7.6 Hz, 1H), 4.34 (q, J = 7.2 Hz, 2H), 1.31 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 163.29, 147.57, 137.15, 128.96, 128.94, 128.26, 128.26, 128.20, 127.47, 94.03, 63.75, 14.17. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{19}Cl_2N_2O_3^+$, ($[M+H]^+$): 441.0767, Found: 441.0764.



3u

ethyl 2-(naphthalen-2-yl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3u**)

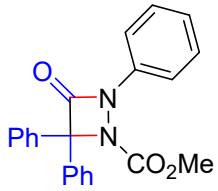
The compound **3u** as obtained as a white solid in 91% yield (57.7 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.90-7.74 (m, 4H), 7.70 (dd, J = 8.5, 2.0 Hz, 1H), 7.60-7.51 (m, 4H), 7.50-7.37 (m, 8H), 3.85 (q, J = 7.0 Hz, 2H), 0.82 (t, J = 7.0 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.69, 158.43, 135.64, 135.02, 133.22, 130.93, 129.22, 128.83, 128.59, 128.28, 127.68, 126.70, 125.29, 116.54, 113.39, 91.84, 63.01, 13.63. **HRMS (ESI, m/z)**: Calcd. for $C_{27}H_{23}N_2O_4^+$, ($[M+H]^+$): 423.1703, Found: 423.1707.



3v

benzyl 2-(naphthalen-2-yl)-3-oxo-4,4-diphenyl-1,2-diazetidine-1-carboxylate (**3v**)

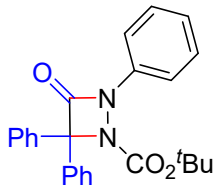
The compound **3v** was obtained as a white solid in 80% yield (58.1 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.89-7.57 (m, 5H), 7.50-7.11 (m, 15H), 6.92 (d, *J* = 7.5 Hz, 2H), 4.71 (s, 2H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ173.33, 165.65, 135.50, 134.74, 133.16, 130.93, 129.42, 129.20, 128.82, 128.58, 128.38, 128.35, 128.13, 127.67, 127.64, 126.68, 125.30, 116.58, 113.48, 91.99, 51.54. **HRMS (ESI, m/z)**: Calcd. for $C_{32}H_{25}N_2O_4^+$, ($[M+H]^+$): 485.1860, Found: 485.1864.



3w

methyl 3-oxo-2,4,4-triphenyl-1,2-diazetidine-1-carboxylate (**3w**)

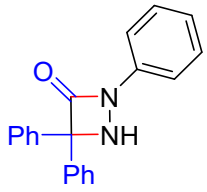
The compound **3w** was obtained as a white solid in 91% yield (48.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.48 (m, 4H), 7.44-7.35 (m, 10H), 7.19-7.12 (m, 1H), 3.33 (s, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.33, 158.96, 137.97, 134.86, 129.22, 128.83, 128.58, 128.13, 124.90, 116.45, 91.96, 53.26. **HRMS (ESI, m/z)**: Calcd. for $C_{22}H_{19}N_2O_3^+$, ($[M+H]^+$): 359.1390, Found: 359.1394.



3x

tert-butyl 3-oxo-2,4,4-triphenyl-1,2-diazetidine-1-carboxylate (**3x**)

The compound **3x** was obtained as a white solid in 79% yield (47.5 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54-7.49 (m, 4H), 7.44-7.34 (m, 10H), 7.13 (tt, *J* = 7.2, 1.2 Hz, 1H), 1.08 (s, 9H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.69, 156.86, 138.10, 135.42, 129.07, 128.74, 128.53, 128.43, 124.73, 116.54, 91.07, 83.17, 27.31. **HRMS (ESI, m/z)**: Calcd. for $C_{25}H_{25}N_2O_3^+$, ($[M+H]^+$): 401.1860, Found: 401.1858.

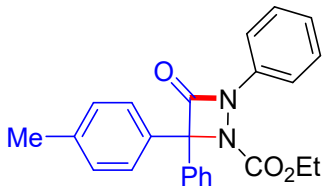


3x-DG

2,4,4-triphenyl-1,2-diazetidin-3-one (**3x-DG**)

The compound **3x-DG** was obtained as a white solid in 77% yield (42.8 mg).

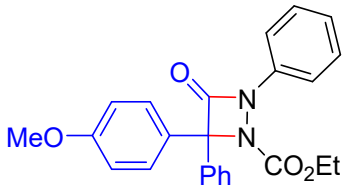
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 8.6 Hz, 2H), 7.54 -7.31 (m, 12H), 7.15-7.08 (m, 1H), 4.96 (s, 1H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 164.40, 137.97 (two peaks were incorporated), 129.08, 124.28, 115.34 (three peaks were incorporated), 82.90. **HRMS (ESI, m/z)**: Calcd. for $C_{20}H_{17}N_2O^+$, ($[M+H]^+$): 301.1336, Found: 301.1339.



3y

ethyl 3-oxo-2,4-diphenyl-4-(p-tolyl)-1,2-diazetidine-1-carboxylate (**3y**)

The compound **3y** as obtained as a white solid in 95% yield (55.1 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56-7.50 (m, 2H), 7.43-7.33 (m, 9H), 7.23-7.18 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.14 (tt, *J* = 7.2, 1.2 Hz, 1H), 3.83 (q, *J* = 7.2 Hz, 2H), 2.37 (s, 3H), 0.81 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.71, 158.43, 139.24, 138.12, 132.09, 129.22, 129.06, 128.76, 128.51, 128.24, 128.17, 124.78, 116.51, 91.72, 77.32, 76.68, 62.88, 21.19, 13.62. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_3^+$, ($[M+H]^+$): 387.1703, Found: 387.1707.

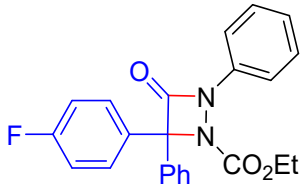


3z

ethyl 4-(4-methoxyphenyl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3z**)

The compound **3z** as obtained as a white solid in 91% yield (54.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.0 Hz, 2H), 7.35-7.20 (m, 9H), 7.02 (t, *J* = 7.5 Hz, 1H),

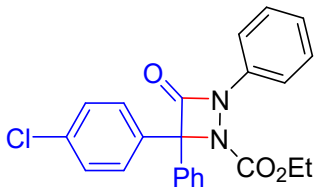
6.79 (d, J = 8.5 Hz, 2H), 3.95-3.71 (q, J = 8.0 Hz, 2H), 3.69 (s, 3H), 0.73 (t, J = 7.0 Hz, 3H). **^{13}C{^{1}H} NMR** (125 Hz, $CDCl_3$) δ 165.72, 160.24, 158.42, 138.06, 135.27, 129.80, 128.94, 128.72, 128.46, 127.96, 127.06, 124.74, 116.45, 113.86, 91.60, 62.86, 55.29, 13.65. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_4^+$, ($[M+H]^+$): 403.1652, Found: 403.1650.



3aa

ethyl 4-(4-fluorophenyl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3aa**)

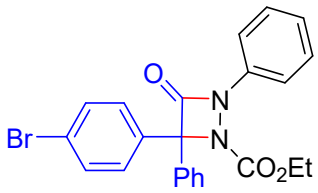
The compound **3aa** as obtained as a white solid in 90% yield (52.7 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54-7.47 (m, 4H), 7.45-7.35 (m, 7H), 7.18-7.14 (tt, J = 8.4 Hz, 1.2 HZ, 1H), 7.14-7.07 (m, 2H), 3.86 (q, J = 7.2 Hz, 2H), 0.85 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.28, 163.1 (d, $^{1}J_{C\text{-}F}$ = 248.3 Hz), 158.19, 137.94, 134.87, 130.94 (d, $^{4}J_{C\text{-}F}$ = 3.3 Hz), 130.27, 130.18, 129.34, 128.73 (d, $^{3}J_{C\text{-}F}$ = 15.0 Hz), 128.13, 124.96, 116.53, 115.59 (d, $^{2}J_{C\text{-}F}$ = 21.7 Hz), 91.02, 63.04, 13.65. **^{19}F{^{1}H} NMR** (471 MHz, $CDCl_3$), δ: -111.80. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}FN_2O_3^+$, ($[M+H]^+$): 391.1452, Found: 391.1455.



3ab

ethyl 4-(4-chlorophenyl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3ab**)

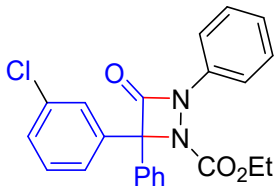
The compound **3ab** as obtained as a white solid in 89% yield (54.2 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.59-7.33 (m, 13H), 7.19-7.13 (m, 1H), 4.00-3.73 (m, 2H), 0.83 (t, J = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 165.06, 158.11, 137.91, 135.28, 134.62, 133.55, 129.57, 129.46, 128.81, 128.78, 128.69, 128.22, 125.00, 116.54, 90.96, 63.08, 13.64. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}Cl^{34.9689}N_2O_3^+$, ($[M+H]^+$): 407.1157, Found: 407.1153; $C_{23}H_{20}Cl^{35.4500}N_2O_3^+$, ($[M+H]^+$): 409.1128, Found: 409.1129



3ac

ethyl 4-(4-bromophenyl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3ac**)

The compound **3ac** as obtained as a white solid in 98% yield (66.3 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 (d, J = 8.8 Hz, 2H), 7.49-7.35 (m, 11H), 7.20-7.12 (m, 1H), 3.87 (q, *J* = 7.2 Hz, 2H), 0.84 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 164.96, 158.08, 137.88, 134.53, 134.05, 131.73, 129.80, 129.47, 128.80, 128.69, 128.23, 124.99, 123.48, 116.51, 90.99, 63.08, 13.62. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}BrN_2O_3^+$, ($[M+H]^+$): 451.0652, Found: 451.0656.

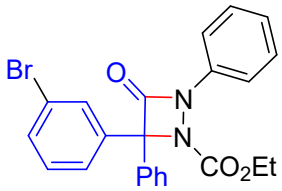


3ad

ethyl 4-(3-chlorophenyl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3ad**)

The compound **3ad** as obtained as a white solid in 89% yield (54.2 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 (q, *J* = 2.0 Hz, 1H), 7.49-7.34 (m, 12H), 7.17 (t, *J* = 7.2 Hz, 1H), 3.87 (dd, J = 11.2, 6.8 Hz, 2H), 0.84 (t, *J* = 7.2 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 164.84, 158.05, 137.87, 136.96, 134.63, 134.44, 129.86, 129.57, 129.33, 128.82, 128.74, 128.38, 128.31, 126.16, 125.02, 116.54, 90.85, 63.12, 13.62.

HRMS (ESI, m/z): Calcd. for $C_{23}H_{20}Cl^{34.9689}N_2O_3^+$, ($[M+H]^+$): 407.1157, Found: 407.1153; $C_{23}H_{20}Cl^{35.4500}N_2O_3^+$, ($[M+H]^+$): 409.1128, Found: 409.1126.

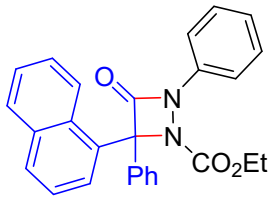


3ae

ethyl 4-(3-bromophenyl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3ae**)

The compound **3ae** as obtained as a white solid in 84% yield (56.8 mg). **^{1}H NMR** (400

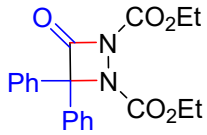
MHz, $CDCl_3$) δ 7.71 (t, *J* = 2.0 Hz, 1H), 7.57-7.50 (m, 2H), 7.48-7.35 (m, 9H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.21- 7.12 (m, 1H), 3.87 (q, *J* = 7.2 Hz, 2H), 0.84 (t, *J* = 7.2 Hz, 3H). **$^{13}C\{^1H\}$ NMR** (100 Hz, $CDCl_3$) δ 164.80, 158.03, 137.84, 137.17, 134.41, 132.27, 131.17, 130.08, 129.56, 128.80, 128.73, 128.29, 126.63, 125.01, 122.69, 116.53, 90.76, 63.12, 13.62. **HRMS (ESI, m/z)**: Calcd. for $C_{23}H_{20}BrN_2O_3^+$, ($[M+H]^+$): 451.0652, Found: 451.0654.



3af

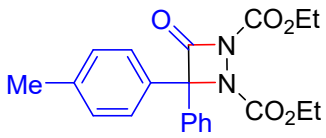
ethyl 4-(naphthalen-1-yl)-3-oxo-2,4-diphenyl-1,2-diazetidine-1-carboxylate (**3af**)

The compound **3af** as obtained as a white solid in 88% yield (55.7 mg). **^1H NMR** (400 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.94 -7.82 (m, 3H), 7.59 (dd, *J* = 8.4, 7.2 Hz, 1H), 7.49-7.27 (m, 11H), 7.17-7.11 (m, 1H), 3.88 (dq, *J* = 10.8, 7.2 Hz, 1H), 3.67 (dd, J = 10.8, 7.2 Hz, 1H), 0.78 (t, *J* = 7.2 Hz, 3H). **$^{13}C\{^1H\}$ NMR** (100 Hz, $CDCl_3$) δ 164.81, 158.49, 138.16, 135.07, 134.18, 130.69, 130.17, 129.86, 129.82, 128.81,128.79, 128.78, 128.20, 127.68, 127.39, 125.82, 125.68, 125.08, 124.87, 116.48, 94.24, 62.97, 13.56. **HRMS (ESI, m/z)**: Calcd. for $C_{27}H_{23}N_2O_3^+$, ($[M+H]^+$): 423.1704, Found: 423.1707.



3ag diethyl 4-oxo-3,3-diphenyl-1,2-diazetidine-1,2-dicarboxylate (**3ag)**

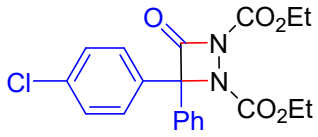
The compound **3ag** was obtained as a white solid in 94% yield. **^1H NMR** (500 MHz, $CDCl_3$) δ 7.65-7.46 (m, 4H), 7.31-7.26 (m, 6H), 4.27-3.98 (m, 2H), 3.82-3.50 (m, 2H), 1.15 (t, *J* = 7.5 Hz, 3H), 0.93 (t, *J* = 7.5 Hz, 3H). **$^{13}C\{^1H\}$ NMR** (100 Hz, $CDCl_3$) δ 167.57, 150.51, 139.02, 137.98, 128.60, 128.45, 128.42, 127.97, 126.73, 126.34, 86.38, 63.02, 61.81, 14.15, 14.04. **HRMS (ESI, m/z)**: Calcd. for $C_{20}H_{21}N_2O_5^+$, ($[M+H]^+$): 369.1445, Found: 369.1442.



3ah

diethyl 4-oxo-3-phenyl-3-(p-tolyl)-1,2-diazetidine-1,2-dicarboxylate (**3ah)**

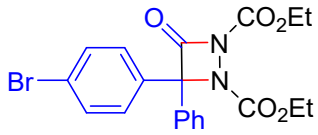
The compound **3ah** was obtained as a white solid in 91% yield (52.2 mg, 1:1 d.r.). **$^1H_{mixture}$ NMR** (400 MHz, $CDCl_3$) δ 7.65 (m, 1H), 7.55-7.42 (m, 2H), 7.35-7.27 (m, 4H), 7.18-7.02 (m, 2H), 4.20-4.02(m, 2H), 3.79-3.68 (m, 1H), 3.68-3.53 (m, 1H), 2.31 (s, 1,5 H), 2.30 (s, 1.5 H), 1.15 (m, 3H), 0.98 (q, *J* = 6.8Hz, 1H), 0.90 (q, *J* = 7.2 Hz, 1H). **$^{13}C\{^1H\}_{mixture}$ NMR** (100 Hz, $CDCl_3$) δ 167.73, 167.66, 150.55, 138.50, 138.49, 138.23, 138.07, 136.26, 135.15, 129.12, 129.11, 128.61, 128.49, 128.48, 128.37, 128.31, 127.91, 126.82, 126.78, 126.70, 126.67, 126.43, 126.38, 126.32, 126.27, 108.76, 108.71, 86.44, 86.42, 62.96, 61.76, 61.73, 21.10, 21.05, 14.17, 14.12, 14.09, 14.01. **HRMS (ESI, m/z)**: Calcd. for $C_{21}H_{23}N_2O_5^+$, ($[M+H]^+$): 383.1601, Found: 383.1603.



3ai

diethyl 3-(4-chlorophenyl)-4-oxo-3-phenyl-1,2-diazetidine-1,2-dicarboxylate (**3ai**)

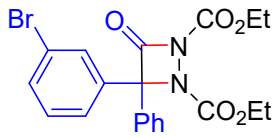
The compound **3ai** was obtained as a white solid in 87% yield (54.4 mg, 1:1 d.r.). **$^1H_{mixture}$ NMR** (400 MHz, $CDCl_3$) δ 7.67-7.56 (m, 2H), 7.45-7.41 (m, 2H), 7.36-7.31 (m, 2H), 7.31-7.26 (m, 3H), 4.22-4.03 (m, 2H), 3.75-3.63 (m, 1H), 3.54 (m, 1H), 1.21-1.13 (m, 3H), 1.00-0.90 (m, 3H). **$^{13}C\{^1H\}_{mixture}$ NMR** (100 Hz, $CDCl_3$) δ 167.22, 150.42, 150.34, 138.60, 137.57, 137.47, 134.78, 134.62, 128.89, 128.73, 128.68, 128.60, 128.17, 128.12, 128.09, 127.59, 127.56, 126.55, 126.21, 126.19, 108.71, 85.81, 85.76, 63.19, 63.12, 61.89, 14.09, 14.01. **HRMS (ESI, m/z)**: Calcd. for $C_{20}H_{20}Cl^{34.9689}N_2O_5^+$, ($[M+H]^+$): 403.1056, Found: 403.1052; $C_{20}H_{20}Cl^{35.4500}N_2O_5^+$, ($[M+H]^+$): 405.1026, Found: 405.1025.



3aj

diethyl 3-(4-bromophenyl)-4-oxo-3-phenyl-1,2-diazetidine-1,2-dicarboxylate (**3aj**)

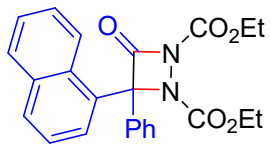
The compound **3al** was obtained as a white solid in 98% yield (61.1 mg, 1:1 d.r.). **^{1}H $_{mixture}$ NMR** (400 MHz, $CDCl_3$) δ 7.74-7.56 (m, 2H), 7.48-7.41 (m, 2H), 7.32 (m, 3H), 7.01 (m, 2H), 4.25-4.00 (m, 2H), 3.70 (m, 1H), 3.62-3.49 (m, 1H), 1.20-1.12 (m, 3H), 1.00-0.89 (m, 3H). **^{13}C{^{1}H}$_{mixture}$ NMR** (100 Hz, $CDCl_3$) δ 167.42, 163.94, 161.47, 150.45, 150.41, 138.83, 137.72, 128.80, 128.72, 128.62, 128.56, 128.37, 128.34, 128.29, 128.26, 128.08, 126.63, 126.24, 126.22, 115.60, 115.59, 115.39, 115.37, 115.02, 114.81, 108.75, 108.72, 85.96, 85.90, 63.14, 63.11, 61.90, 61.81, 14.17, 14.14, 14.02, 14.00. **HRMS (ESI, m/z)**: Calcd. for $C_{20}H_{20}Br^{79.9183}N_2O_5^+$, ($[M+H]^+$): 447.0551, Found: 447.0553; $C_{20}H_{20}Br^{80.9163}N_2O_5^+$, ($[M+H]^+$): 449.0530, Found: 449.0532.



3ak

diethyl 3-(3-bromophenyl)-4-oxo-3-phenyl-1,2-diazetidine-1,2-dicarboxylate (**3ak**)

The compound **3ak** was obtained as a white solid in 81% yield (54.3 mg). **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.61 (d, J = 8.0 Hz, 1H), 7.45-7.26 (m, 6H), 7.17 (t, J = 8.0 Hz, 1H), 4.23 (ddt, J = 22.5, 17.5, 10 Hz, 2H), 3.68 (p, J = 7.0 Hz, 2H), 3.55 (p, J = 7.0 Hz, 1H), 1.23 (t, J = 7.0 Hz, 3H), 0.90 (t, J = 7.0 Hz, 3H). **^{13}C{^{1}H} NMR** (100 Hz, $CDCl_3$) δ 167.07, 150.46, 140.06, 138.47, 131.60, 129.60, 129.29, 128.95, 128.66, 126.27, 125.43, 122.28, 108.88, 85.56, 63.38, 62.00, 14.21, 14.11. **HRMS (ESI, m/z)**: Calcd. for $C_{20}H_{20}Br^{79.9183}N_2O_5^+$, ($[M+H]^+$): 447.0551, Found: 447.0550; $C_{20}H_{20}Br^{80.9163}N_2O_5^+$, ($[M+H]^+$): 449.0530, Found: 449.0529.



3al

diethyl 3-(naphthalen-1-yl)-4-oxo-3-phenyl-1,2-diazetidine-1,2-dicarboxylate (**3al**)

The compound **3an** was obtained as a white solid in 90% yield (56.4 mg, 5.8:1 d.r.). **^{1}H $_{mixture}$ NMR** (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.8 Hz, 2H), 7.08 (t, *J* = 15.6 Hz, 1H), 7.45-7.35 (m, 5H), 7.32-7.29 (m, 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 4.37 (q, *J* = 7.2 Hz, 4H), 1.36 (t, *J* = 7.2 Hz, 6H). **^{13}C{^{1}H}$_{mixture}$ NMR** (100 Hz, $CDCl_3$) δ 163.21, 157.71, 148.22, 134.82, 134.29, 134.18, 130.51, 130.19, 130.06, 129.78, 129.09, 128.86, 128.49, 128.34, 128.22, 128.03, 128.00, 127.81, 127.27, 127.02, 126.59, 125.87, 125.76, 125.21, 112.91, 94.23, 89.25, 64.27, 63.14, 14.25, 14.16, 13.51. **HRMS (ESI, m/z)**: Calcd. for $C_{24}H_{23}N_2O_5^+$, ($[M+H]^+$): 419.1602, Found: 419.1605.

10. References

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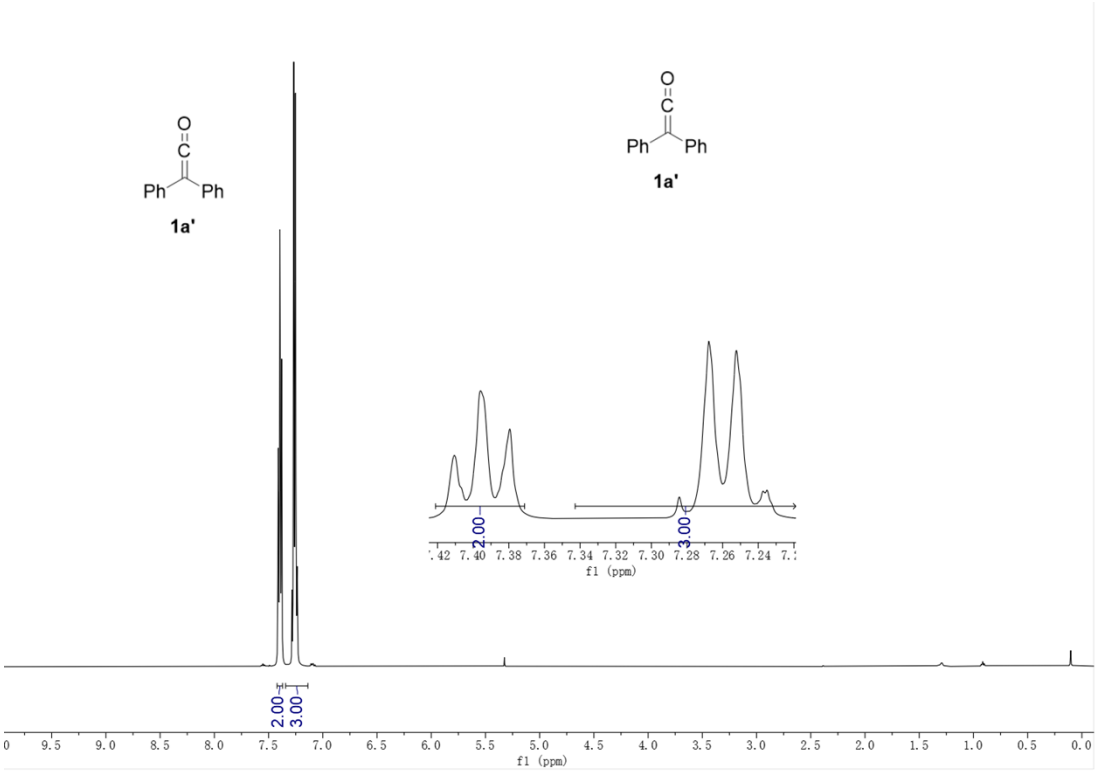
11. NMR spectra of isolated compounds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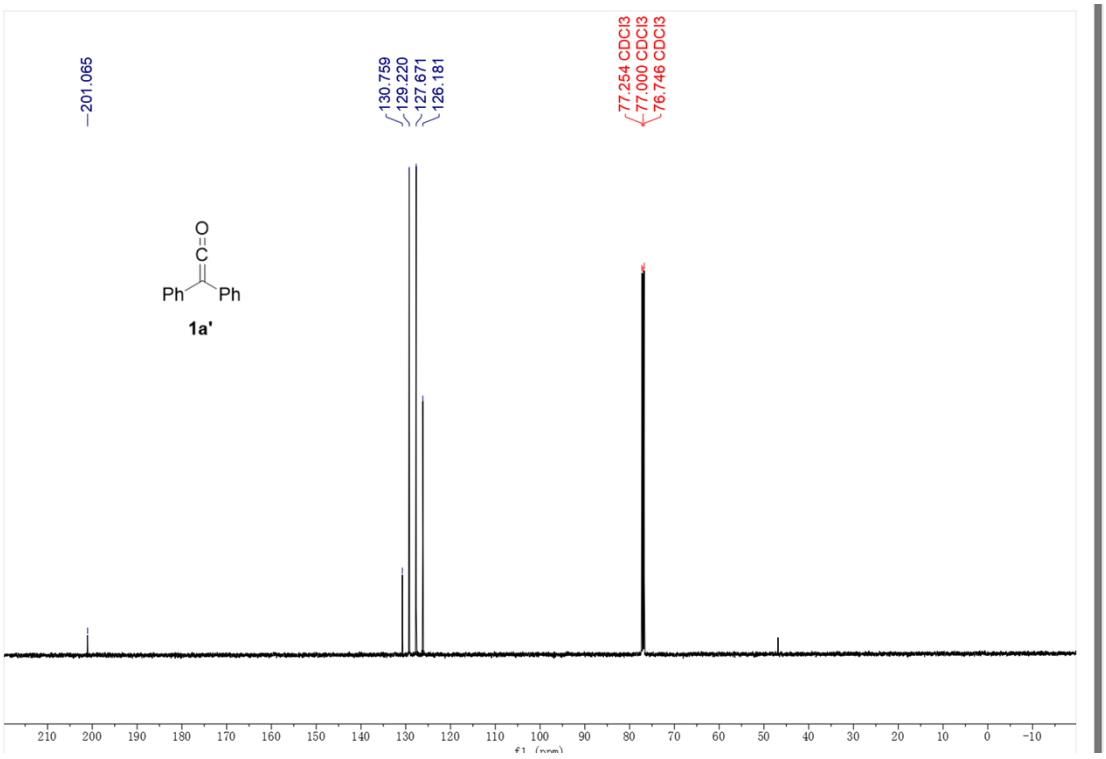
f1 (ppm)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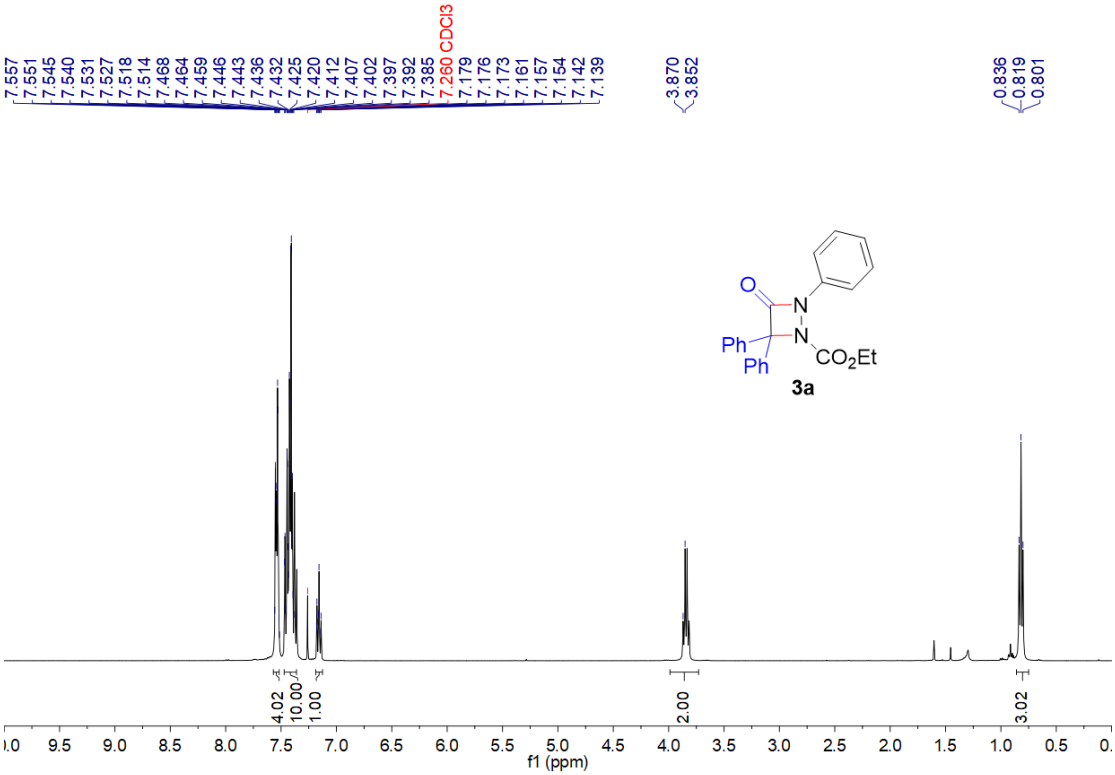
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7.436
7.432
7.425
7.420
7.412
7.407
7.402
7.397
7.392
7.385
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7.179
7.176
7.173
7.161
7.157
7.154
7.142
7.139
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f1 (ppm)
Ph
Ph
CO2Et
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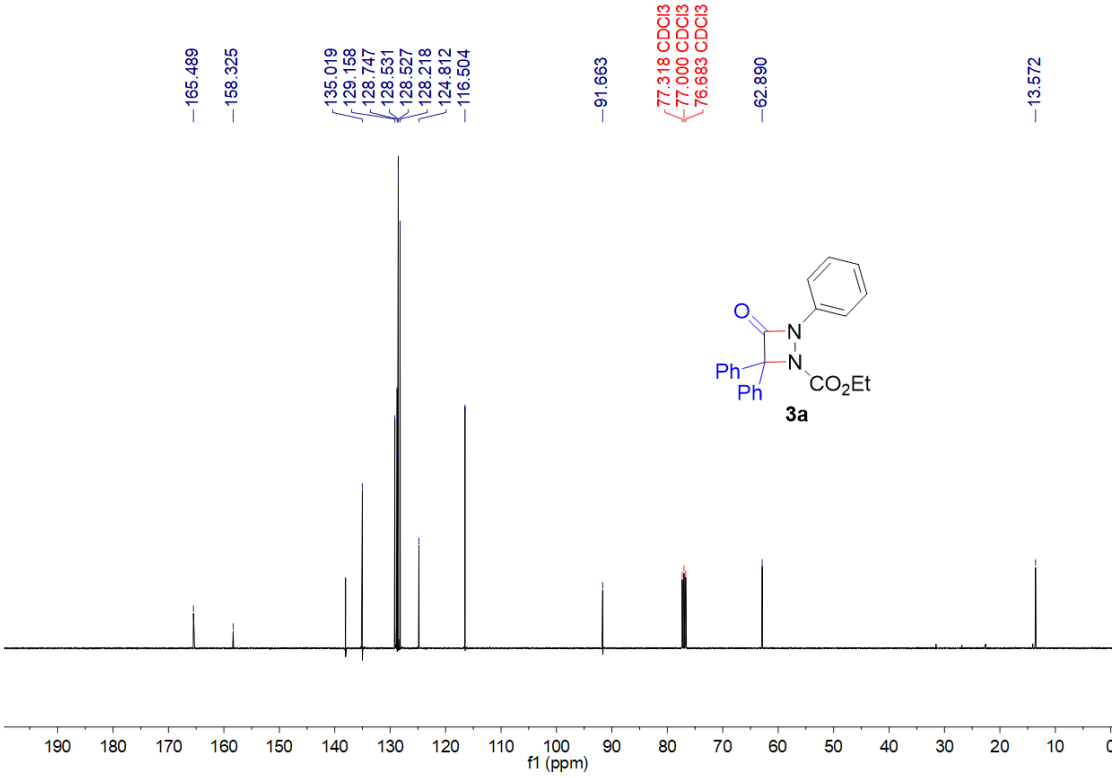
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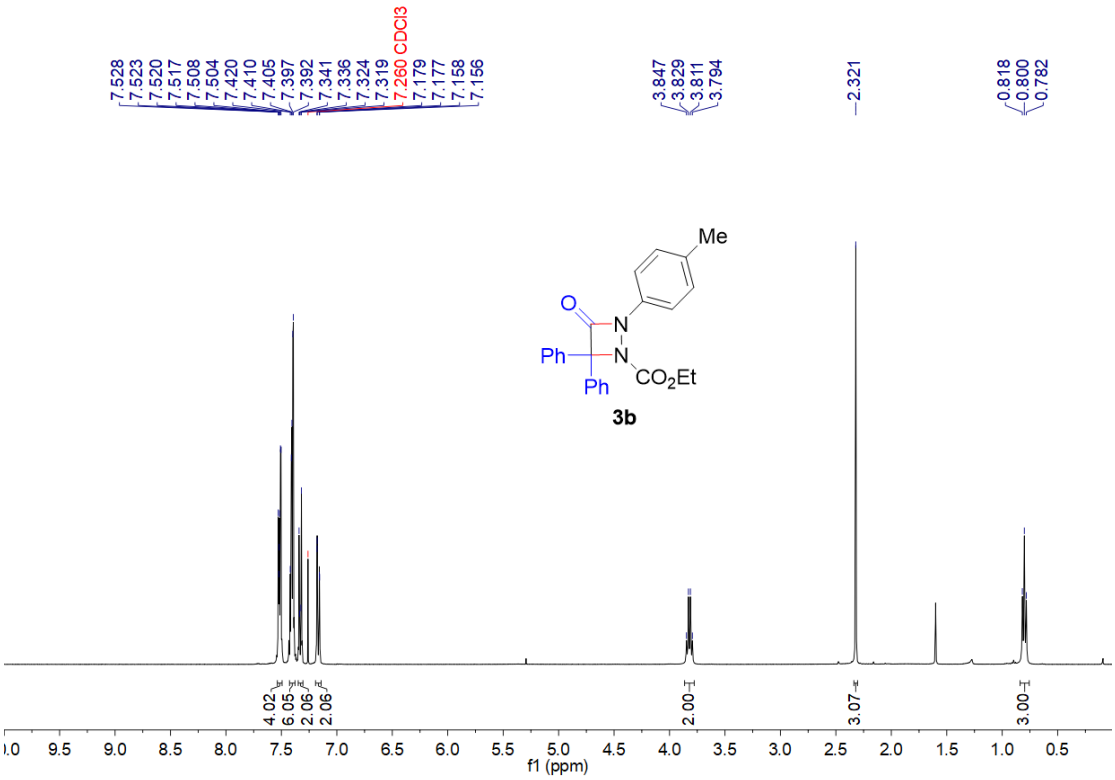
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7.158
7.156
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3.794
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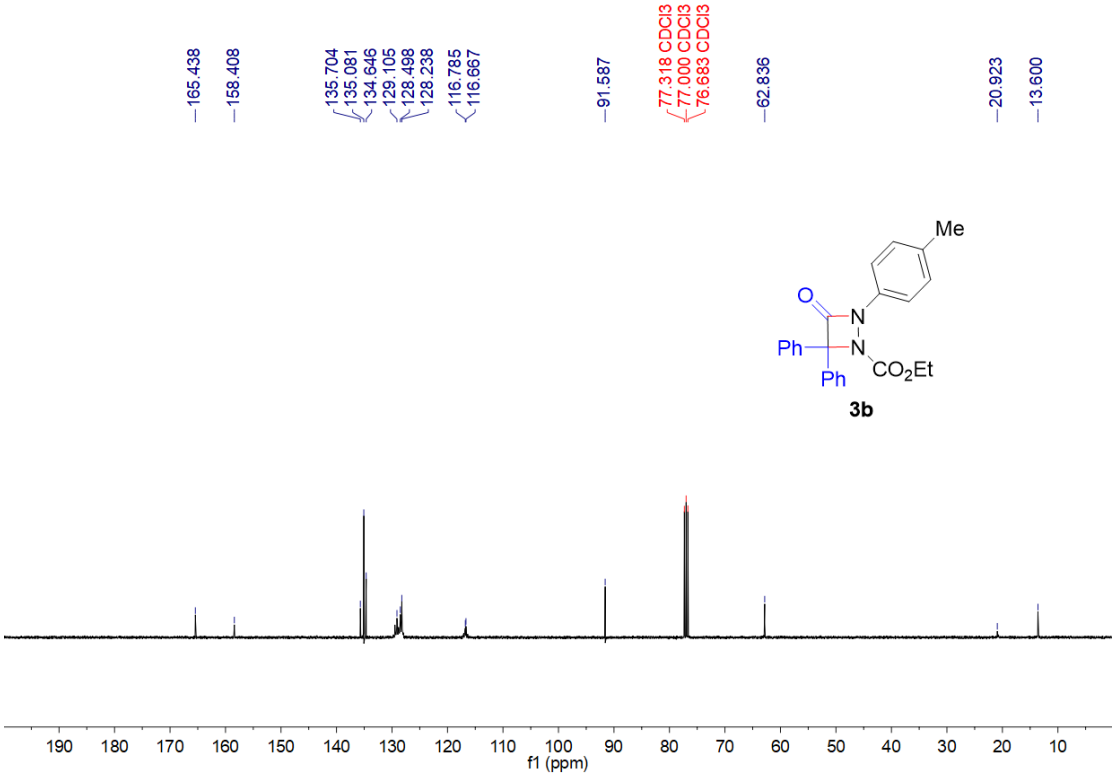
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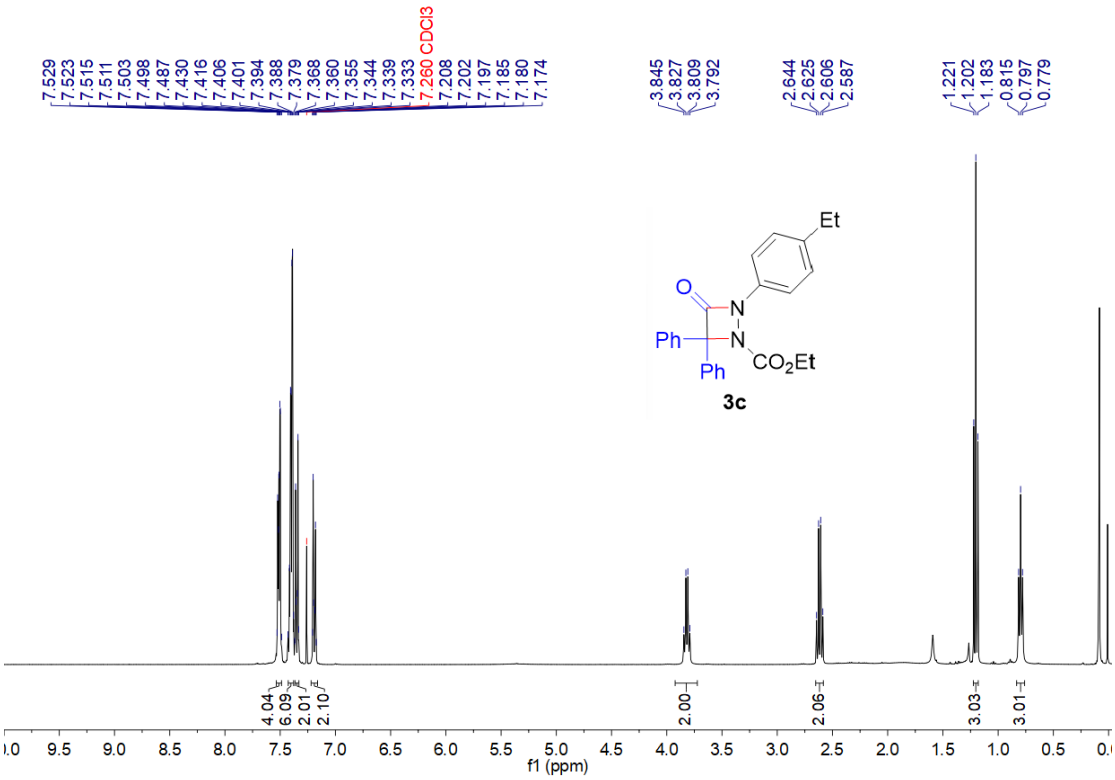
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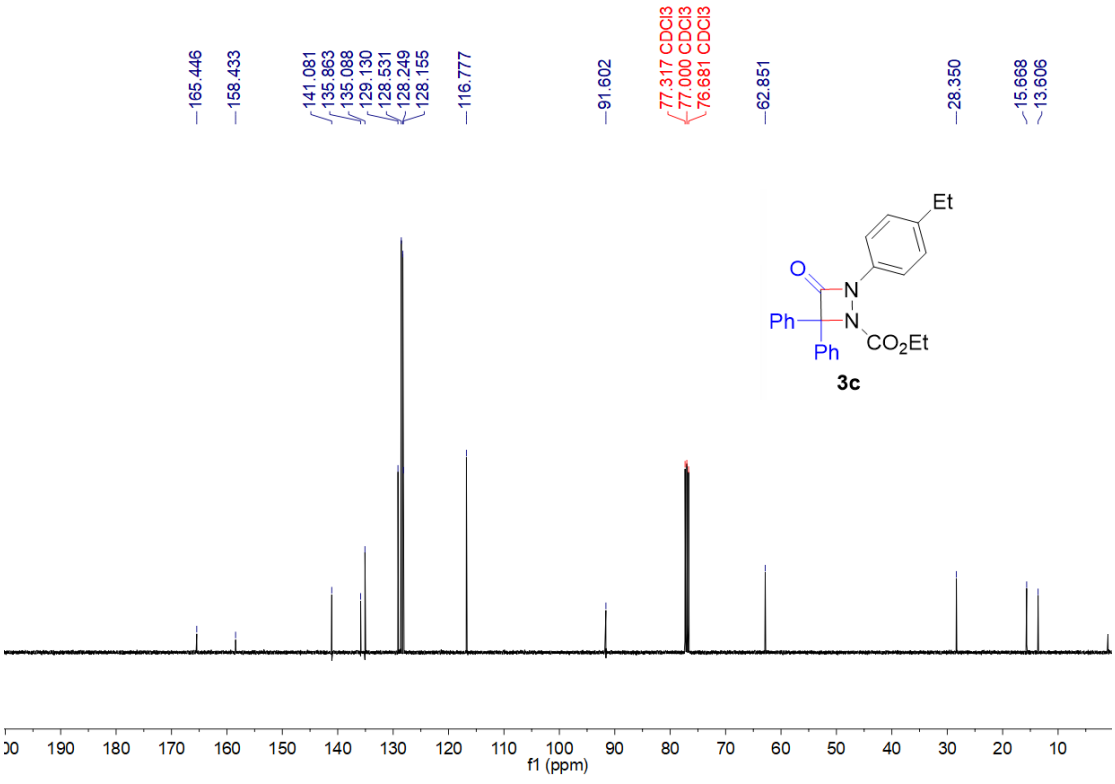
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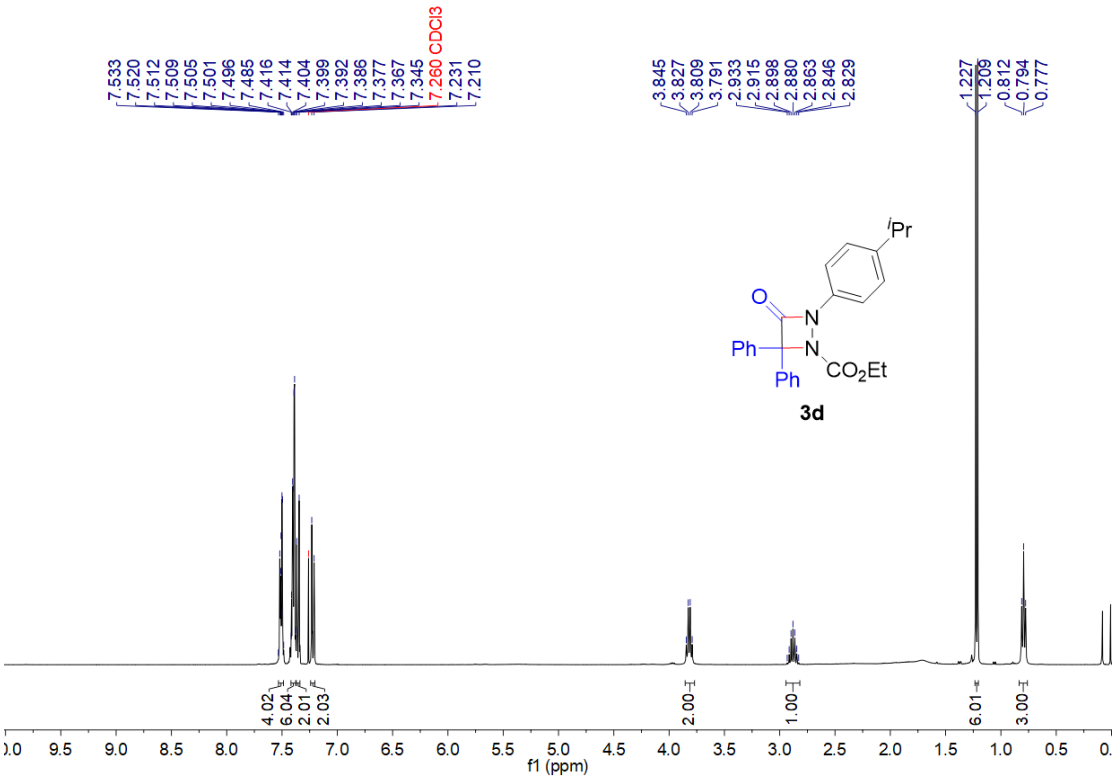
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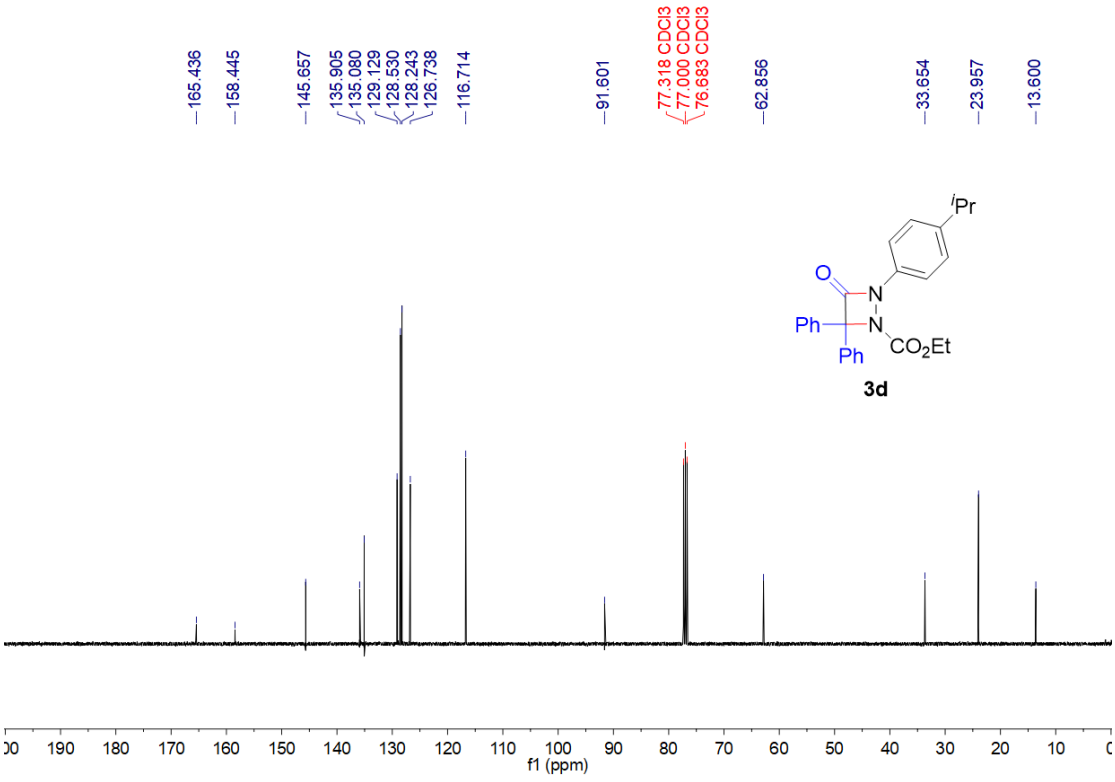
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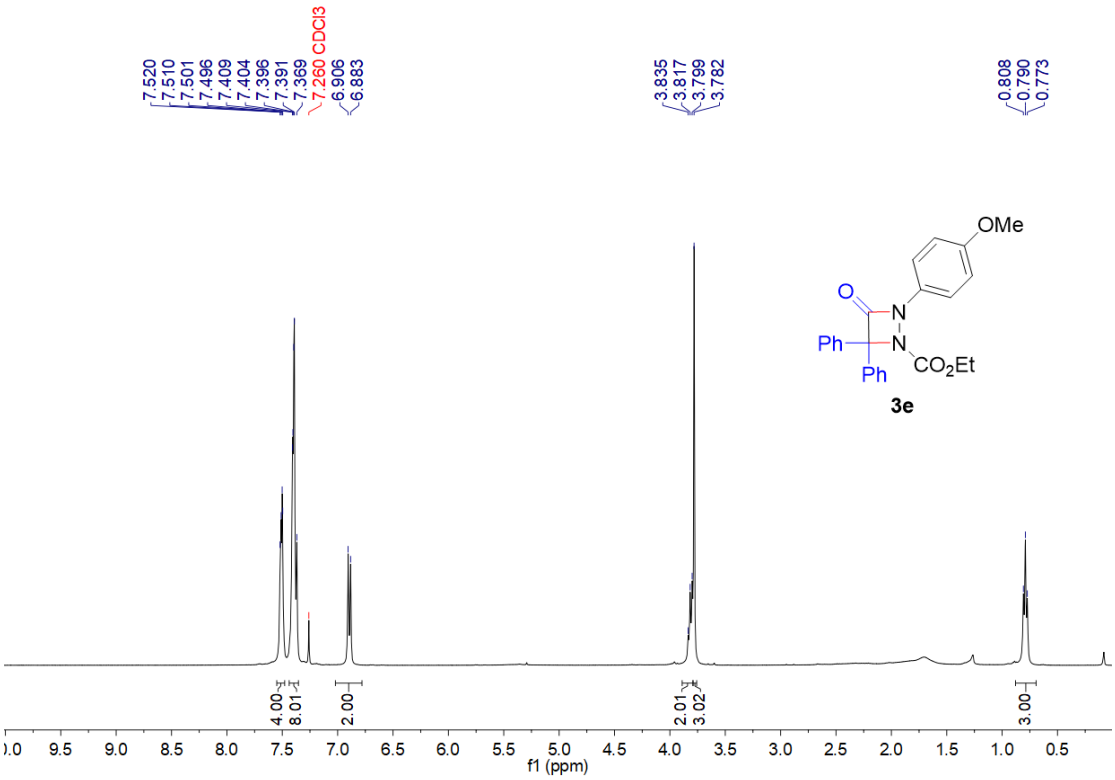
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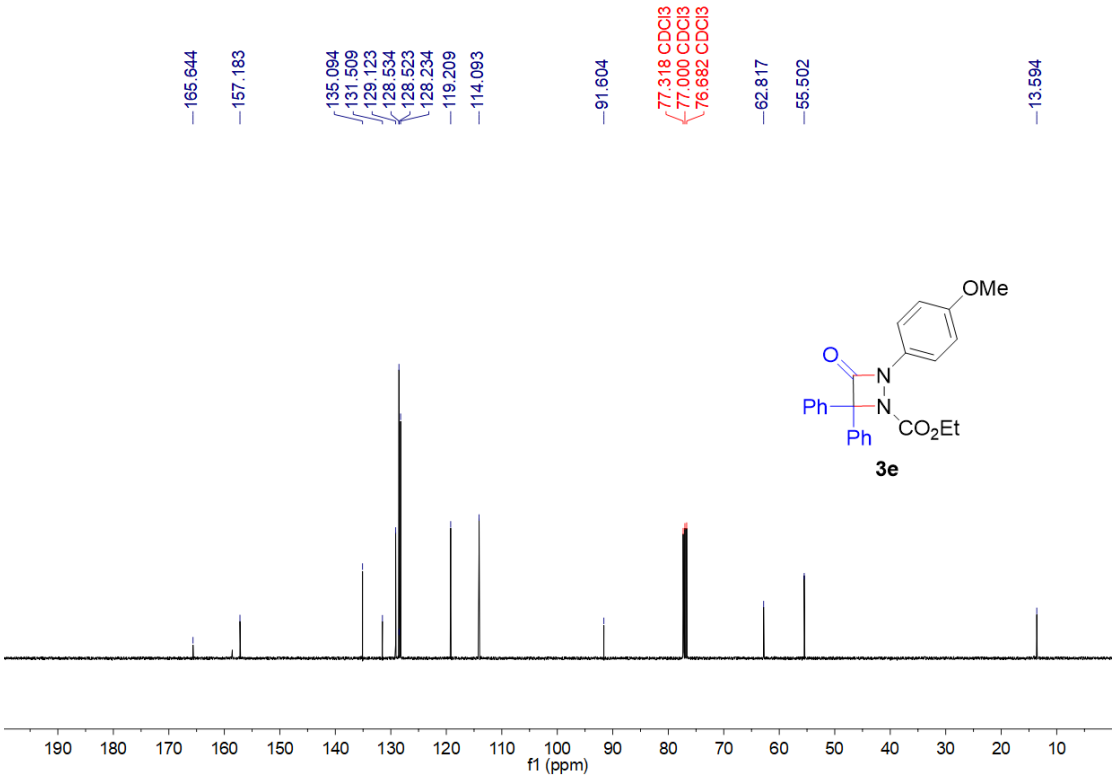
f1 (ppm)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OMe
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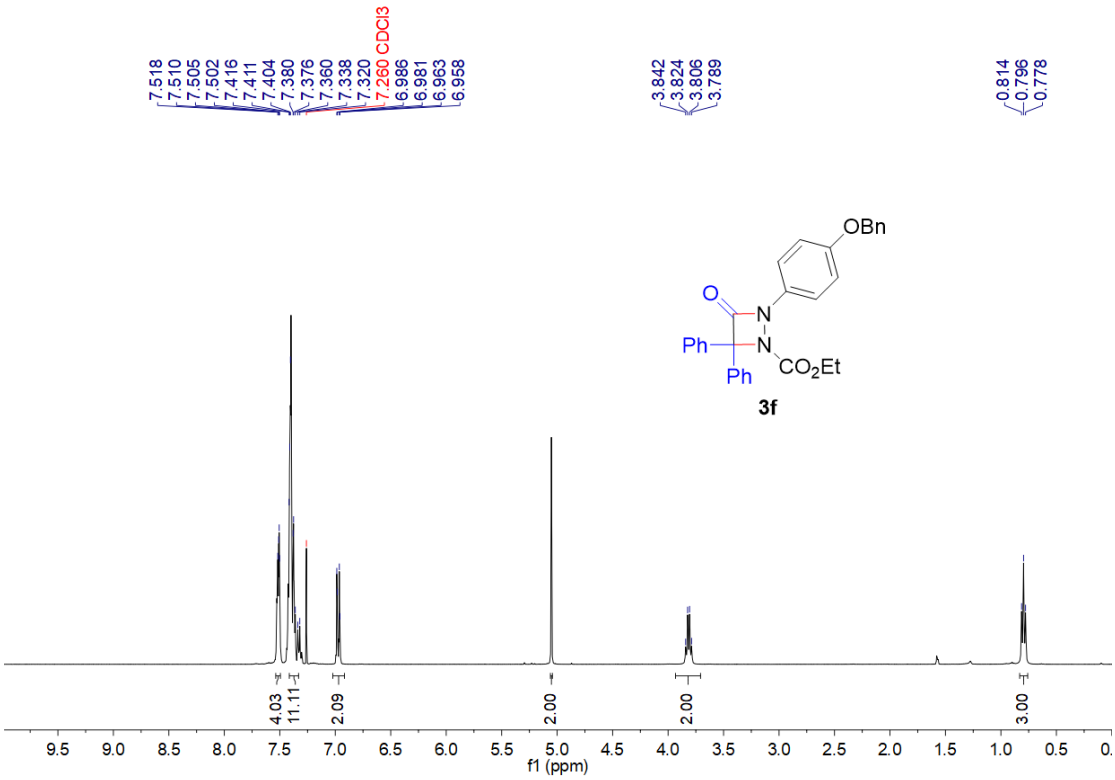
N
Ph
Ph
CO2Et
3e
f1 (ppm)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7.510
7.505
7.502
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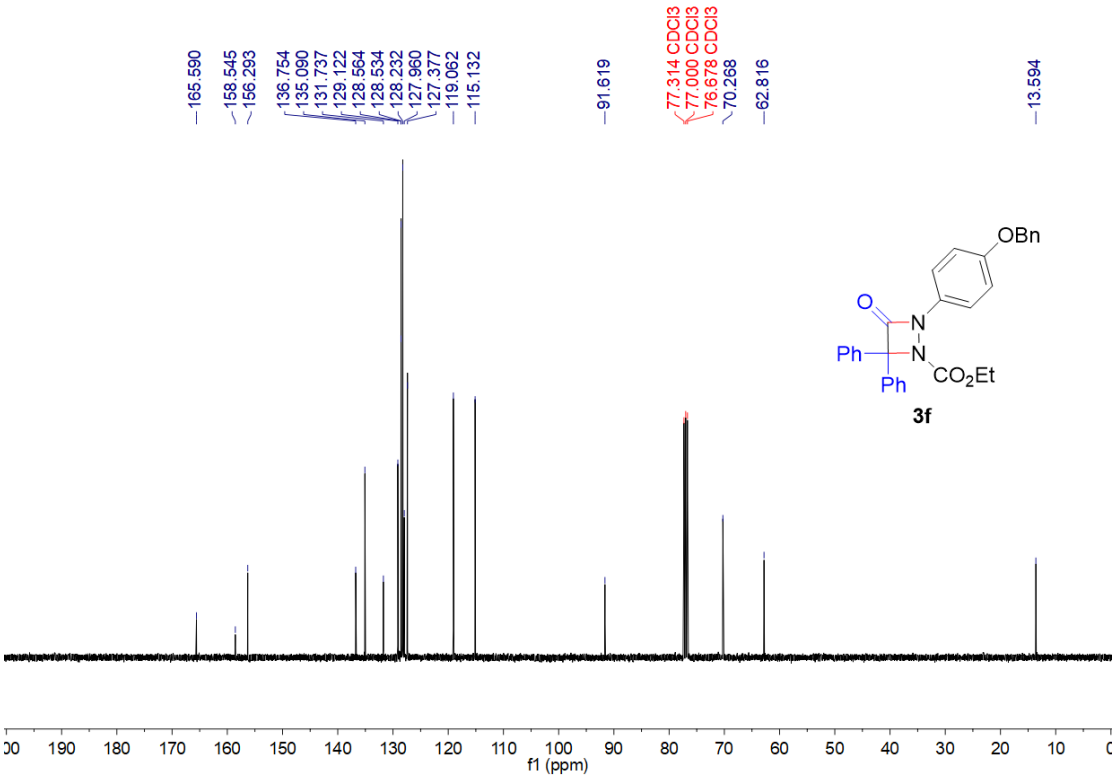
7.411
7.404
7.380
7.376
7.360
7.338
7.320
7.260 CDCl3
6.986
6.981
6.963
6.958
3.842
3.824
3.806
3.789
0.814
0.796
0.778
OBn
O
N
N
Ph
Ph
CO2Et
3f
4.03
11.11
2.09
2.00
2.00
3.00
f1 (ppm)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135.090
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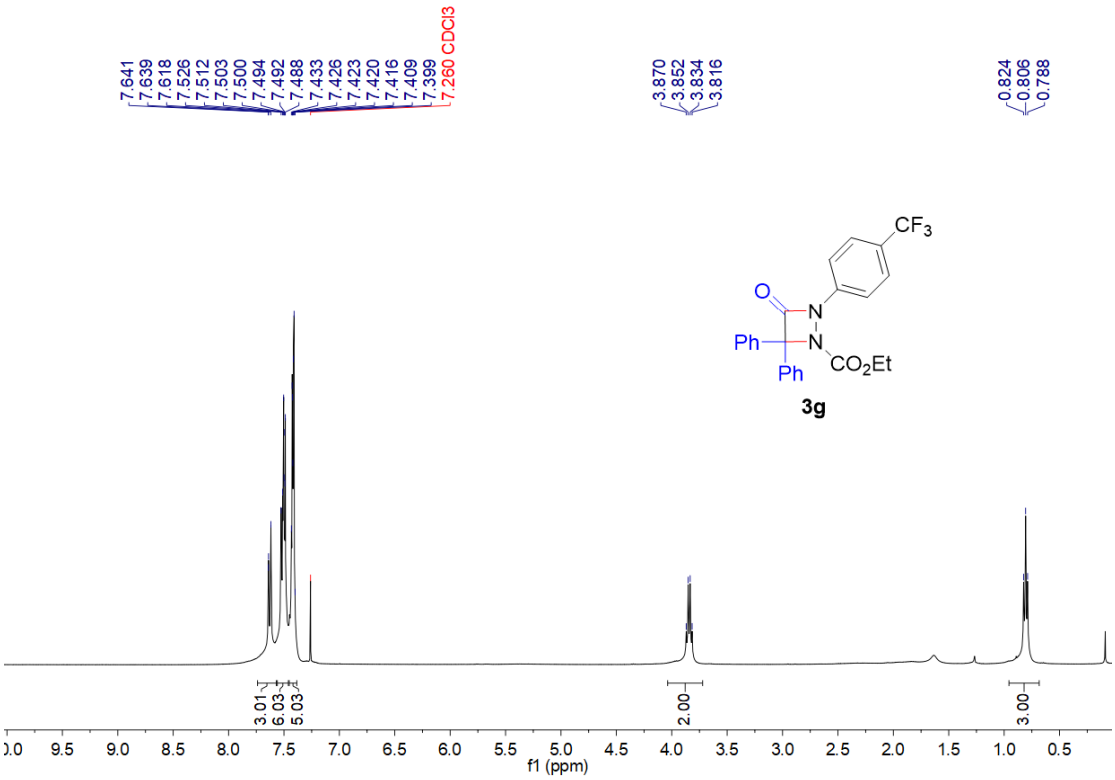
129.122
128.564
128.534
128.232
127.960
127.377
119.062
115.132
91.619
77.314 CDCl3
77.000 CDCl3
76.678 CDCl3
70.268
62.816
13.594
OBn
O
N
N
Ph
Ph
CO2Et
3f
f1 (ppm)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7.500
7.494
7.492
7.488
7.433
7.426
7.423
7.420
7.416
7.409
7.399
7.260 CDCl3
3.870
3.852
3.834
3.816
0.824
0.806
0.788
CF3
O
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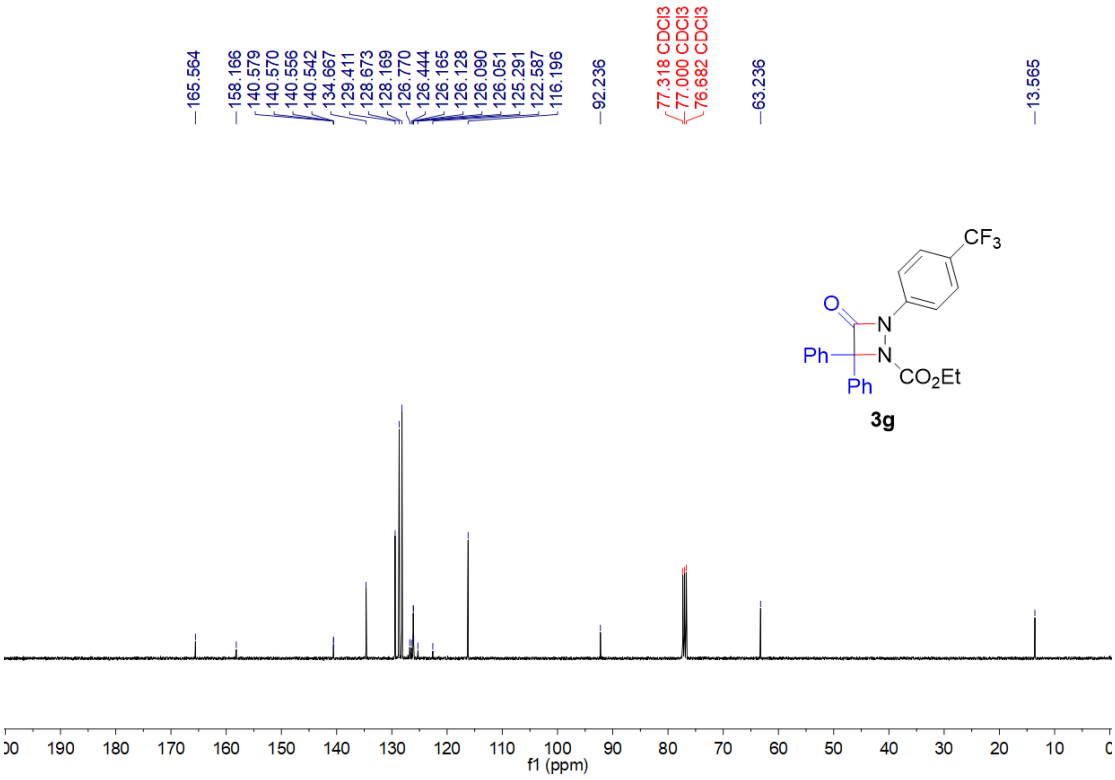
Ph
N
CO2Et
Ph
3g
3.01
6.03
5.03
2.00
3.00
f1 (ppm)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140.542
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128.169
126.770
126.444
126.165
126.128
126.090
126.051
125.291
122.587
116.196
92.236
77.318 CDCl3
77.000 CDCl3
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63.236
13.565
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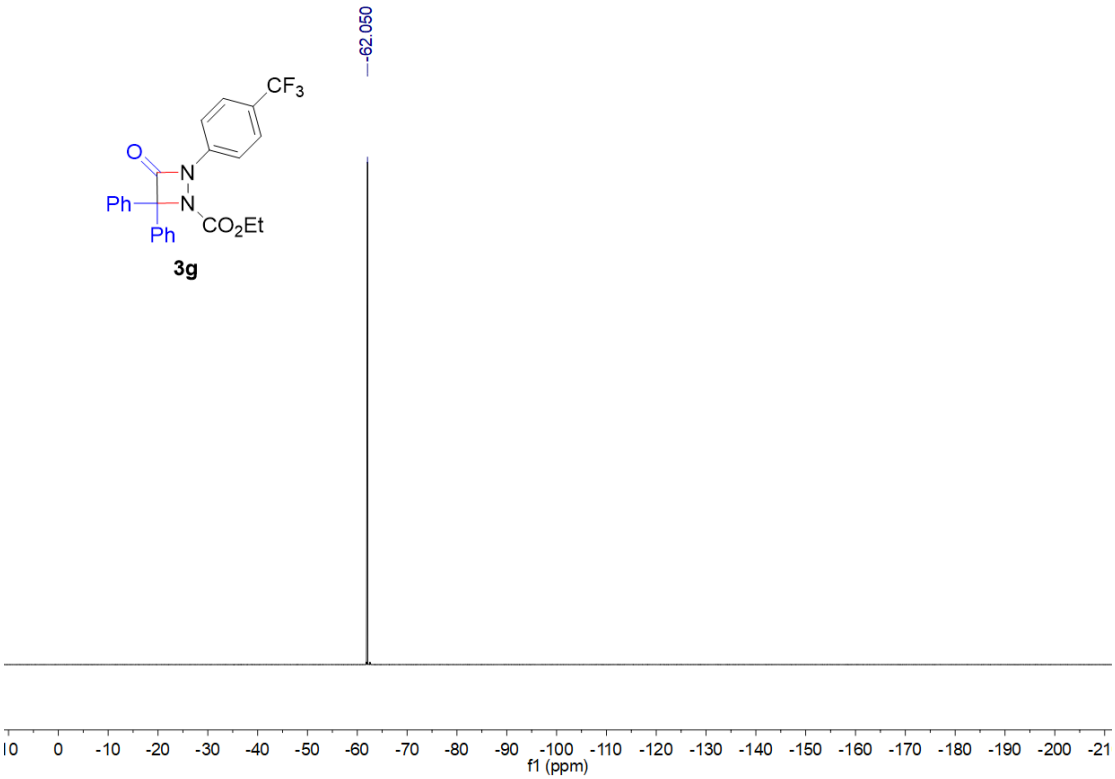
N
Ph
N
CO2Et
Ph
3g
f1 (ppm)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CF3
O
N
N
Ph
Ph
CO2Et
3g
f1 (ppm)


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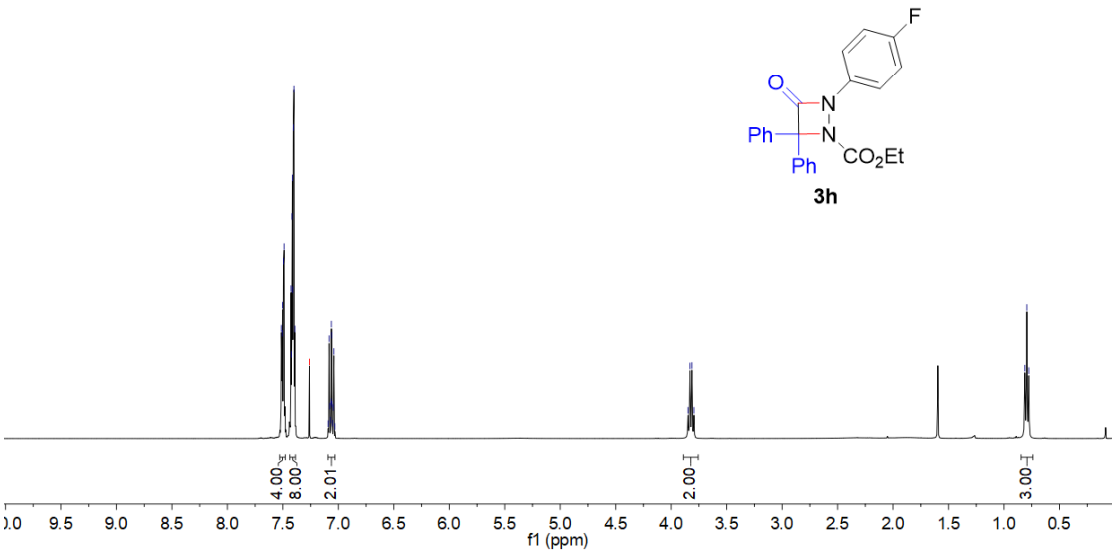
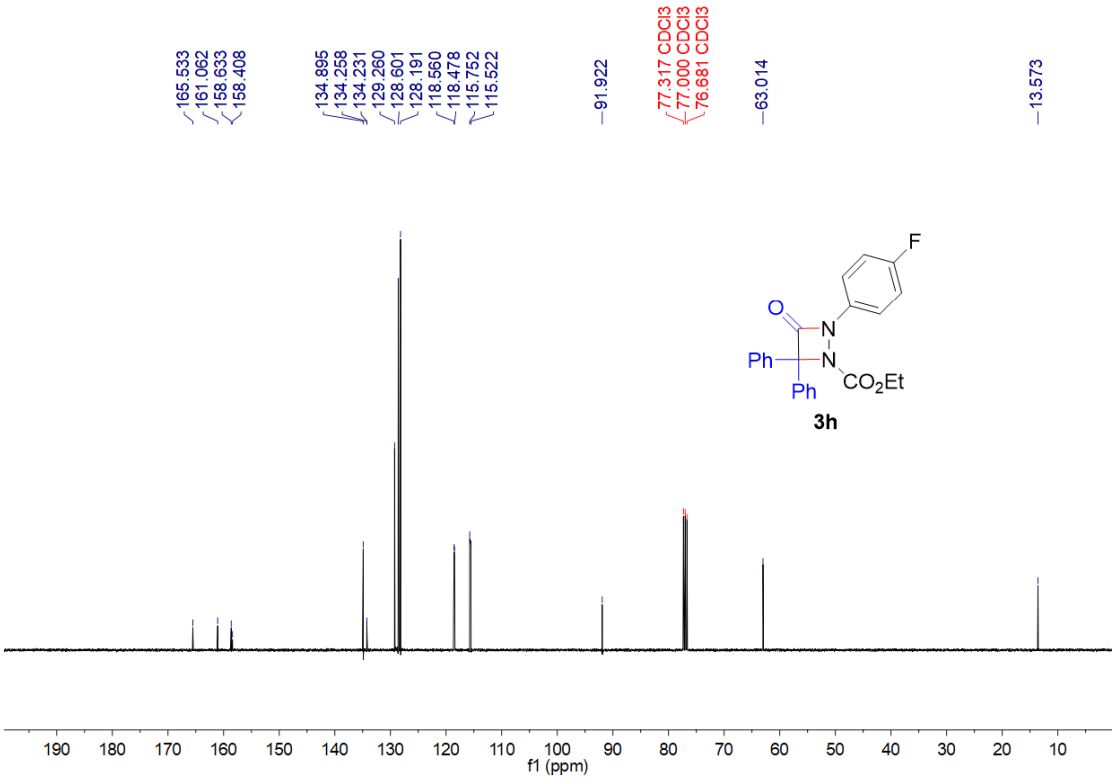
7.505
7.502
7.495
7.489
7.426
7.423
7.418
7.415
7.412
7.406
7.400
7.392
7.260 CDCl3
7.092
7.084
7.078
7.072
7.067
7.063
7.061
7.057
7.052
7.046
7.040
7.032
3.849
3.831
3.813
3.795
0.812
0.795
0.777


F
O
N
N
Ph
Ph
CO2Et
3h
4.00
8.00
2.01
2.00
3.00
f1 (ppm)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158.633
158.408
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134.258
134.231
129.260
128.601
128.191
118.560
118.478
115.752
115.522
91.922
77.317 CDCl3
77.000 CDCl3
76.681 CDCl3
63.014
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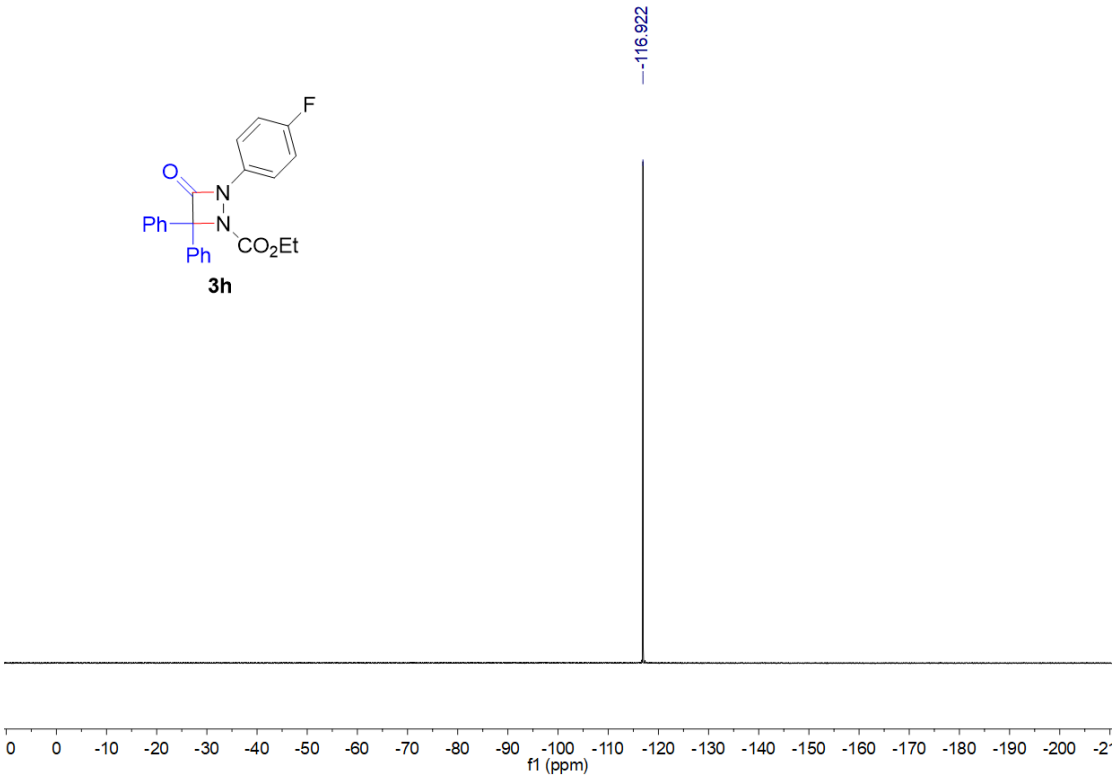
F
O
N
Ph
N
CO2Et
Ph
3h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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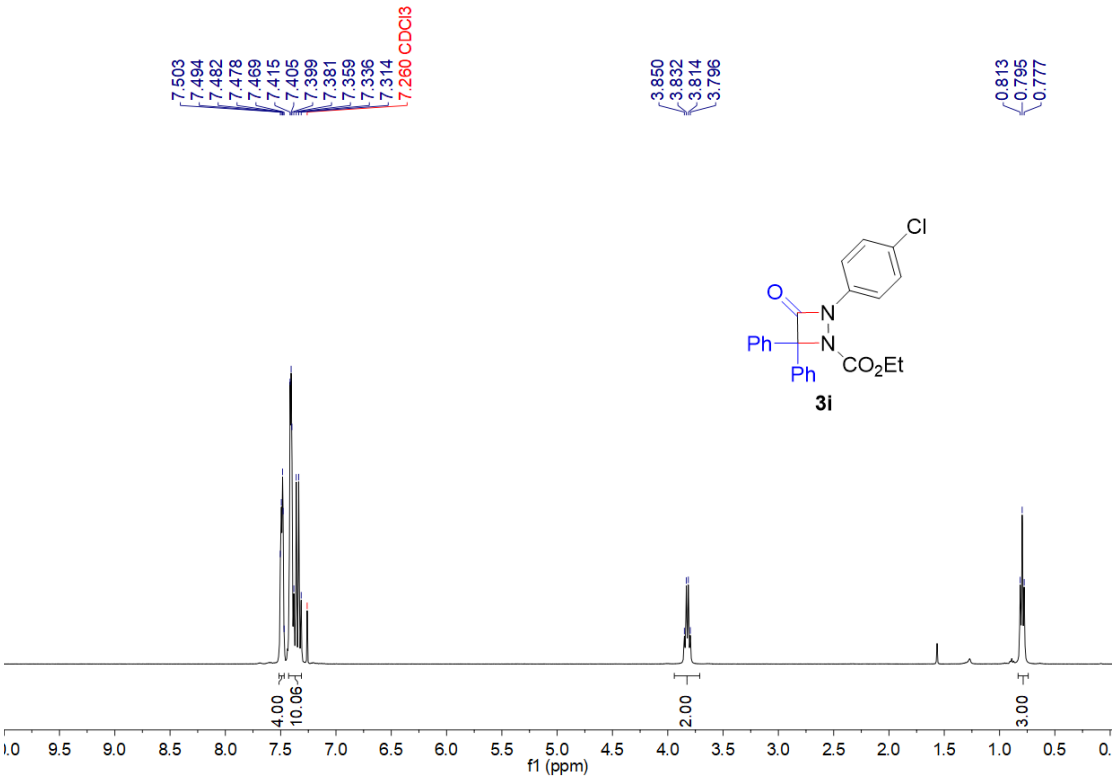
F
O
N
Ph
N
CO2Et
Ph
3h
0 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)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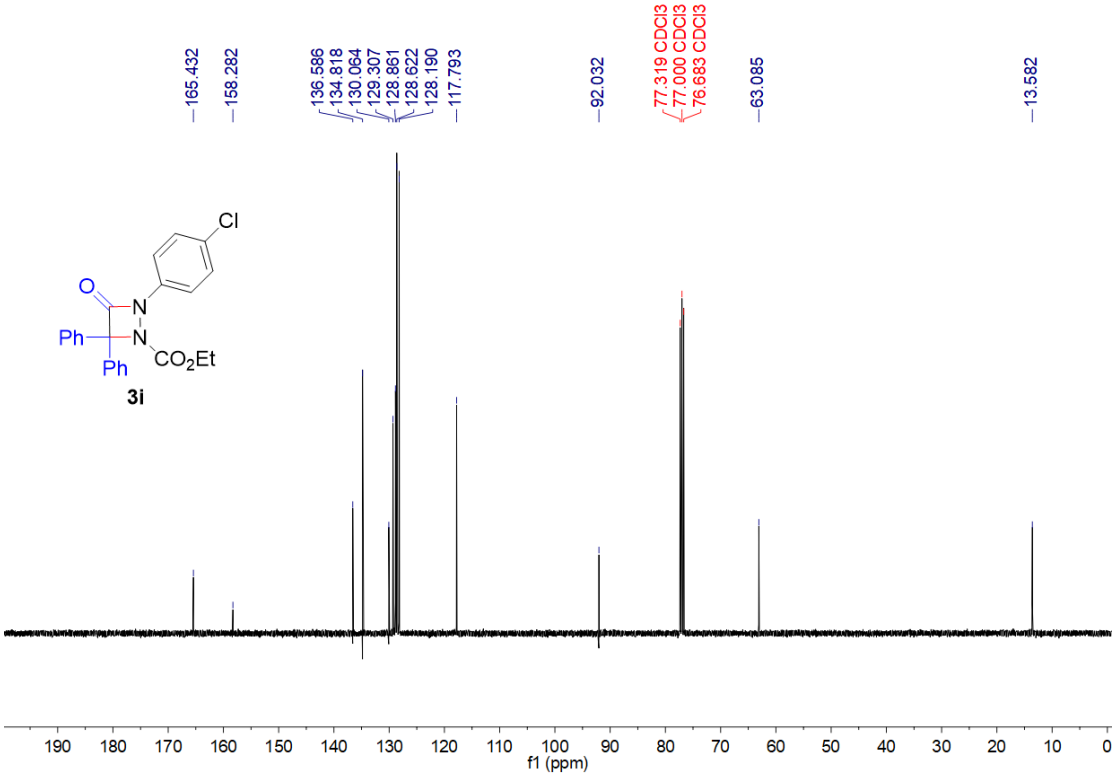
7.494
7.482
7.478
7.469
7.415
7.405
7.399
7.381
7.359
7.336
7.314
7.260 CDCl3
3.850
3.832
3.814
3.796
0.813
0.795
0.777
Cl
O
N
Ph
N
CO2Et
Ph
3i
4.00
10.06
2.00
3.00
f1 (ppm)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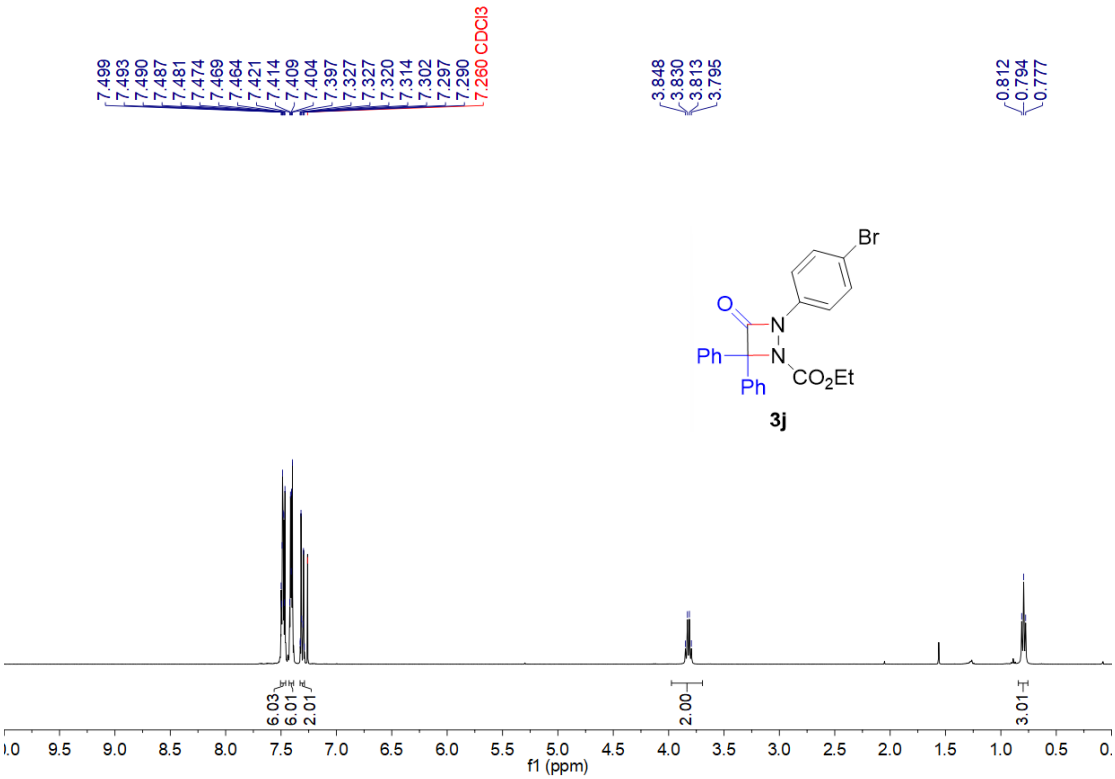
158.282
136.586
134.818
130.064
129.307
128.861
128.622
128.190
117.793
92.032
77.319 CDCl3
77.000 CDCl3
76.683 CDCl3
63.085
13.582
Cl
O
N
Ph
N
CO2Et
Ph
3i
f1 (ppm)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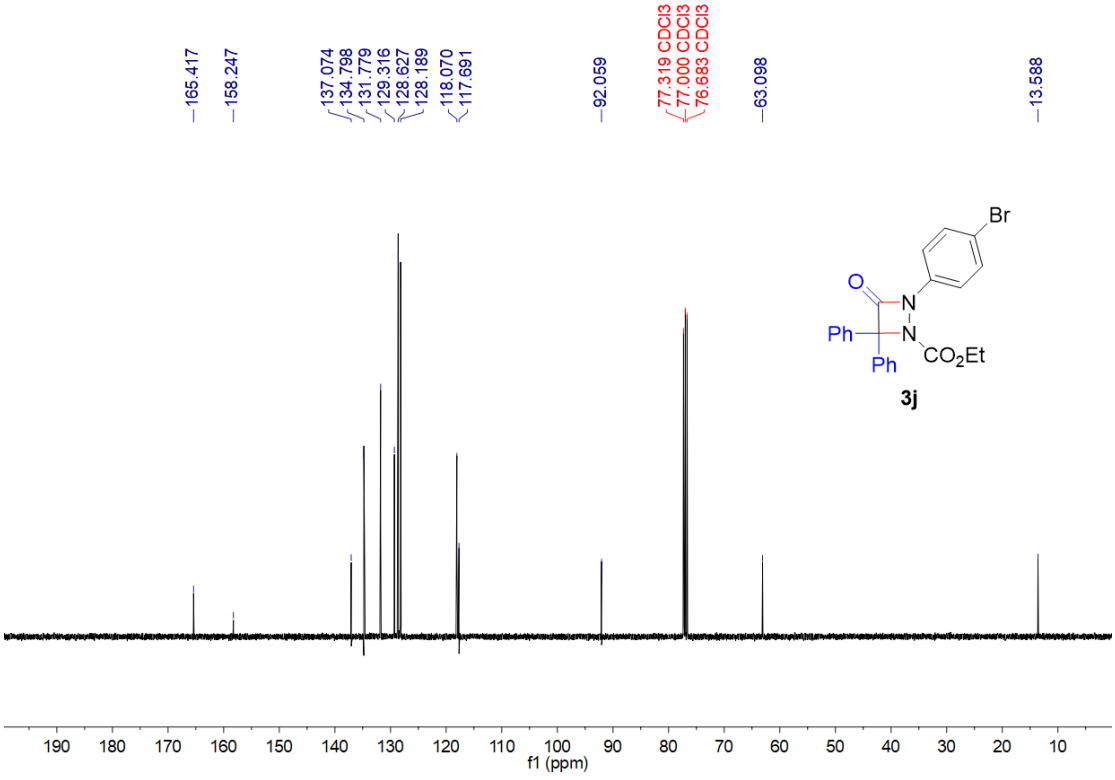
7.493
7.490
7.487
7.481
7.474
7.469
7.464
7.421
7.414
7.409
7.404
7.397
7.327
7.320
7.314
7.302
7.297
7.290
7.260 CDCl3
3.848
3.830
3.813
3.795
0.812
0.794
0.777
Br
O
N
N
Ph
Ph
CO2Et
3j
6.03
6.01
2.01
2.00
3.01
f1 (ppm)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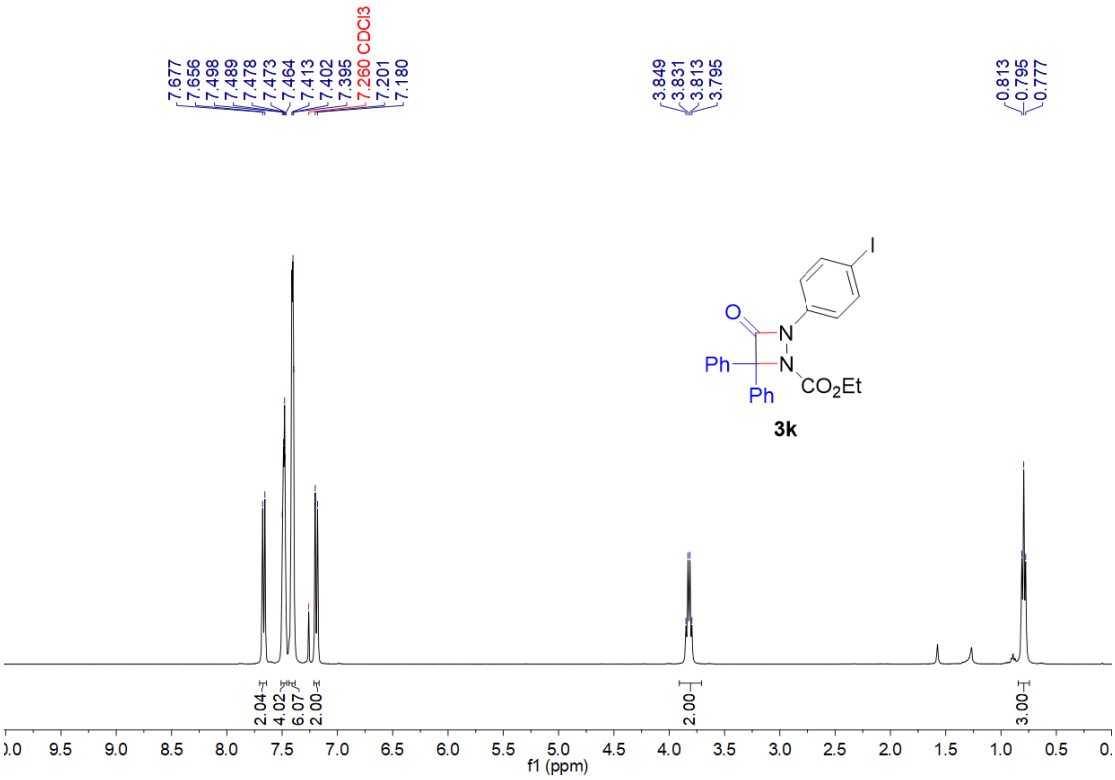
128.627
128.189
118.070
117.691
92.059
77.319 CDCl3
77.000 CDCl3
76.683 CDCl3
63.098
13.588
Br
O
N
N
Ph
Ph
CO2Et
3j
f1 (ppm)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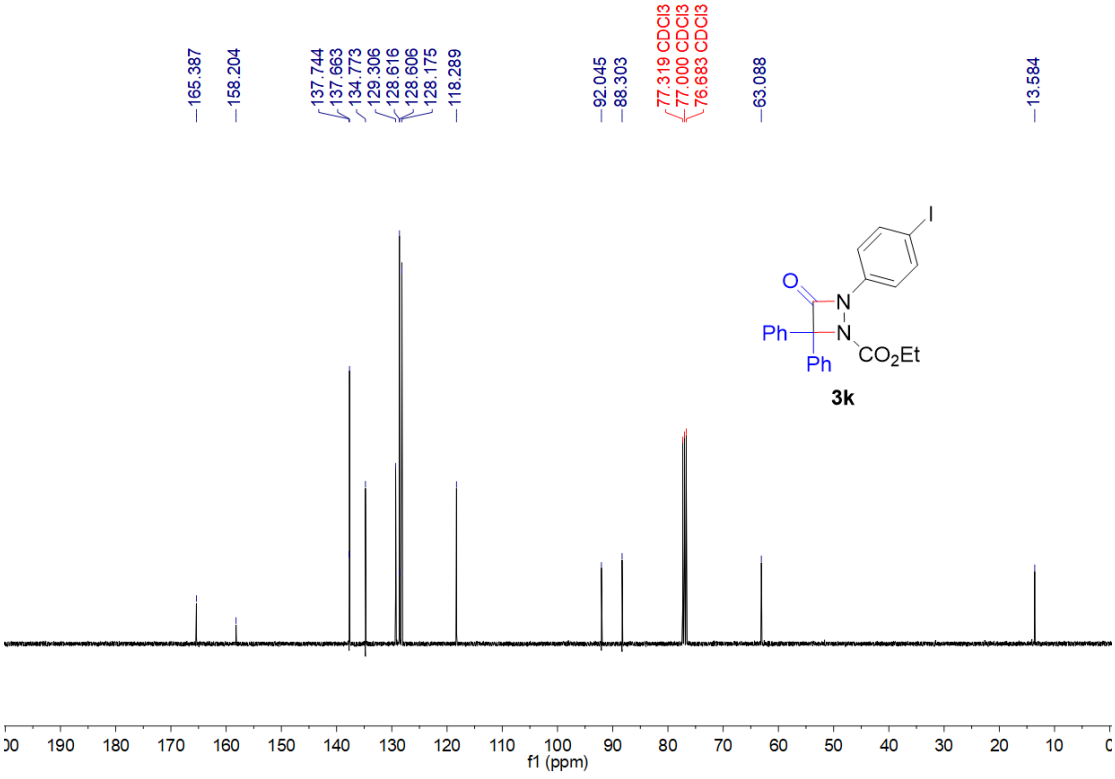
7.464
7.413
7.402
7.396
7.260 CDCl3
7.201
7.180
3.849
3.831
3.813
3.795
0.813
0.795
0.777
I
O
N
Ph
N
CO2Et
Ph
3k
2.04
4.02
6.07
2.00
2.00
3.00
f1 (ppm)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137.663
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129.306
128.616
128.606
128.175
118.289
92.045
88.303
77.319 CDCl3
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63.088
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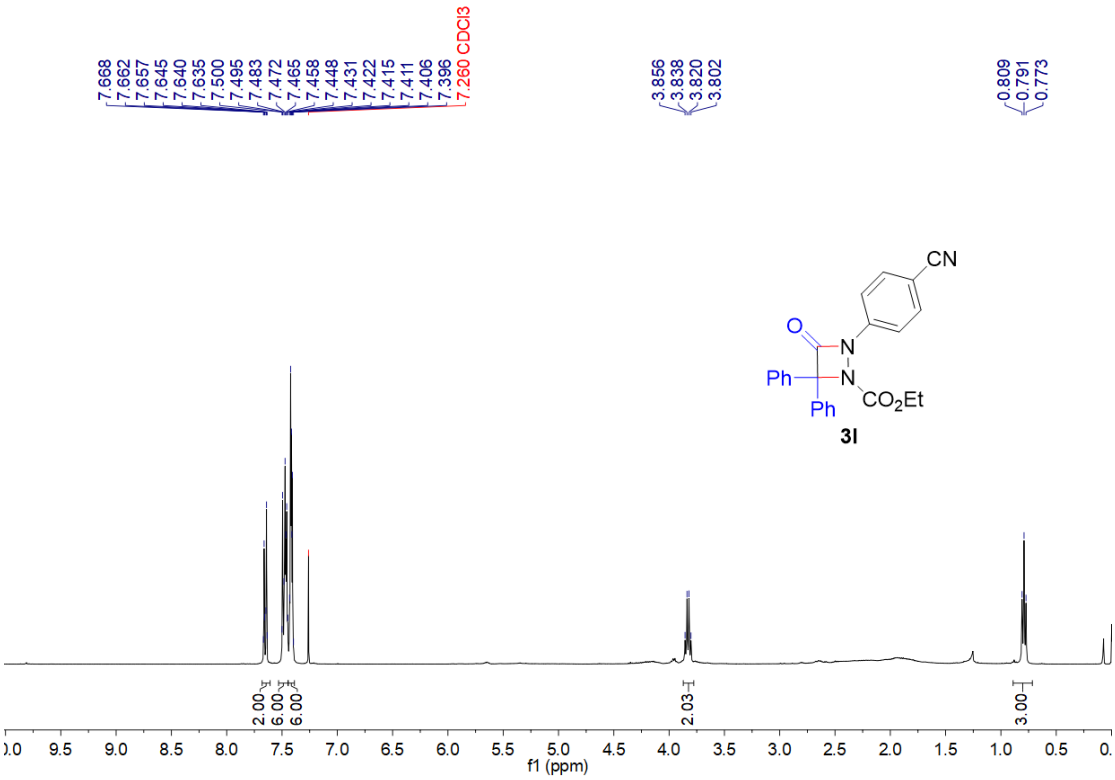
I
O
N
Ph
N
CO2Et
Ph
3k
f1 (ppm)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7.635
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7.495
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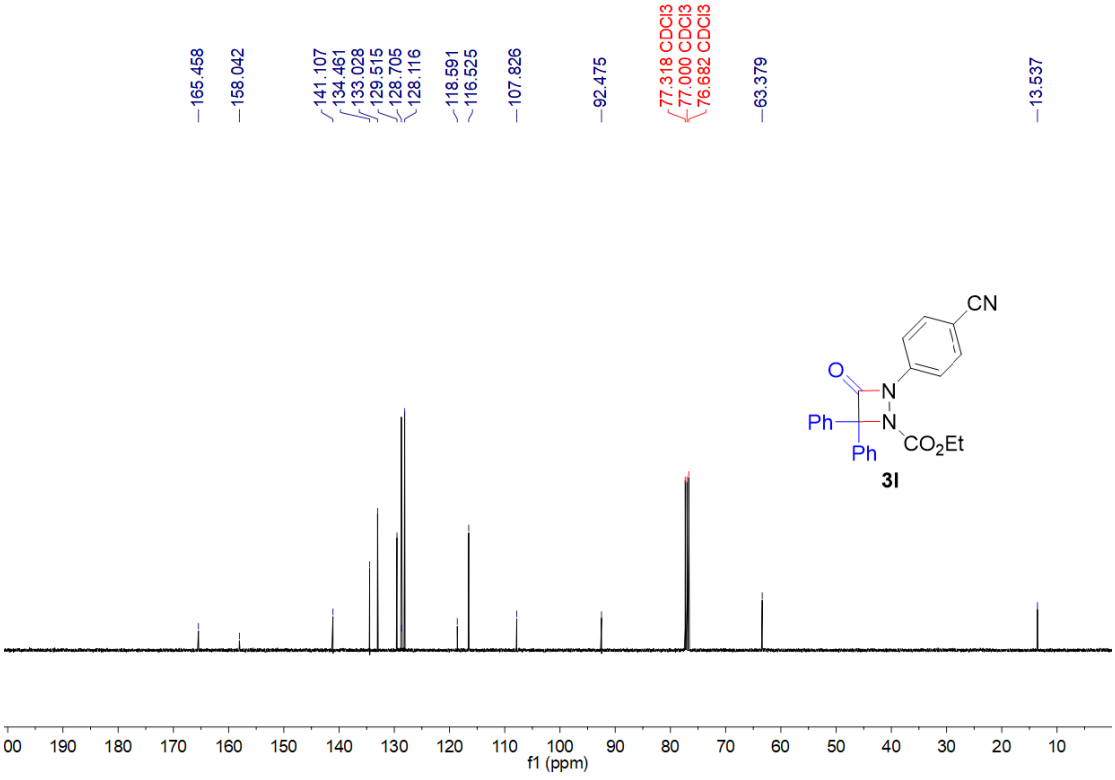
7.472
7.465
7.458
7.448
7.431
7.422
7.415
7.411
7.406
7.396
7.260 CDCl3
3.856
3.838
3.820
3.802
0.809
0.791
0.773
CN
O
N
N
Ph
Ph
CO2Et
3l
2.00
6.00
6.00
2.03
3.00
f1 (ppm)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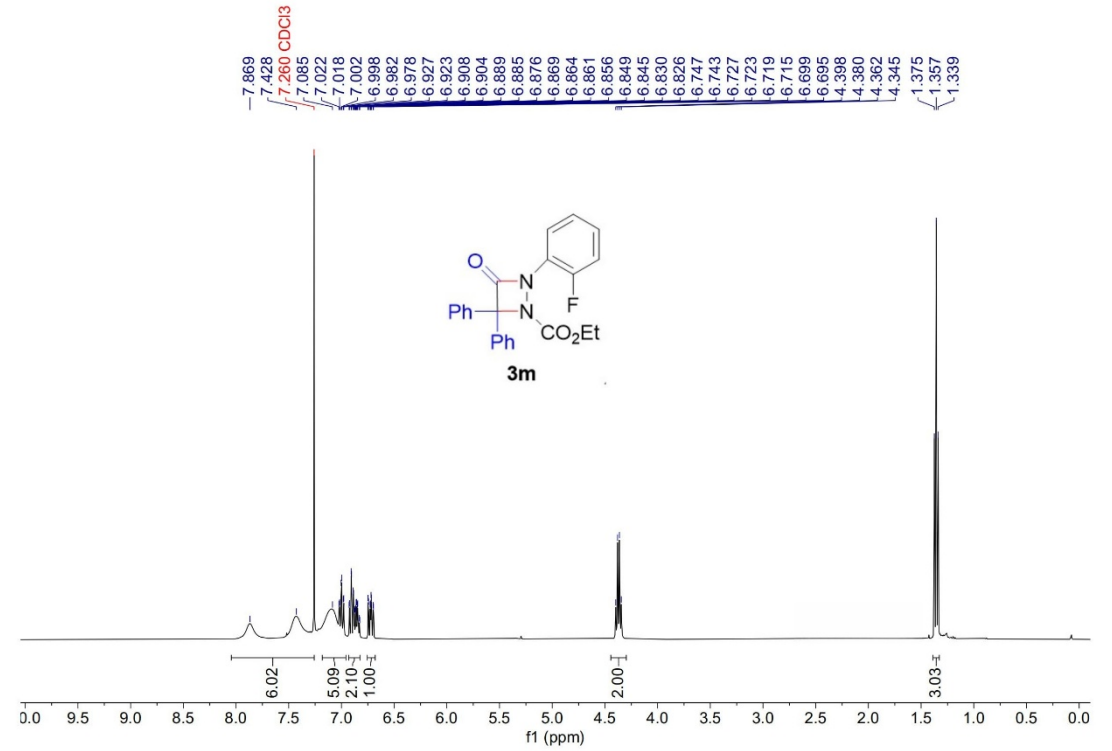
128.705
128.116
118.591
116.525
107.826
92.475
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
63.379
13.537
CN
O
N
N
Ph
Ph
CO2Et
3l
f1 (ppm)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7.002
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6.982
6.978
6.927
6.923
6.908
6.904
6.889
6.885
6.876
6.869
6.864
6.861
6.856
6.849
6.845
6.830
6.826
6.747
6.743
6.727
6.723
6.719
6.715
6.699
6.695
4.398
4.380
4.362
4.345
1.375
1.357
1.339
O
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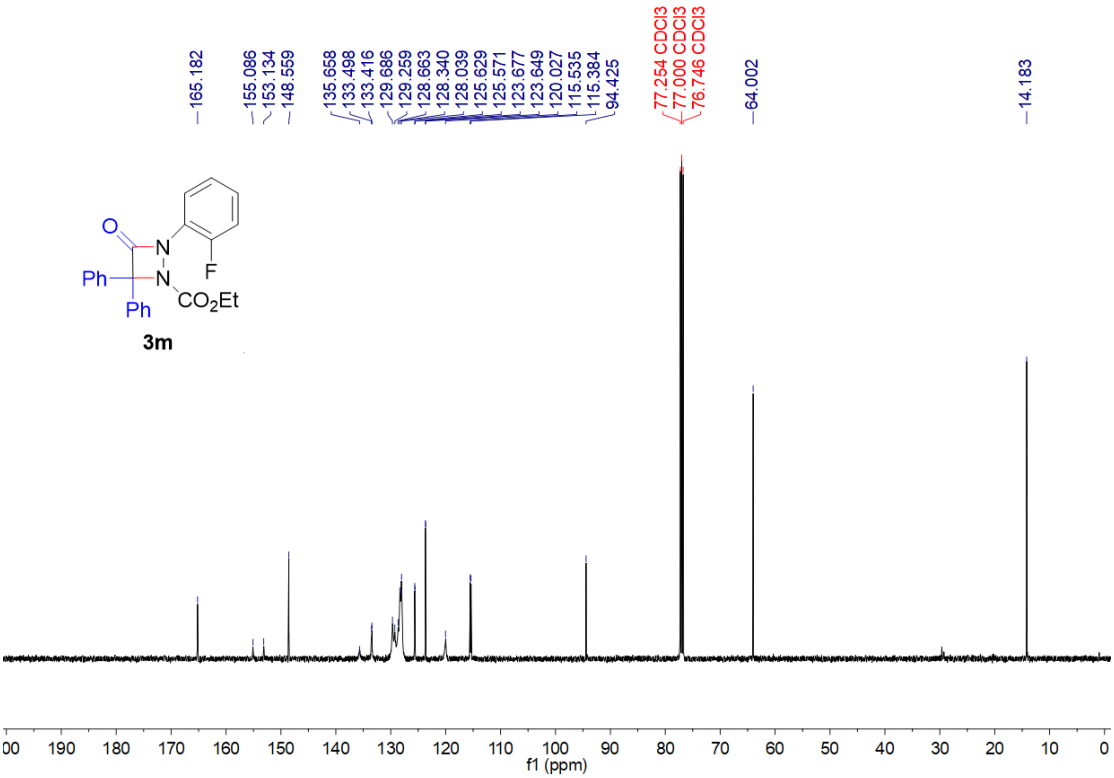
Ph
Ph
CO2Et
3m
6.02
5.09
2.10
1.00
2.00
3.03
f1 (ppm)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129.686
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128.863
128.340
128.039
125.629
125.571
123.677
123.649
120.027
115.535
115.384
94.425
77.254 CDCl3
77.000 CDCl3
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64.002
14.183
O
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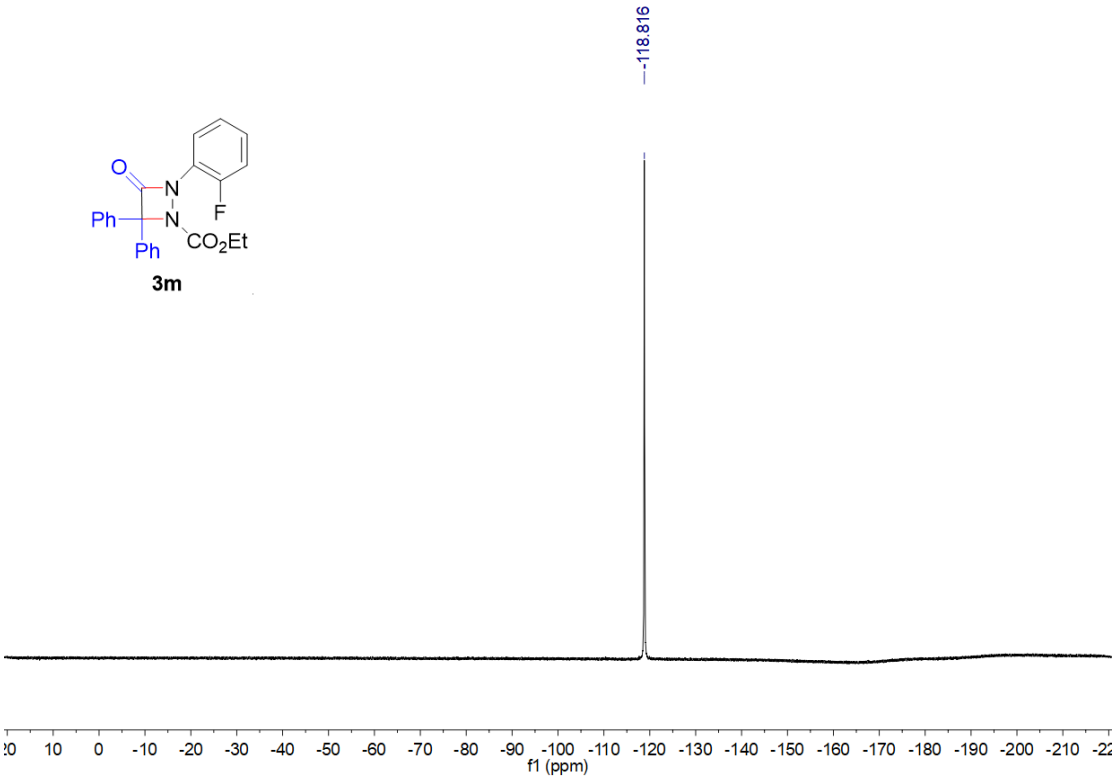
F
Ph
Ph
CO2Et
3m
f1 (ppm)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Ph
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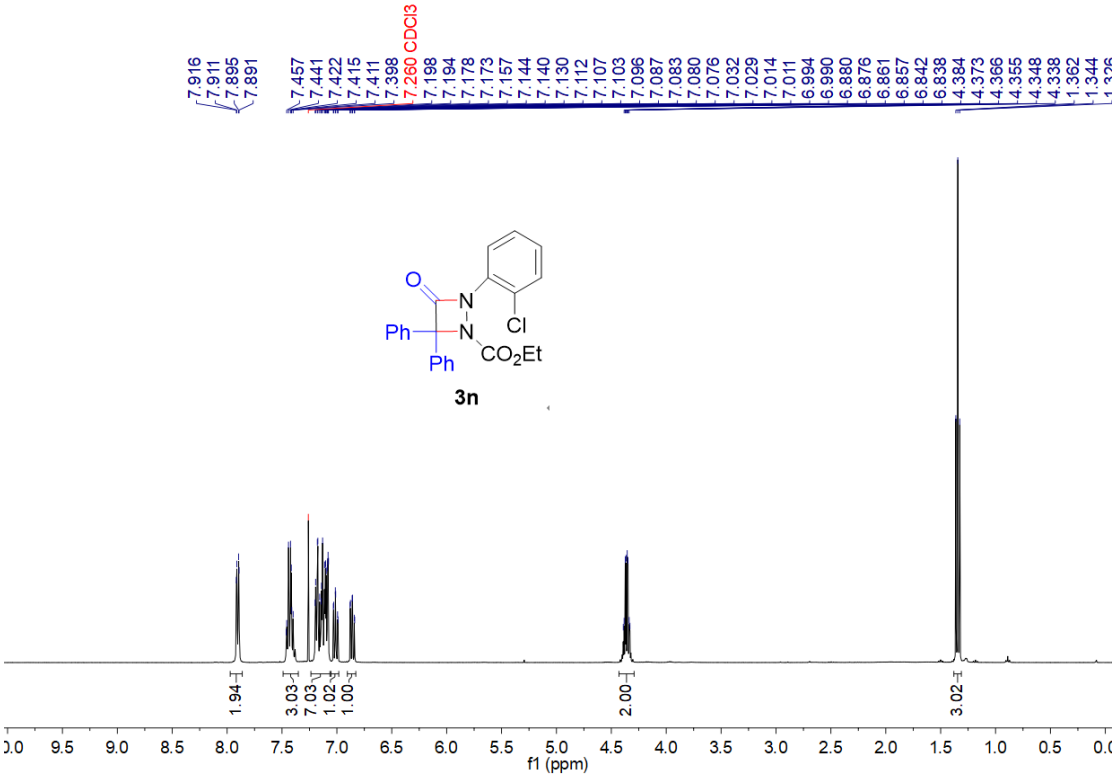
f1 (ppm)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7.411
7.398
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7.178
7.173
7.157
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7.130
7.112
7.107
7.103
7.096
7.087
7.083
7.080
7.076
7.032
7.029
7.014
7.011
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6.990
6.880
6.876
6.861
6.857
6.842
6.838
4.384
4.373
4.366
4.355
4.348
4.338
1.362
1.344
O
N
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Ph
Ph
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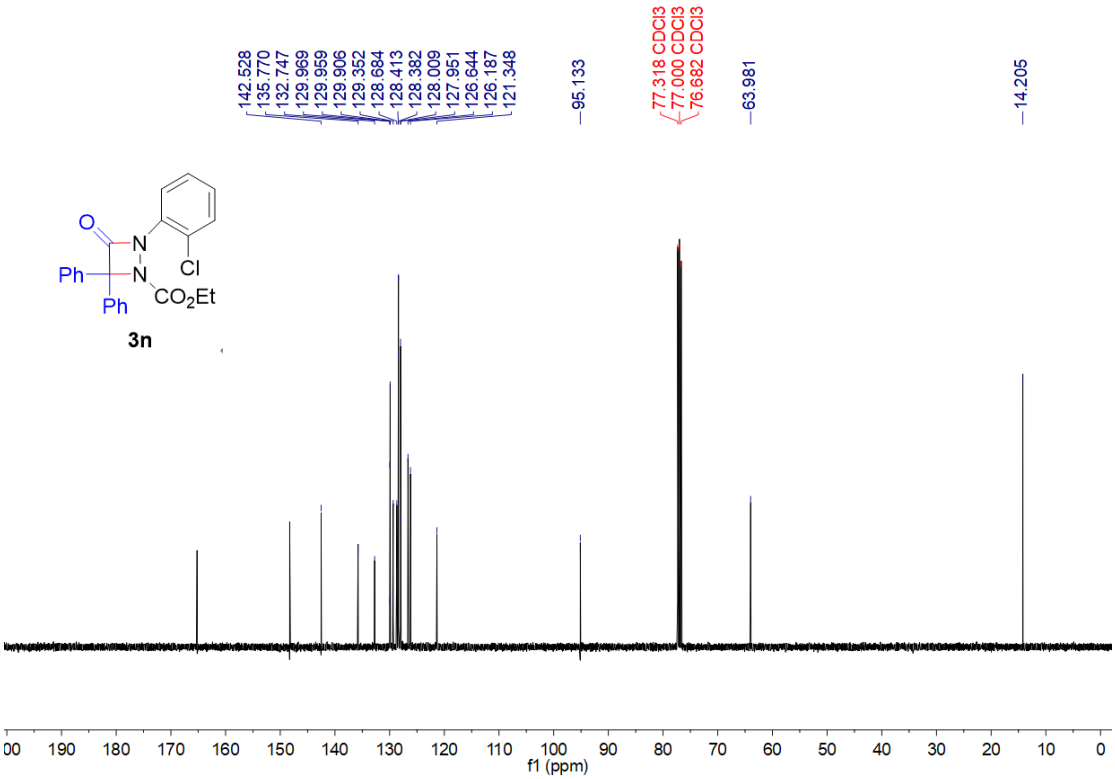
2.00
3.02
f1 (ppm)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127.951
126.644
126.187
121.348
95.133
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
63.981
14.205
O
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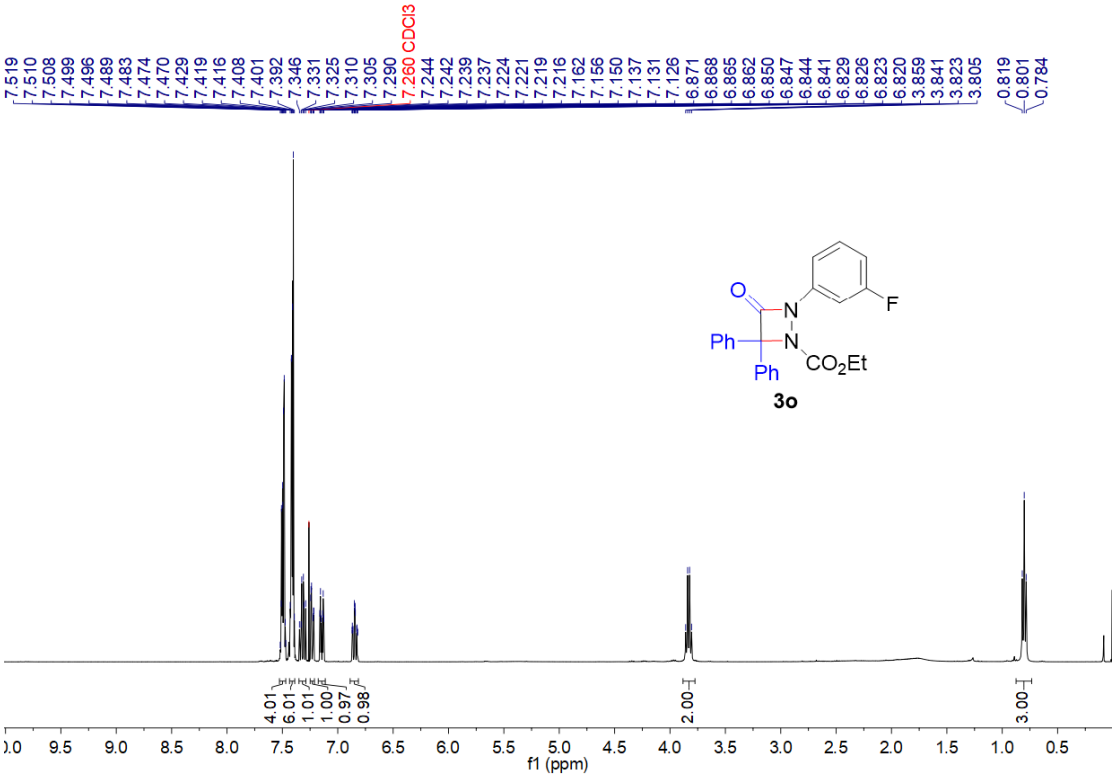
N
Cl
Ph
Ph
CO2Et
3n
f1 (ppm)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7.489
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7.474
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7.419
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7.408
7.401
7.392
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7.331
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7.305
7.290
7.260 CDCl3
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7.239
7.237
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7.221
7.219
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7.162
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7.126
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6.865
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6.850
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6.844
6.841
6.829
6.826
6.823
6.820
3.859
3.841
3.823
3.805
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0.801
0.784
O
N
N
F
Ph
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1.01
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0.98
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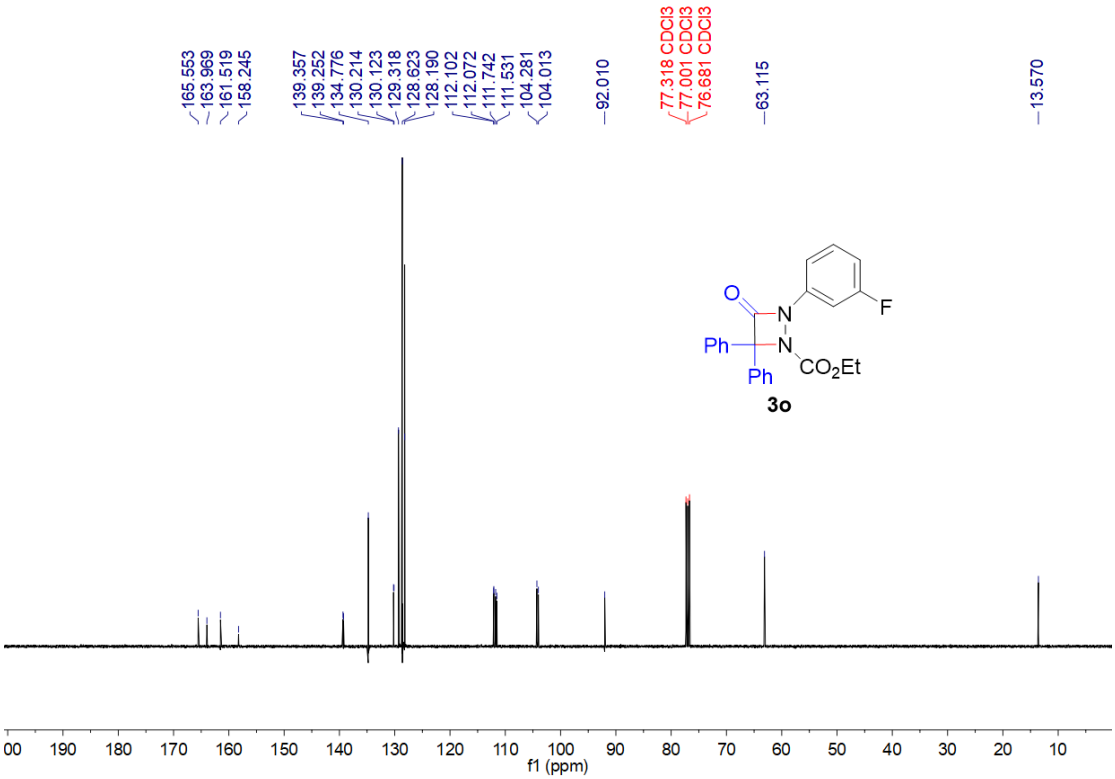
3.00
f1 (ppm)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129.318
128.623
128.190
112.102
112.072
111.742
111.531
104.281
104.013
92.010
77.318 CDCl3
77.001 CDCl3
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63.115
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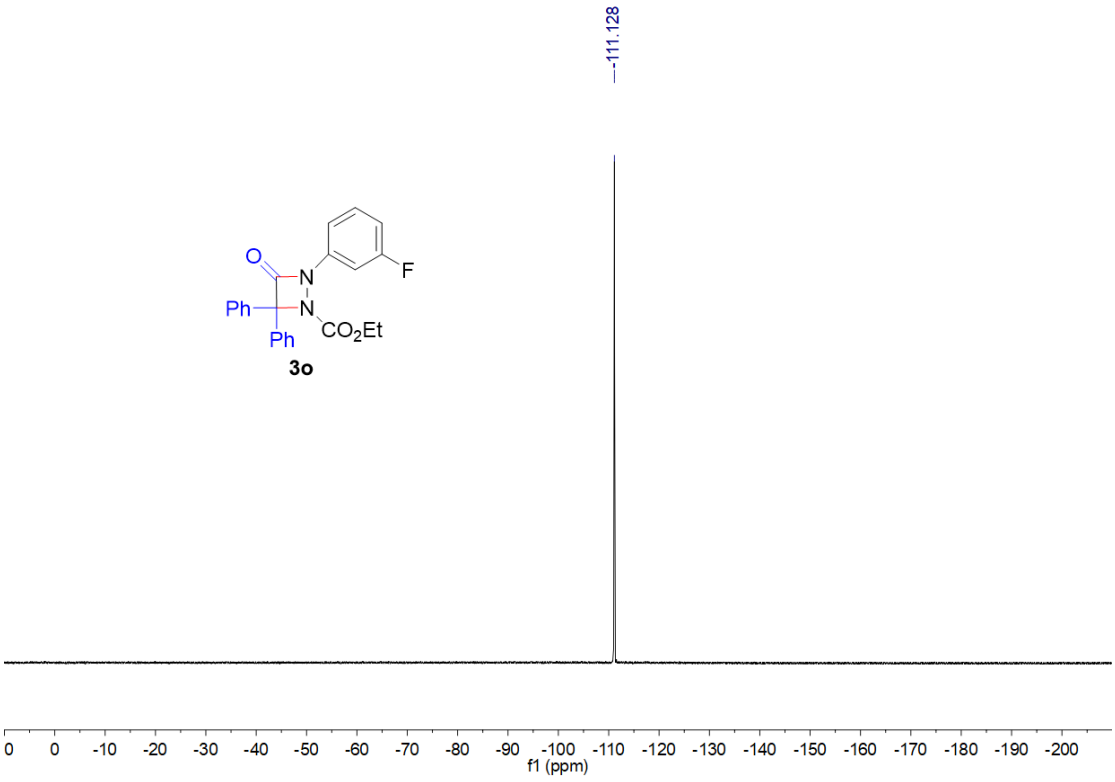
F
Ph
Ph
CO2Et
3o
f1 (ppm)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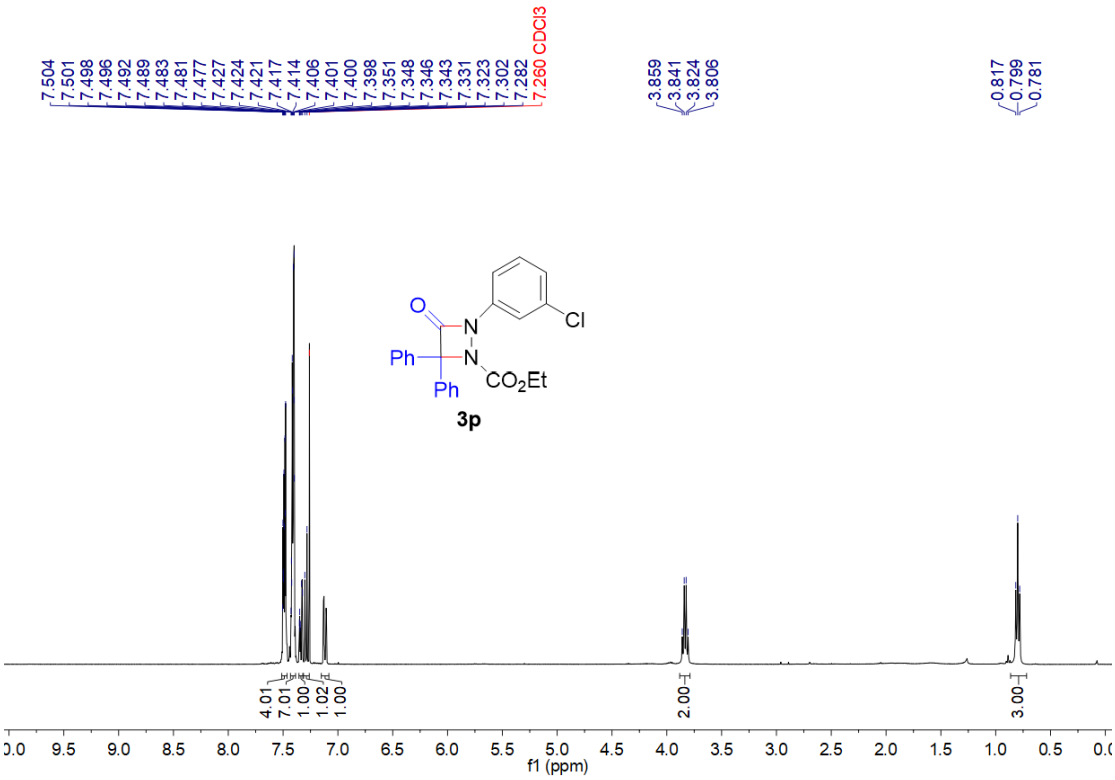
f1 (ppm)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7.498
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7.417
7.414
7.406
7.401
7.400
7.398
7.351
7.348
7.346
7.343
7.331
7.323
7.302
7.282
7.260 CDCl3
3.859
3.841
3.824
3.806
0.817
0.799
0.781
O
N
N
Cl
Ph
Ph
CO2Et
3p
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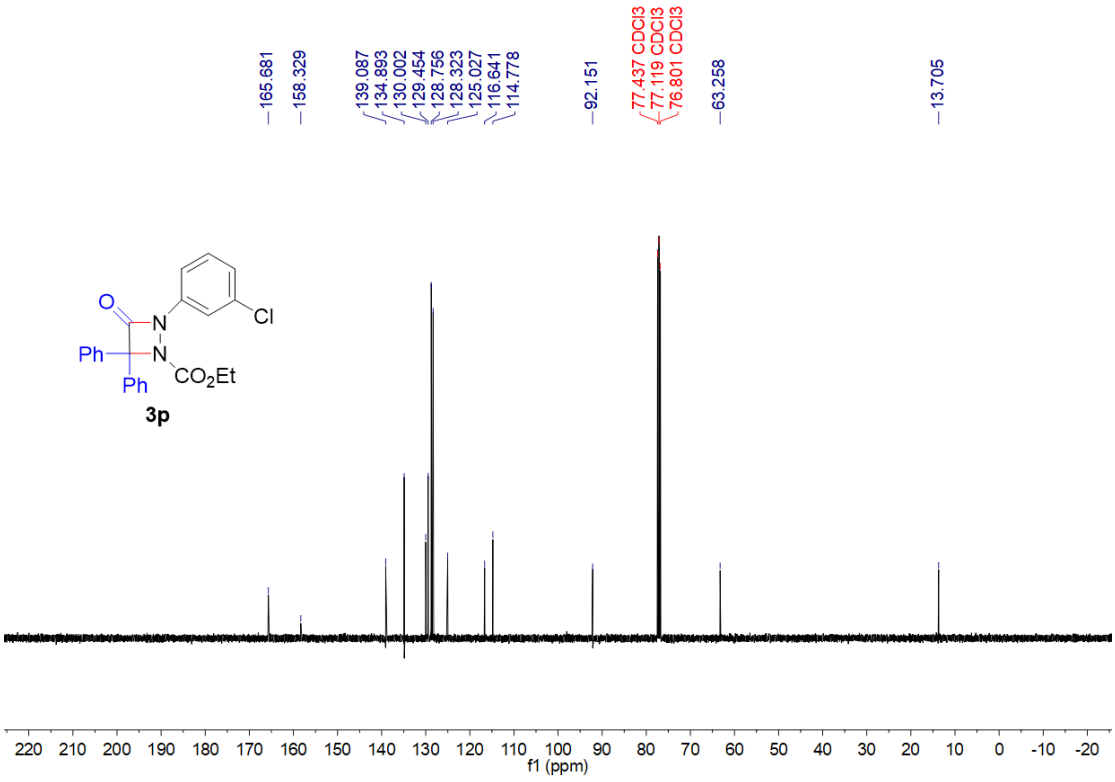
7.01
1.00
1.02
1.00
2.00
3.00
f1 (ppm)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92.151
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Ph
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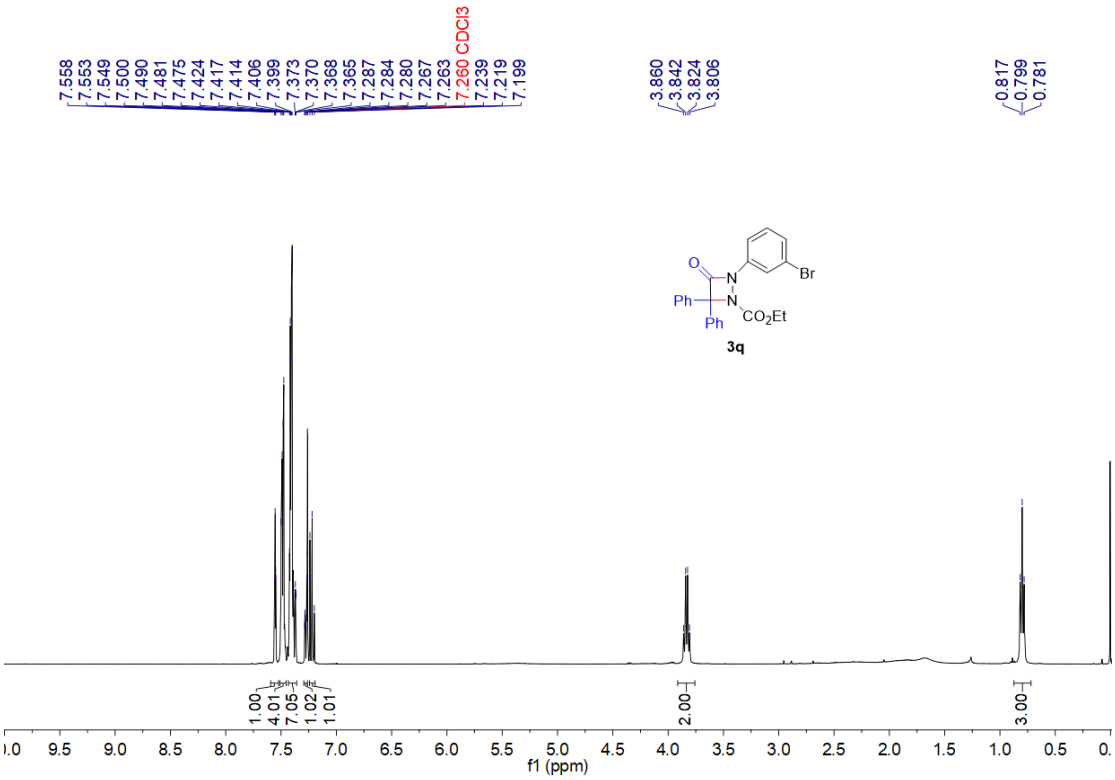
CO2Et
3p
f1 (ppm)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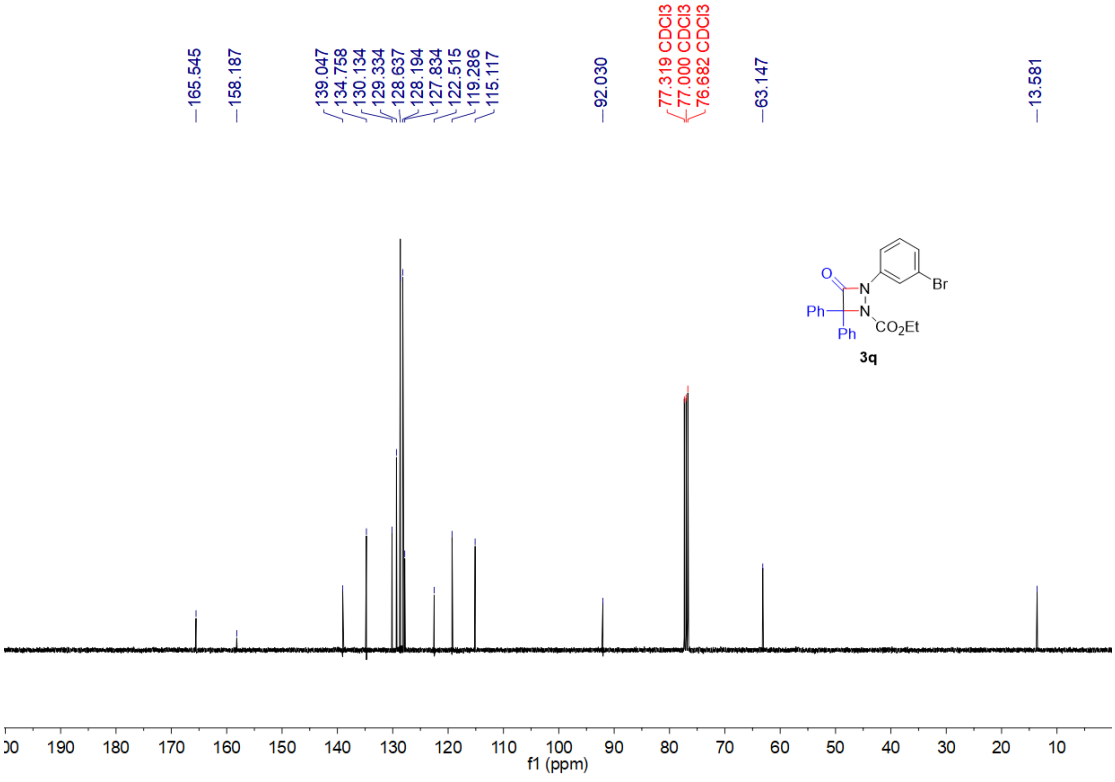
7.263
7.260 CDCl3
7.239
7.219
7.199
3.860
3.842
3.824
3.806
0.817
0.799
0.781
Br
O
N
Ph
N
CO2Et
Ph
3q
1.00
4.01
7.05
1.02
1.01
2.00
3.00
f1 (ppm)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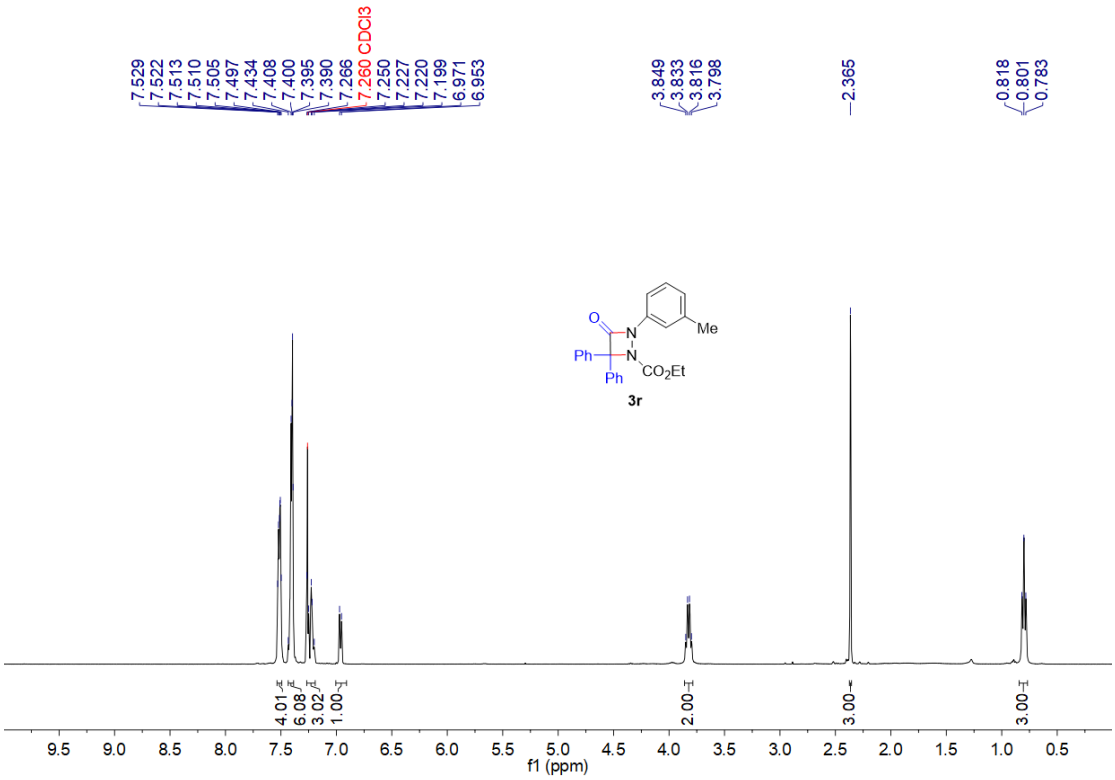
158.187
139.047
134.758
130.134
129.334
128.637
128.194
127.834
122.515
119.286
115.117
92.030
77.319 CDCl3
77.000 CDCl3
76.682 CDCl3
63.147
13.581
Br
O
N
Ph
N
CO2Et
Ph
3q
f1 (ppm)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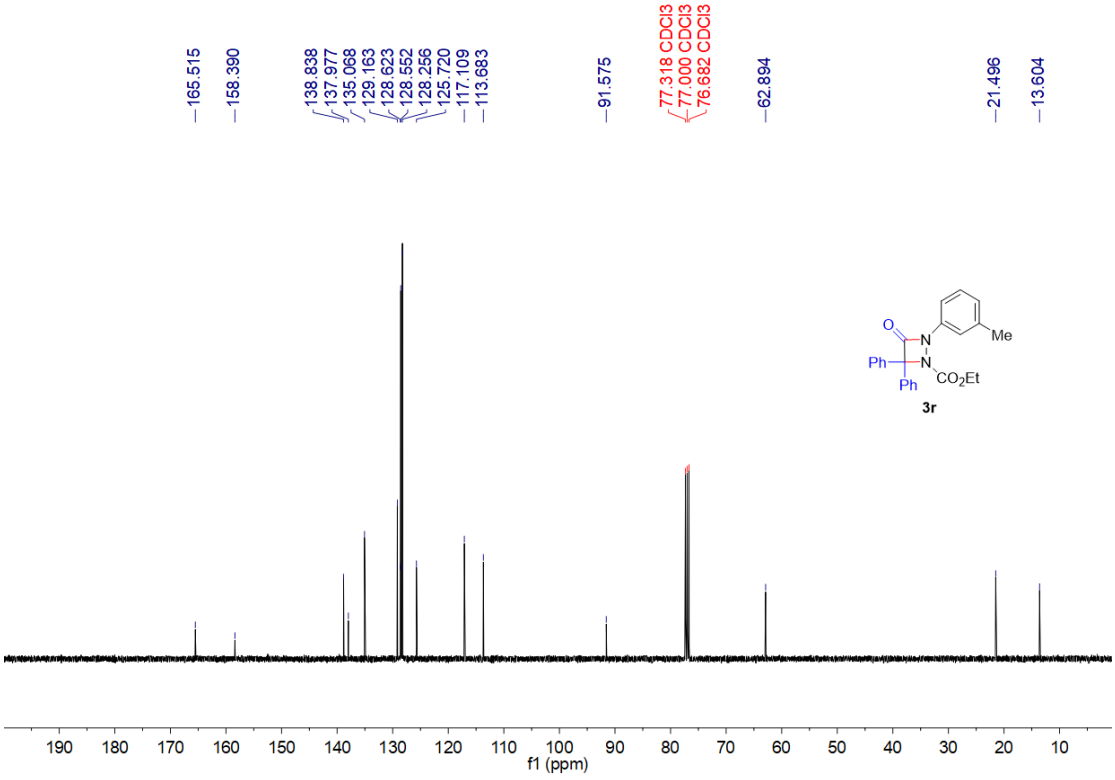
7.513
7.510
7.505
7.497
7.434
7.408
7.400
7.395
7.390
7.266
7.260 CDCl3
7.250
7.227
7.220
7.199
6.971
6.953
3.849
3.833
3.816
3.798
2.365
0.818
0.801
0.783
Me
Ph
Ph
CO2Et
3r
4.01
6.08
3.02
1.00
2.00
3.00
3.00
f1 (ppm)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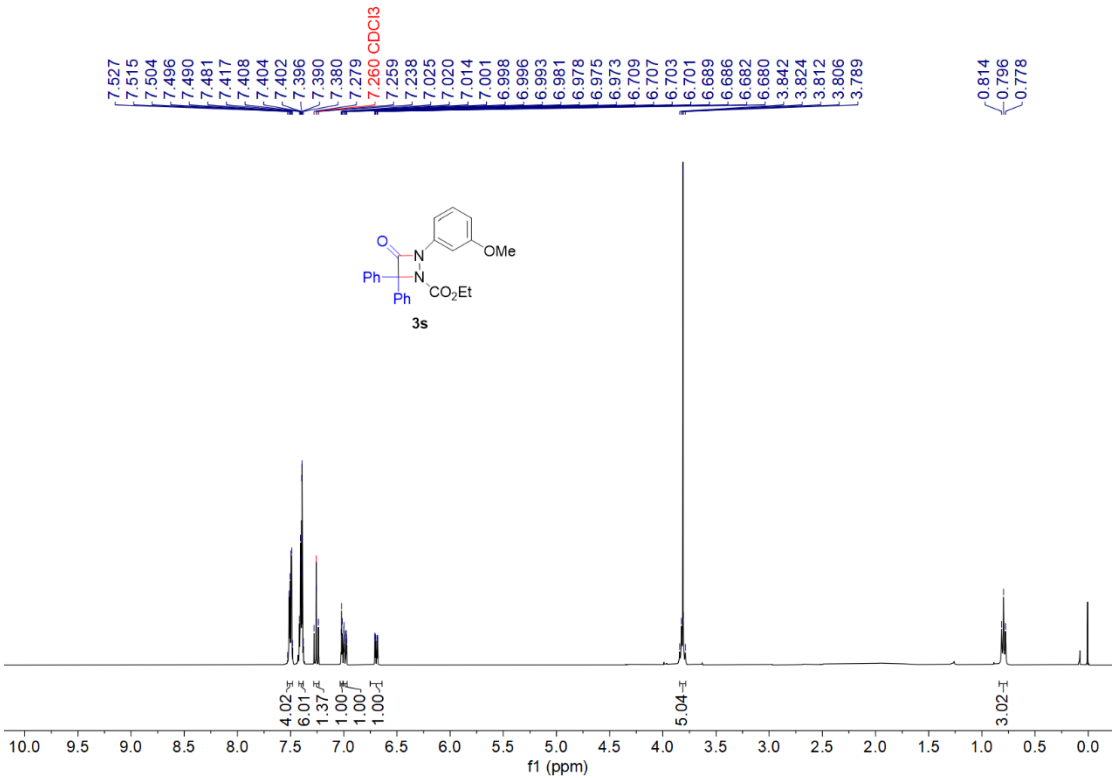
128.552
128.256
125.720
117.109
113.683
91.575
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
62.894
21.496
13.604
Me
Ph
Ph
CO2Et
3r
f1 (ppm)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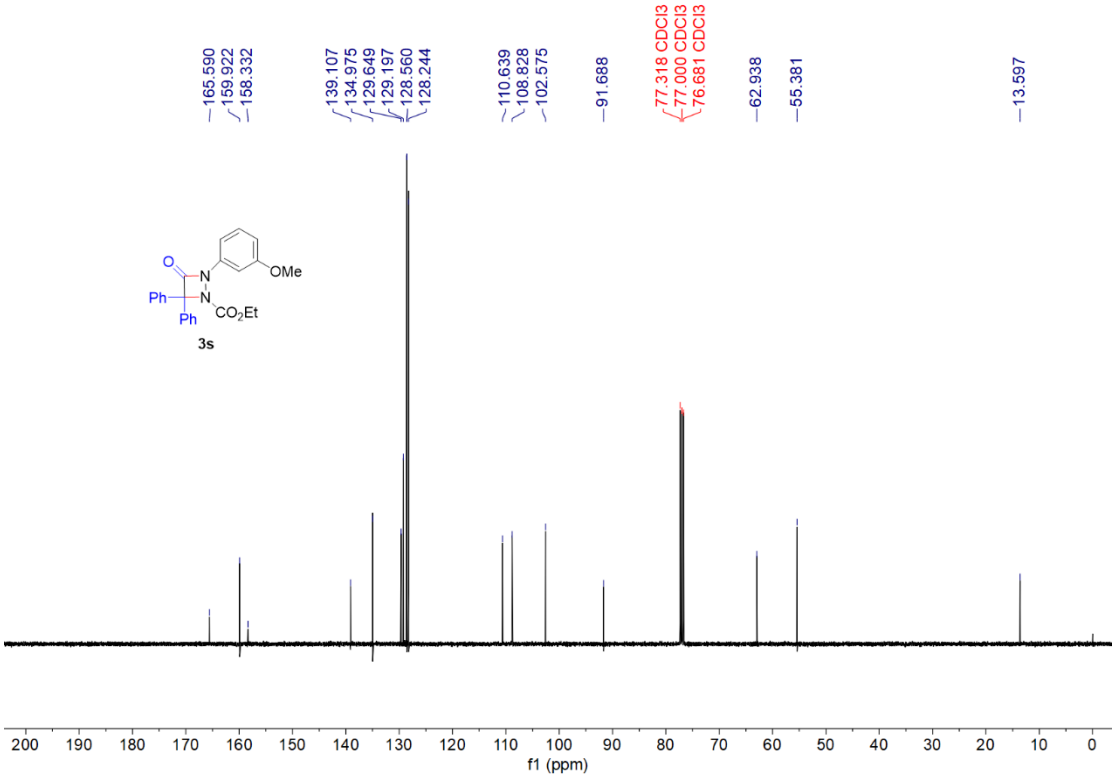
7.504
7.496
7.490
7.481
7.417
7.408
7.404
7.402
7.396
7.390
7.380
7.279
7.260 CDCl3
7.259
7.238
7.025
7.020
7.014
7.001
6.998
6.996
6.993
6.981
6.978
6.975
6.973
6.709
6.707
6.703
6.701
6.689
6.686
6.682
6.680
3.842
3.824
3.812
3.806
3.789
0.814
0.796
0.778
OMe
Ph
Ph
CO2Et
3s
4.02
6.01
1.37
1.00
1.00
1.00
5.04
3.02
f1 (ppm)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159.922
158.332
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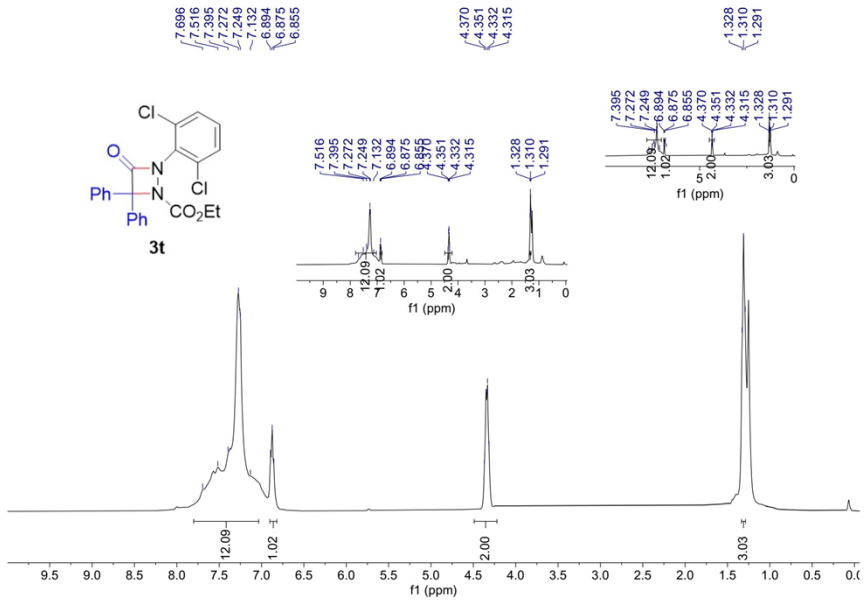
129.649
129.197
128.560
128.244
110.639
108.828
102.575
91.688
77.318 CDCl3
77.000 CDCl3
76.681 CDCl3
62.938
55.381
13.597
OMe
Ph
Ph
CO2Et
3s
f1 (ppm)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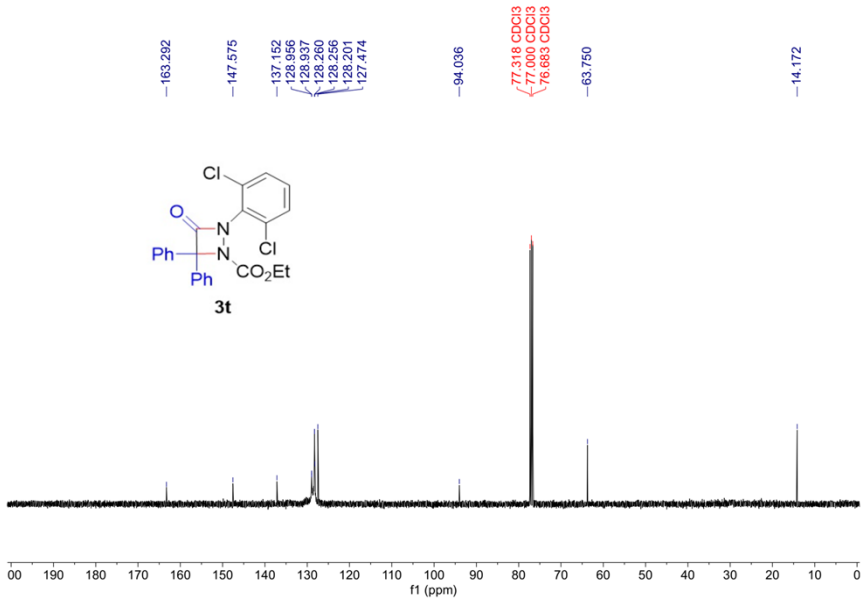
7.395
7.272
7.249
7.132
6.894
6.875
6.855
4.370
4.351
4.332
4.315
1.328
1.310
1.291
Cl
O
N
N
Cl
Ph
Ph
CO2Et
3t
12.09
1.02
2.00
3.03
f1 (ppm)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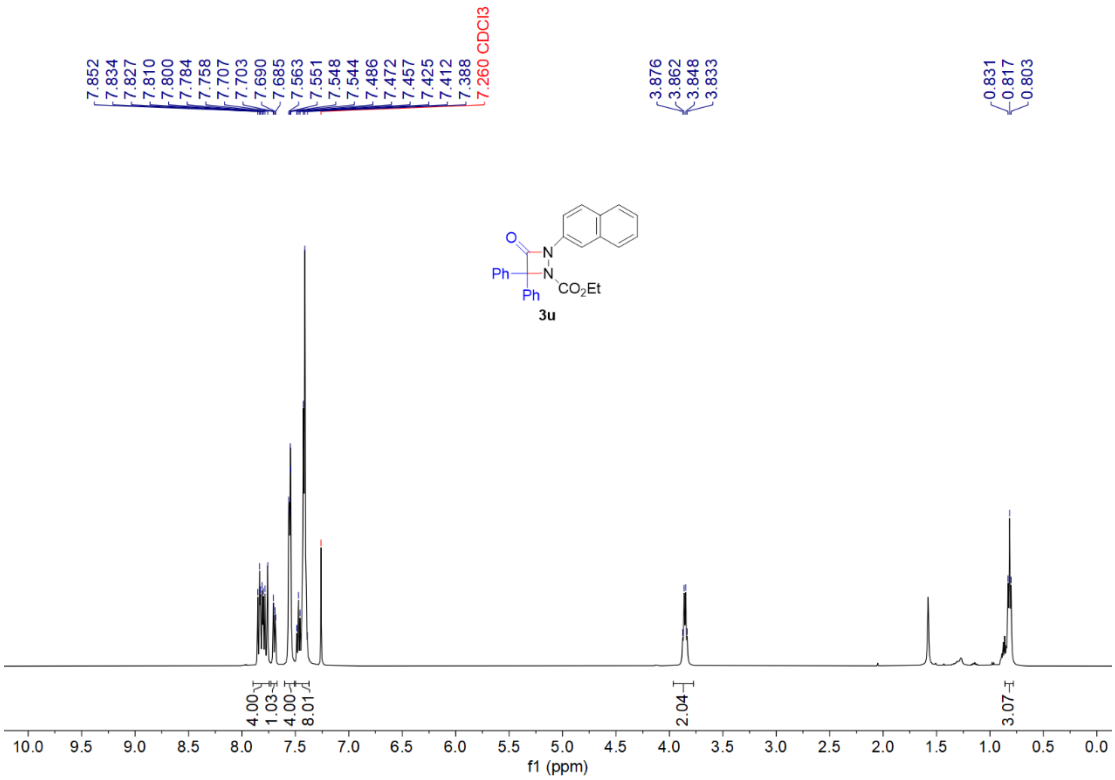
137.152
128.956
128.937
128.260
128.256
128.201
127.474
94.036
77.318 CDCl3
77.000 CDCl3
76.683 CDCl3
63.750
14.172
Cl
O
N
N
Cl
Ph
Ph
CO2Et
3t
f1 (ppm)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7.827
7.810
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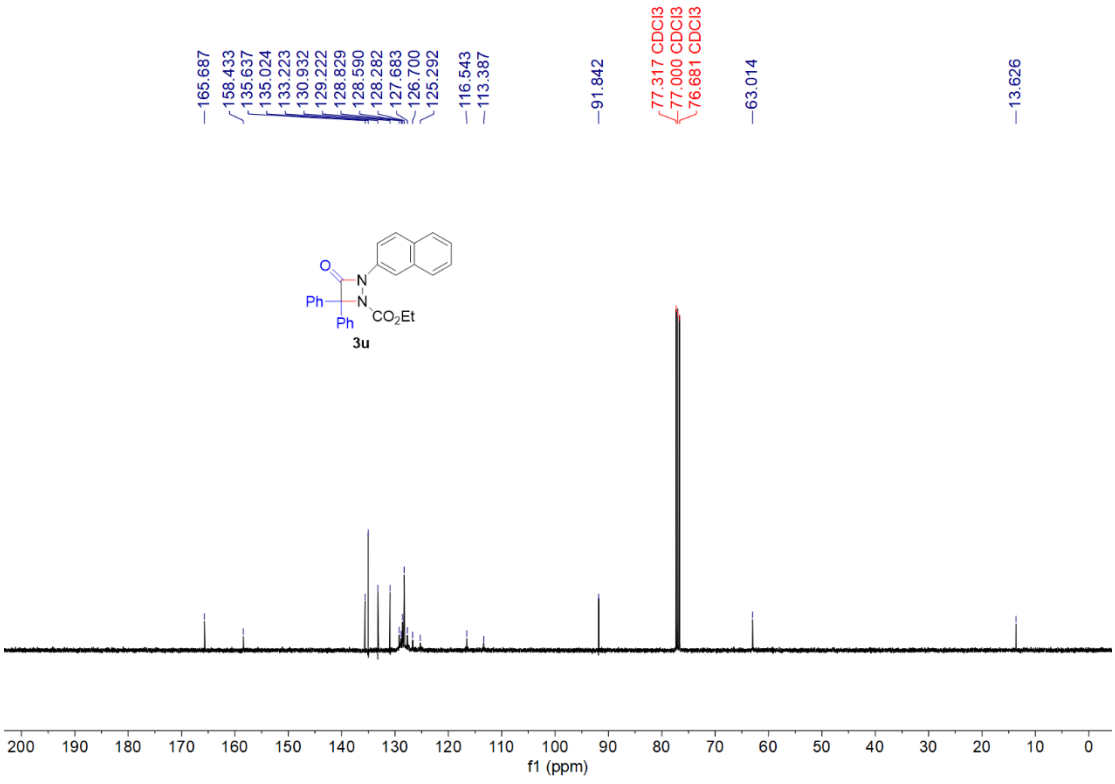
7.784
7.758
7.707
7.703
7.690
7.685
7.563
7.551
7.548
7.544
7.486
7.472
7.457
7.425
7.412
7.388
7.260 CDCl3
3.876
3.862
3.848
3.833
0.831
0.817
0.803
O
N
Ph
N
CO2Et
Ph
3u
4.00
1.03
4.00
8.01
2.04
3.07
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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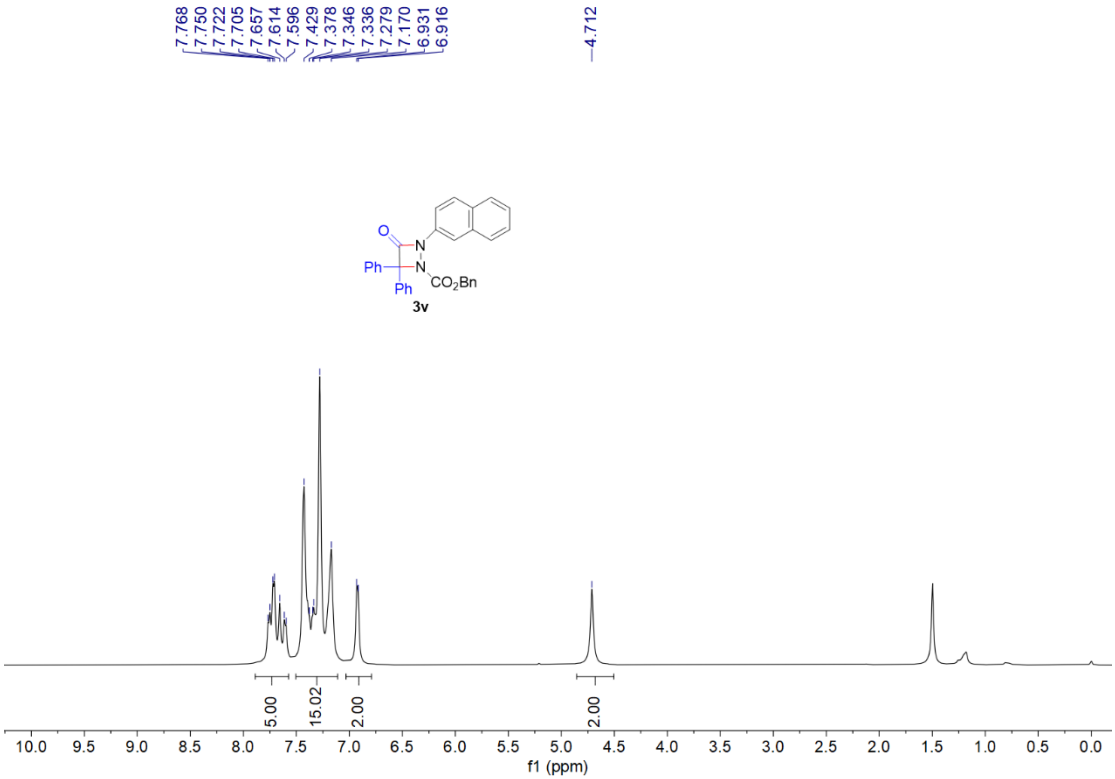
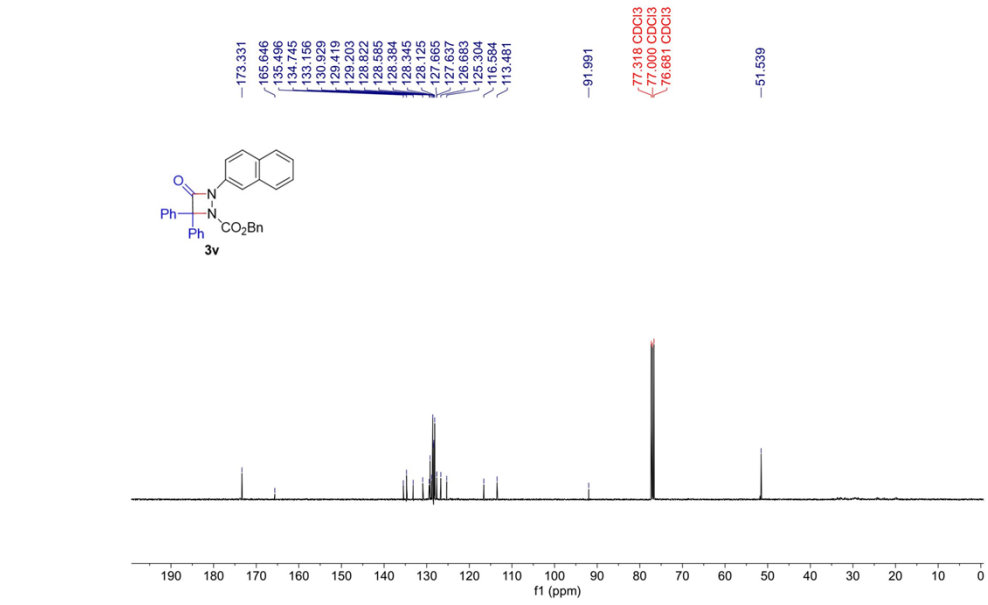
158.433
135.637
135.024
133.223
130.932
129.222
128.829
128.590
128.282
127.683
126.700
125.292
116.543
113.387
91.842
77.317 CDCl3
77.000 CDCl3
76.681 CDCl3
63.014
13.626
O
N
Ph
N
CO2Et
Ph
3u
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.768
7.750
7.722
7.705
7.657
7.614
7.596
7.429
7.378
7.346
7.336
7.279
7.170
6.931
6.916
4.712
3v
5.00
15.02
2.00
2.00
f1 (ppm)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135.496
134.745
133.156
130.929
129.419
129.203
128.822
128.585
128.384
128.345
128.125
127.665
127.637
126.683
125.304
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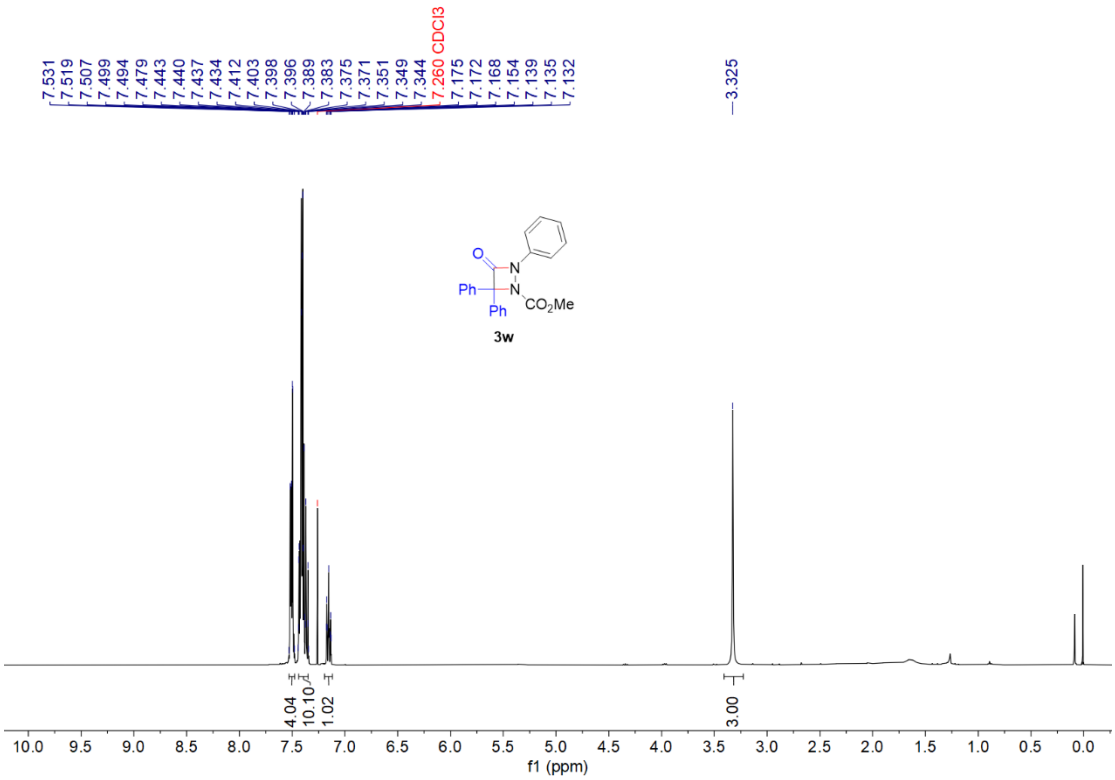
113.481
91.991
77.318 CDCl3
77.000 CDCl3
76.681 CDCl3
51.539
3v
f1 (ppm)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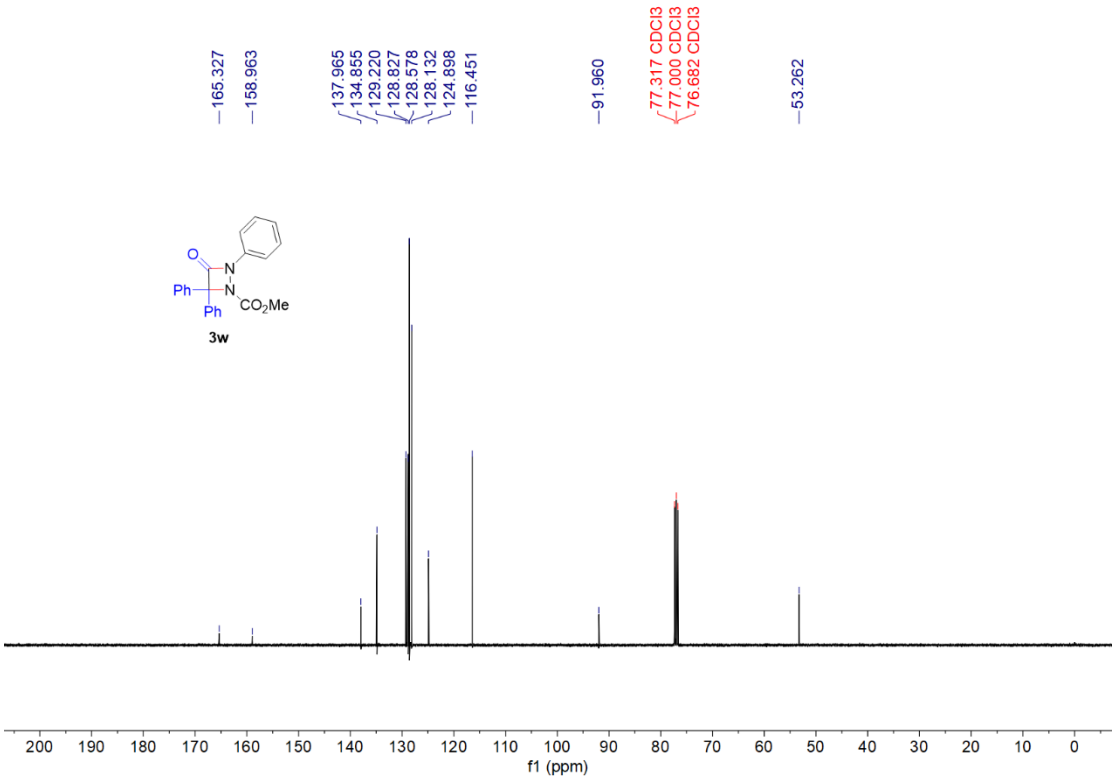
7.519
7.507
7.499
7.494
7.479
7.443
7.440
7.437
7.434
7.412
7.403
7.398
7.396
7.389
7.383
7.375
7.371
7.351
7.349
7.344
7.260 CDCl3
7.175
7.172
7.168
7.154
7.139
7.135
7.132
3.325
Ph
Ph
CO2Me
3w
4.04
10.10
1.02
3.00
f1 (ppm)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158.963
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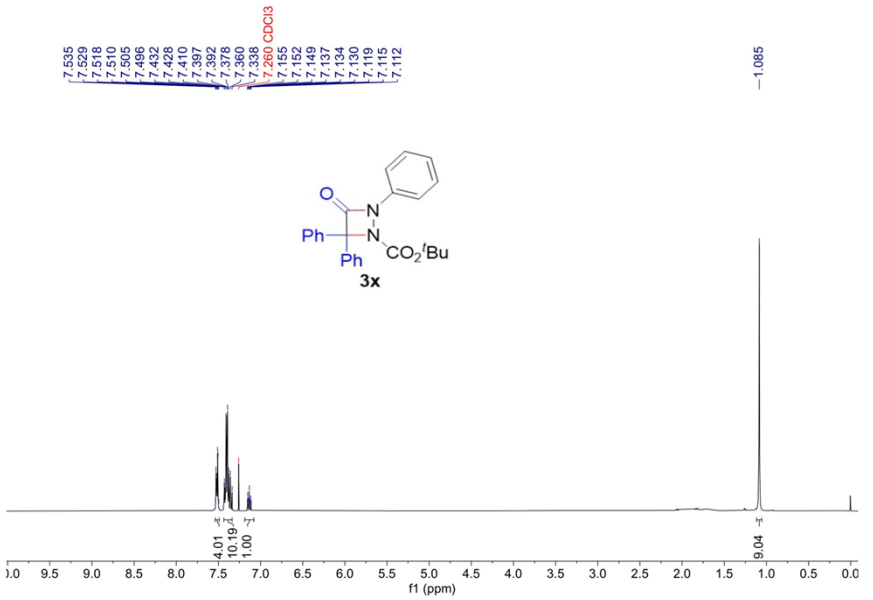
129.220
128.827
128.578
128.132
124.898
116.451
91.960
77.317 CDCl3
77.000 CDCl3
76.682 CDCl3
53.262
Ph
Ph
CO2Me
3w
f1 (ppm)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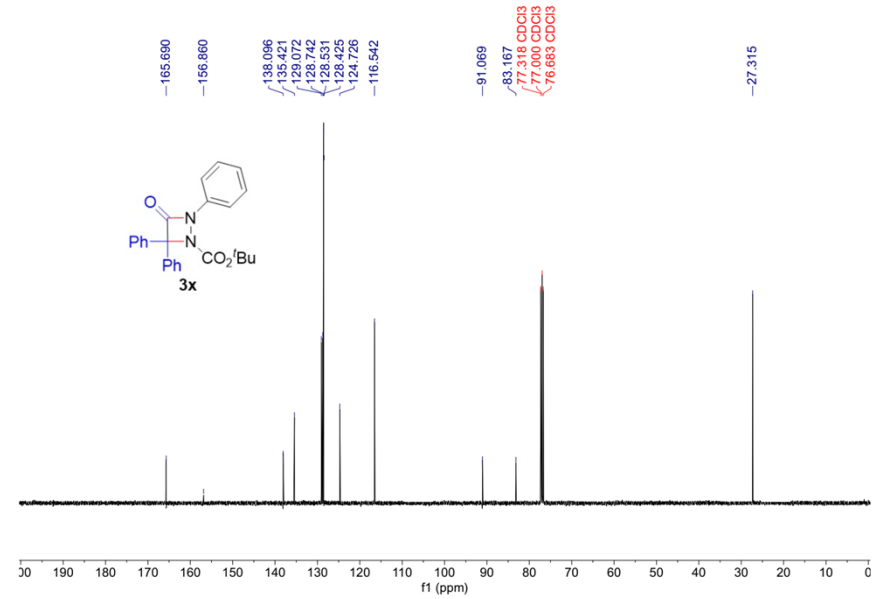
7.510
7.505
7.496
7.432
7.428
7.410
7.397
7.392
7.378
7.360
7.338
7.260 CDCl3
7.155
7.152
7.149
7.137
7.134
7.130
7.119
7.115
7.112
1.085
Ph
Ph
CO2tBu
3x
4.01
10.19
1.00
9.04
f1 (ppm)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135.421
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128.742
128.531
126.425
124.726
116.542
91.069
83.167
77.318 CDCl3
77.000 CDCl3
76.683 CDCl3
27.315
Ph
Ph
CO2tBu
3x
f1 (ppm)


7.683
7.646
7.624
7.580
7.487
7.475
7.472
7.468
7.463
7.455
7.450
7.447
7.436
7.405
7.387
7.368
7.365
7.347
7.342
7.331
7.321
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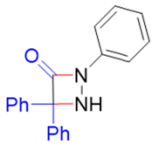
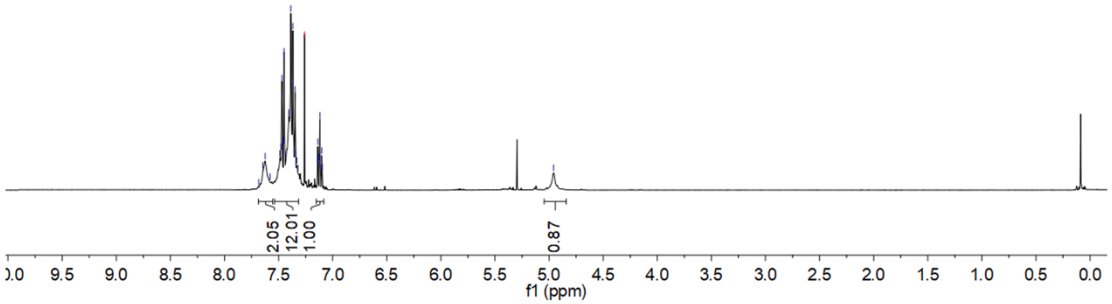
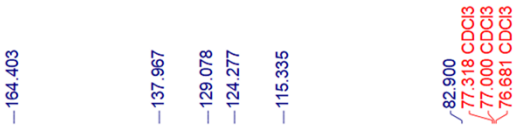
7.122
7.119
7.115
7.103
7.100
7.097
4.959


O
N
Ph
NH
Ph

3x-DG


2.05
12.01
1.00
0.87
f1 (ppm)


164.403
137.967
129.078
124.277
115.335
82.900
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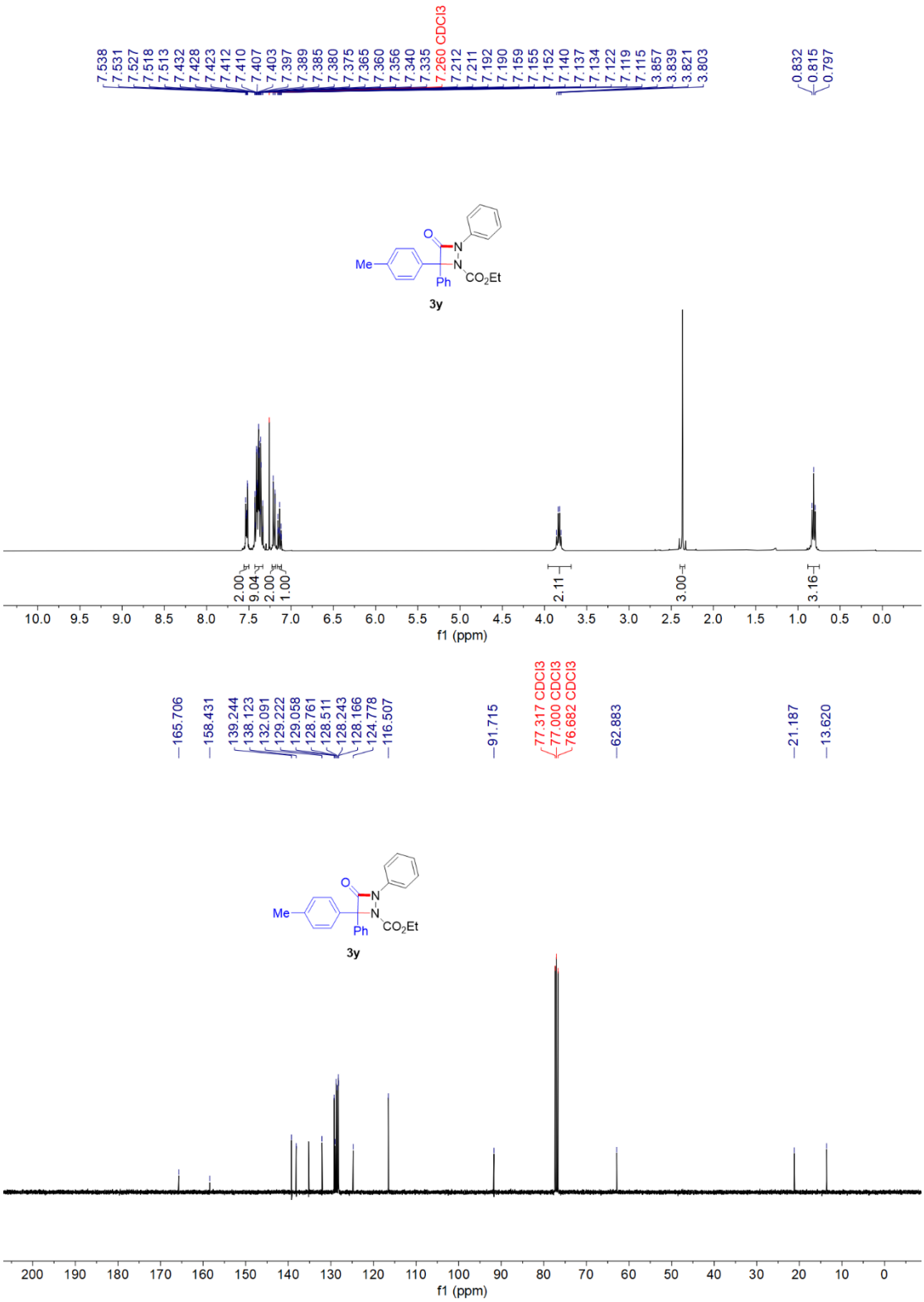
77.000 CDCl3
76.681 CDCl3


O
N
Ph
NH
Ph

3x-DG

f1 (ppm)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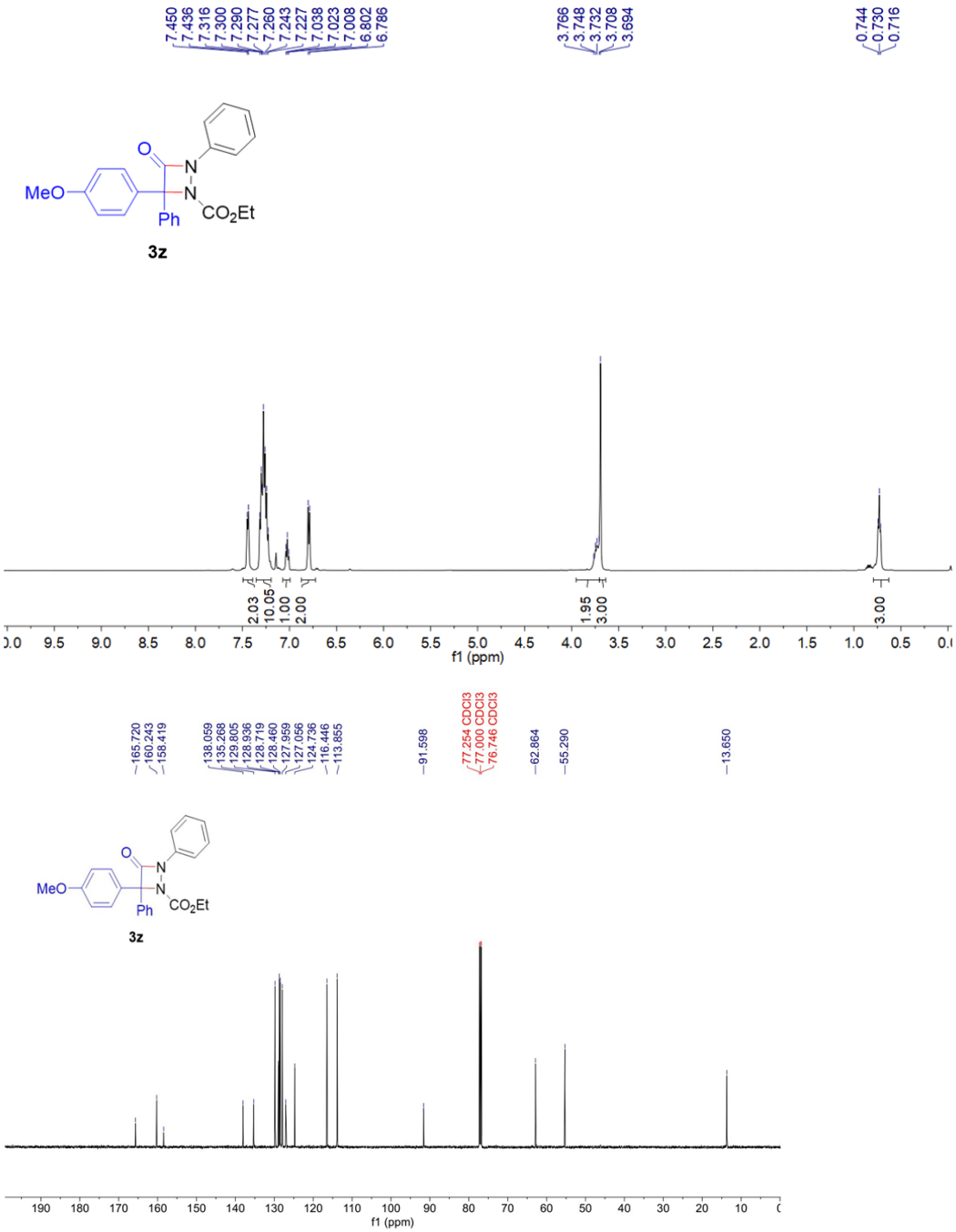
7.531
7.527
7.518
7.513
7.432
7.428
7.423
7.412
7.410
7.407
7.403
7.397
7.389
7.385
7.380
7.375
7.365
7.360
7.356
7.340
7.335
7.260 CDCl3
7.212
7.211
7.192
7.190
7.159
7.155
7.152
7.140
7.137
7.134
7.122
7.119
7.115
3.857
3.839
3.821
3.803
0.832
0.815
0.797
Me
Ph
CO2Et
3y
2.00
9.04
2.00
1.00
2.11
3.00
3.16
f1 (ppm)
165.706
158.431
139.244
138.123
132.091
129.222
129.058
128.761
128.511
128.243
128.166
124.778
116.507
91.715
77.317 CDCl3
77.000 CDCl3
76.682 CDCl3
62.883
21.187
13.620
Me
Ph
CO2Et
3y
f1 (ppm)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7.436
7.316
7.300
7.290
7.277
7.260
7.243
7.227
7.038
7.023
7.008
6.802
6.786
3.766
3.748
3.732
3.708
3.694
0.744
0.730
0.716
MeO
Ph
CO2Et
3z
2.03
10.05
1.00
2.00
1.95
3.00
3.00
f1 (ppm)
165.720
160.243
158.419
138.059
135.268
129.805
128.936
128.719
128.460
127.959
127.056
124.736
116.446
113.855
91.598
77.254 CDCl3
77.000 CDCl3
76.746 CDCl3
62.864
55.290
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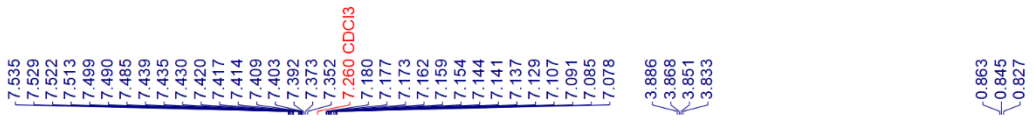
MeO
Ph
CO2Et
3z
f1 (ppm)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7.529
7.522
7.513
7.499
7.490
7.485
7.439
7.435
7.430
7.420
7.417
7.414
7.409
7.403
7.392
7.373
7.352
7.260 CDCl3
7.180
7.177
7.173
7.162
7.159
7.154
7.144
7.141
7.137
7.129
7.107
7.091
7.085
7.078
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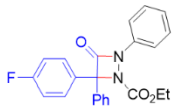
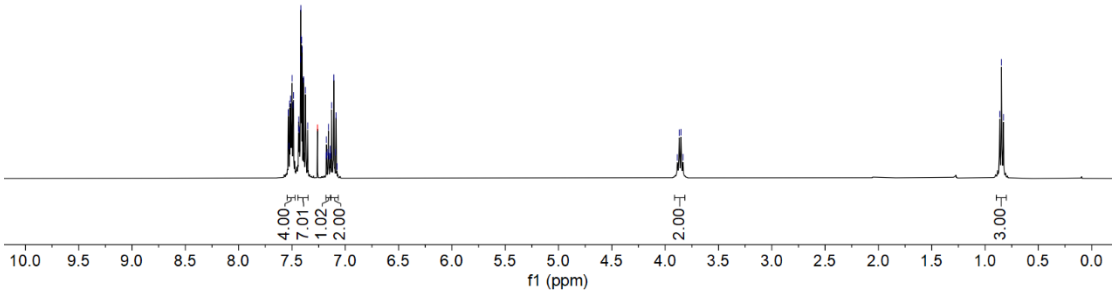
3.868
3.851
3.833
0.863
0.845
0.827


O
N
N
F
Ph
CO2Et

3aa


4.00
7.01
1.02
2.00
2.00
3.00
10.0 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)


165.278
164.340
161.857
158.186
137.938
134.866
130.955
130.922
130.268
130.184
129.342
128.805
128.655
128.128
124.961
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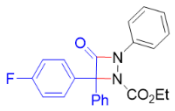
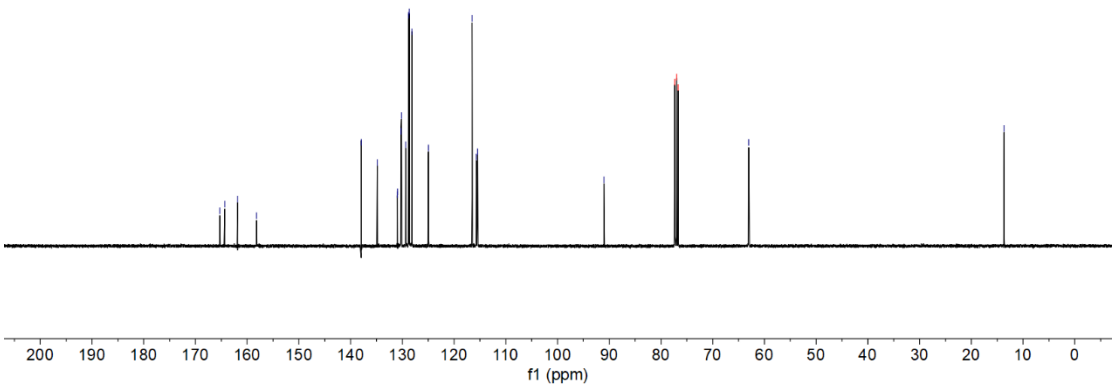
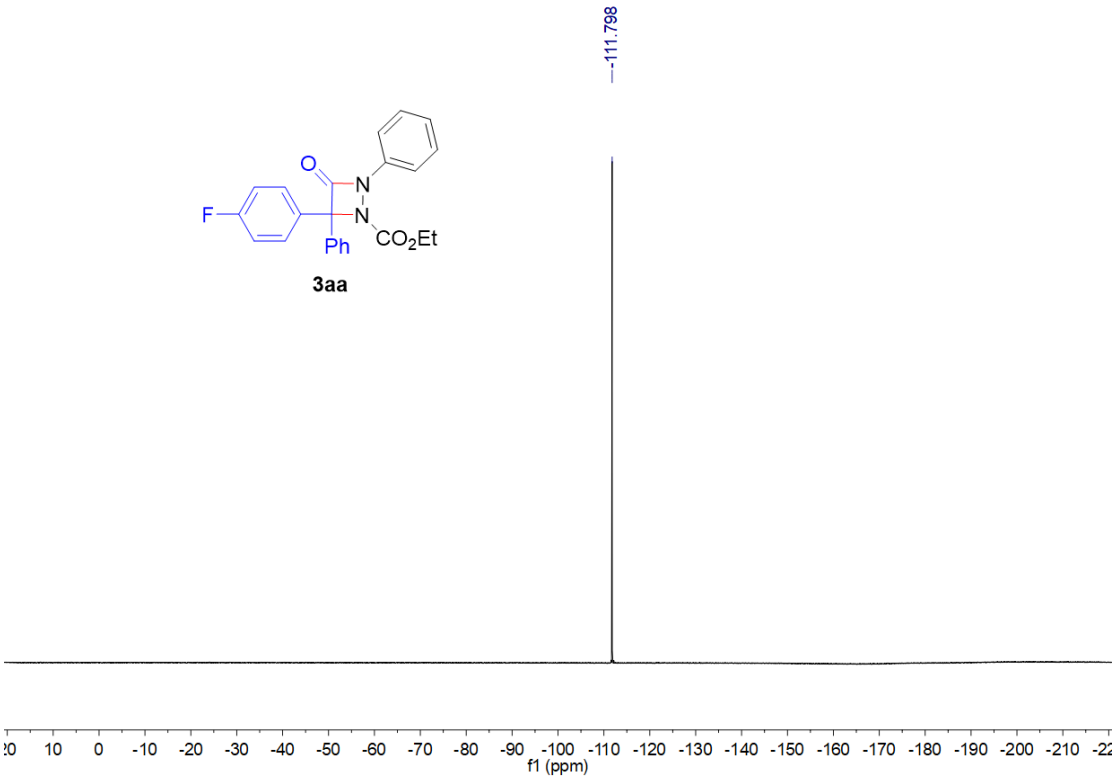
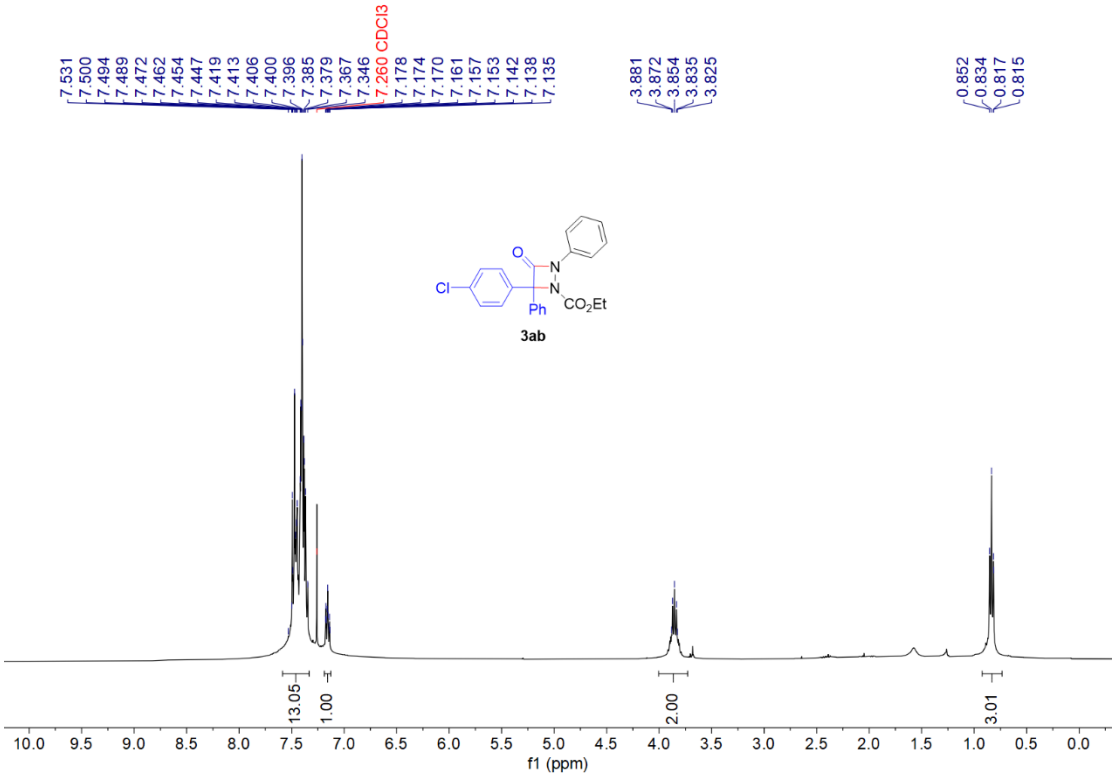
115.696
115.479
91.021
77.317 CDCl3
77.000 CDCl3
76.682 CDCl3
63.038
13.649

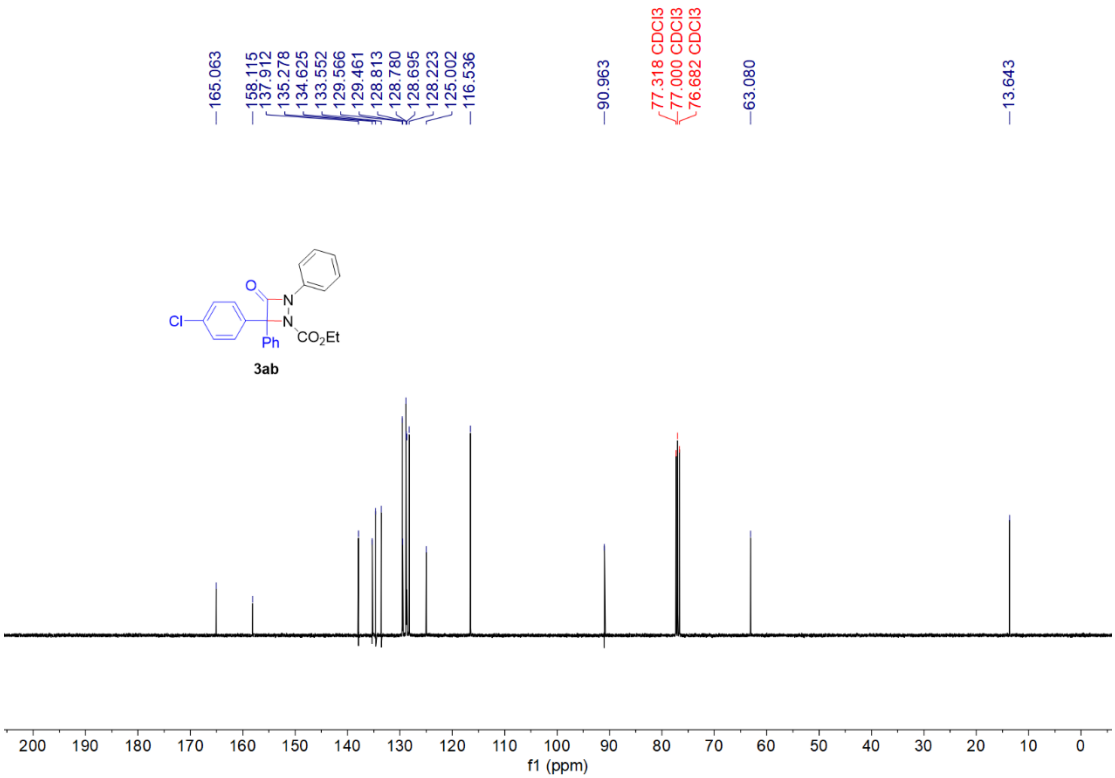

O
N
N
F
Ph
CO2Et

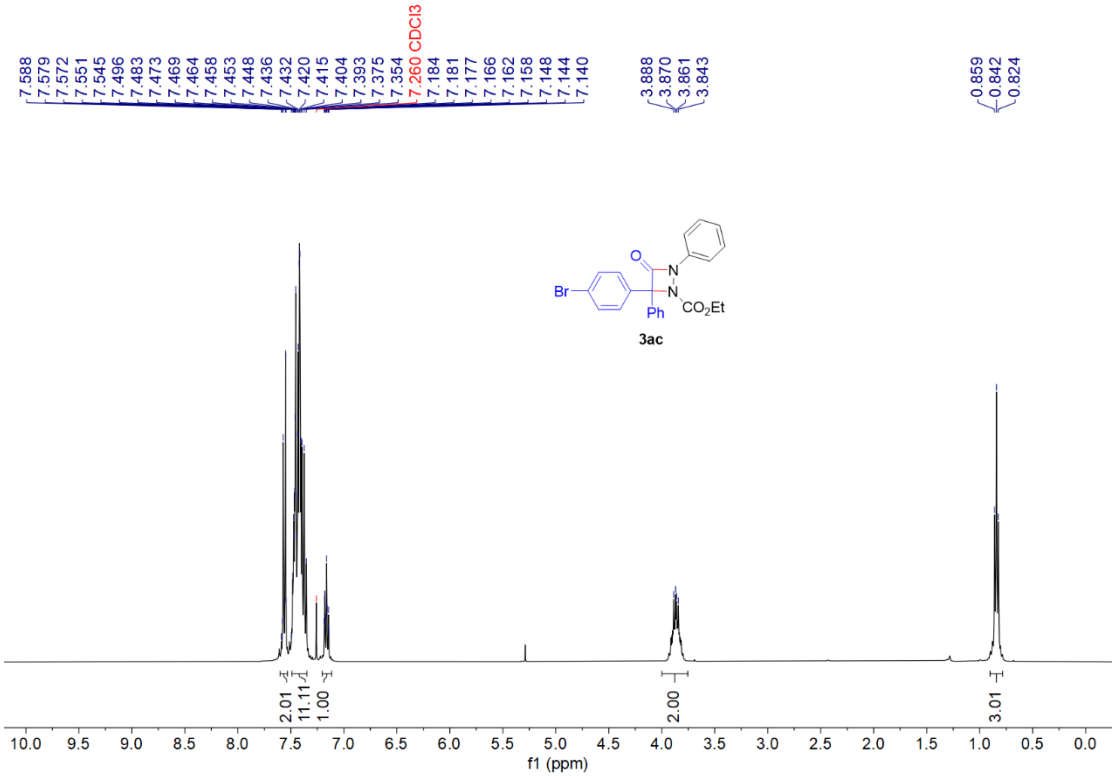
3aa


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-111.798
O
N
N
F
Ph
CO2Et
3aa
f1 (ppm)


7.531
7.500
7.494
7.489
7.472
7.462
7.454
7.447
7.419
7.413
7.406
7.400
7.396
7.385
7.379
7.367
7.346
7.260 CDCl3
7.178
7.174
7.170
7.161
7.157
7.153
7.142
7.138
7.135
3.881
3.872
3.854
3.835
3.825
0.852
0.834
0.817
0.815
O
N
N
Cl
Ph
CO2Et
3ab
13.05
1.00
2.00
3.01
f1 (ppm)


165.063
158.115
137.912
135.278
134.625
133.552
129.566
129.461
128.813
128.780
128.695
128.223
125.002
116.536
90.963
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
63.080
13.643
O
N
N
Cl
Ph
CO2Et
3ab
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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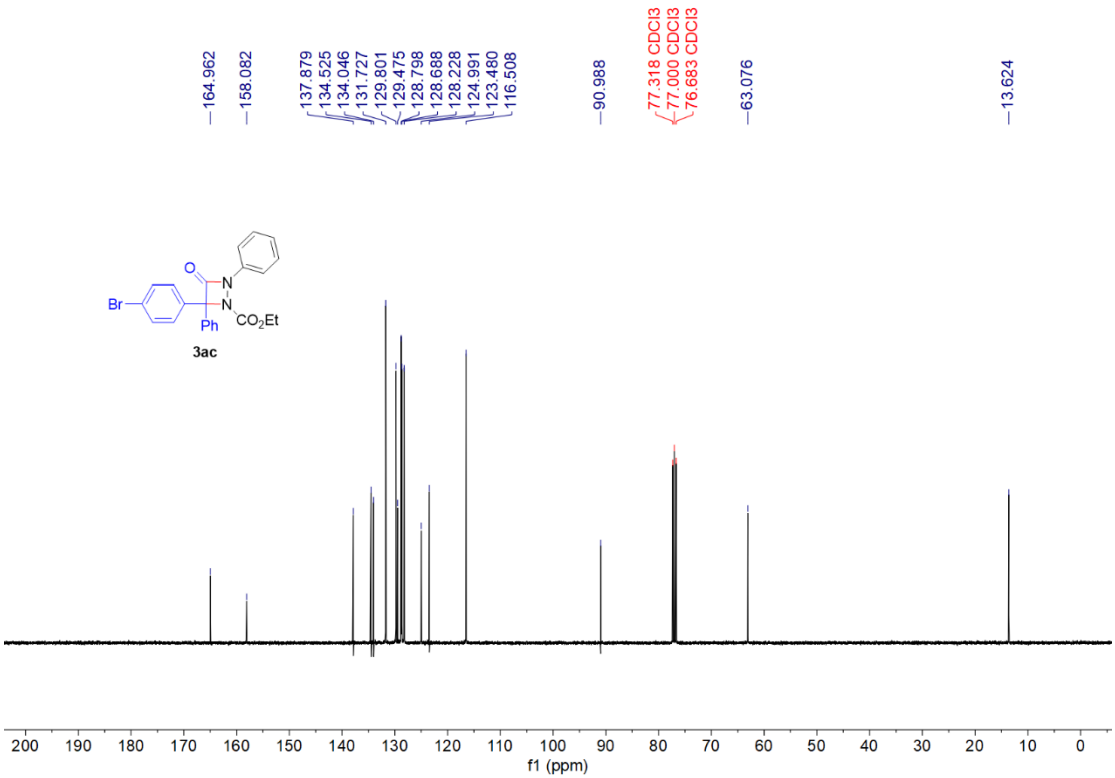
7.448
7.436
7.432
7.420
7.415
7.404
7.393
7.375
7.354
7.260 CDCl3
7.184
7.181
7.177
7.166
7.162
7.158
7.148
7.144
7.140
3.888
3.870
3.861
3.843
0.859
0.842
0.824
O
N
N
Br
Ph
CO2Et
3ac
2.01
11.11
1.00
2.00
3.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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123.480
116.508
90.988
77.318 CDCl3
77.000 CDCl3
76.683 CDCl3
63.076
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Br
O
N
N
Ph
CO2Et
3ac
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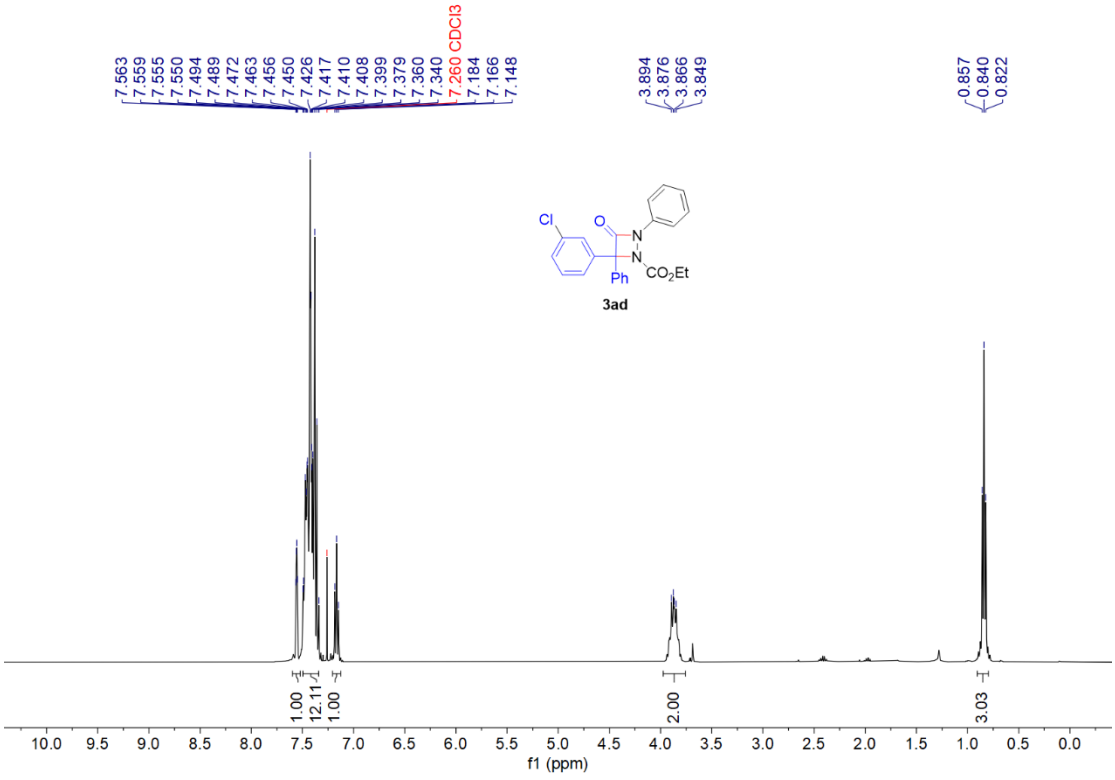
f1 (ppm)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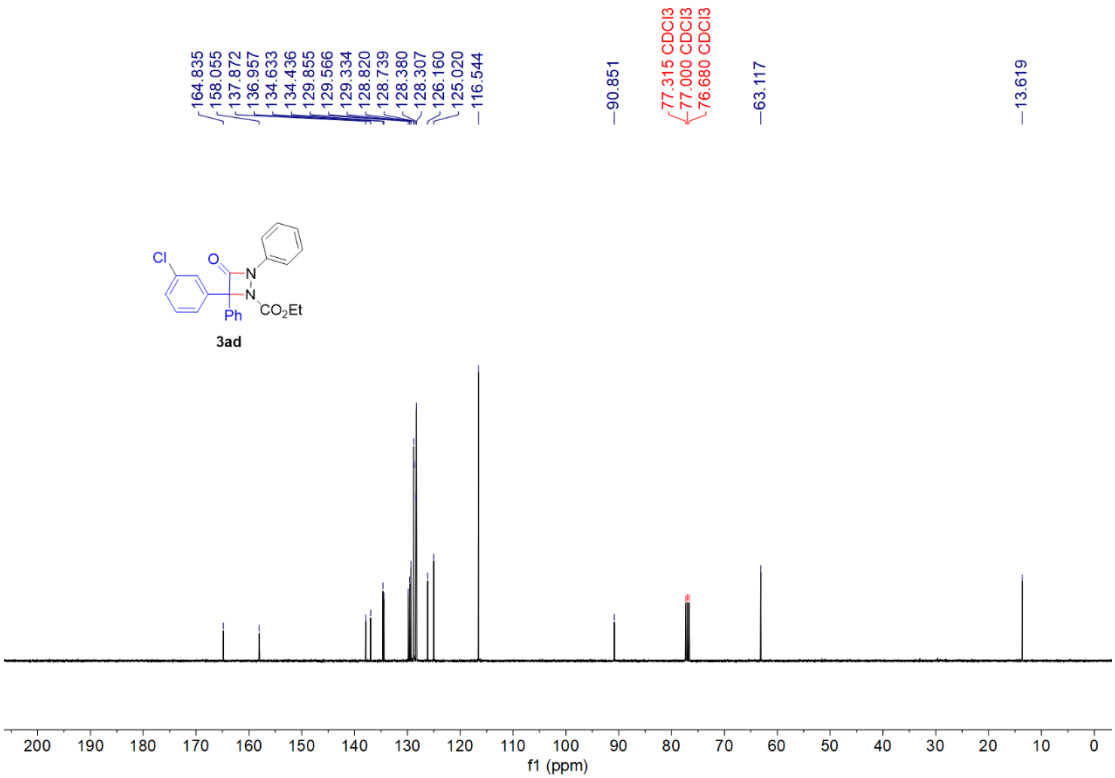
7.408
7.399
7.379
7.360
7.340
7.260 CDCl3
7.184
7.166
7.148
3.894
3.876
3.866
3.849
0.857
0.840
0.822
Cl
O
N
N
Ph
CO2Et
3ad
1.00
12.11
1.00
2.00
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.566
129.334
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128.307
126.160
125.020
116.544
90.851
77.315 CDCl3
77.000 CDCl3
76.680 CDCl3
63.117
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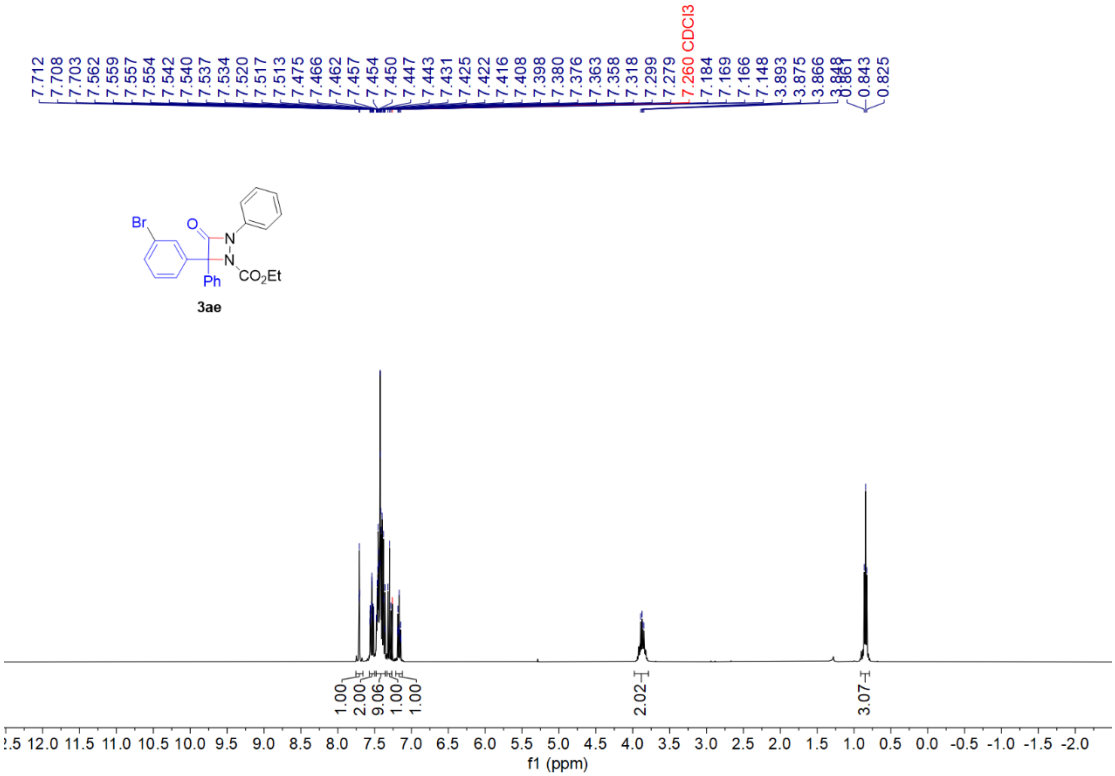
Cl
O
N
N
Ph
CO2Et
3ad
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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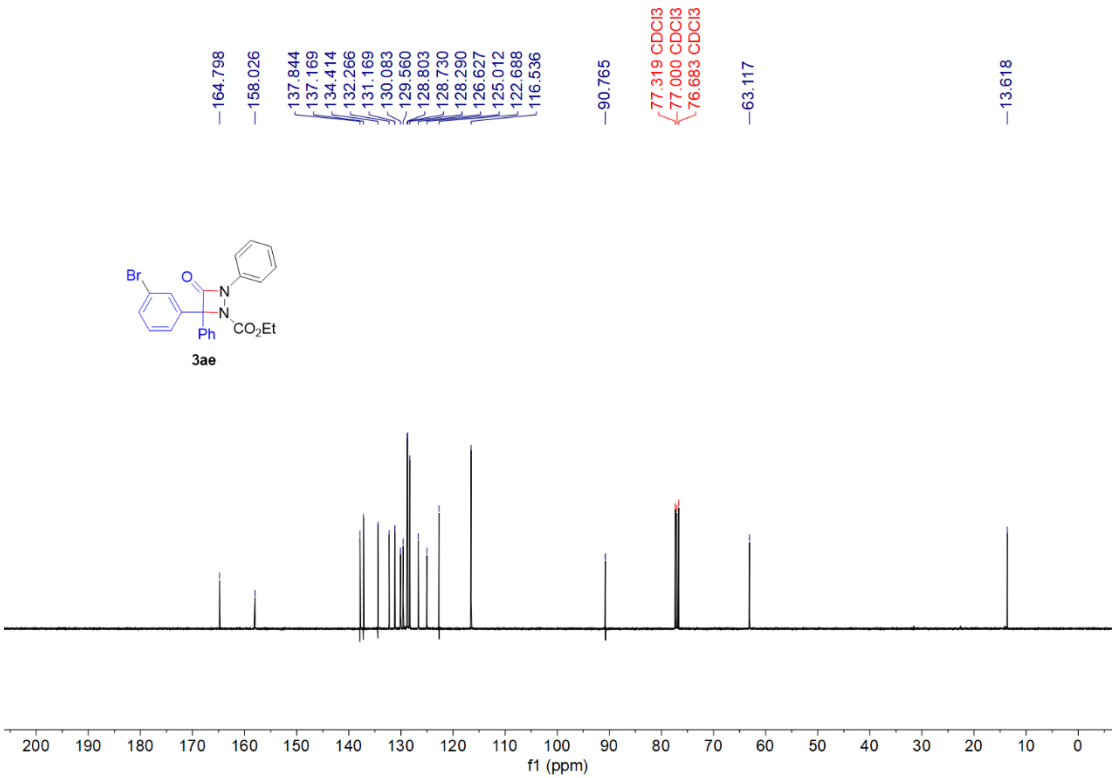
7.513
7.475
7.466
7.462
7.457
7.454
7.450
7.447
7.443
7.431
7.425
7.422
7.416
7.408
7.398
7.380
7.376
7.363
7.358
7.318
7.299
7.279
7.260 CDCl3
7.184
7.169
7.166
7.148
3.893
3.875
3.866
0.843
0.825
Br
O
N
N
Ph
CO2Et
3ae
1.00
2.00
9.06
1.00
1.00
2.02
3.07
f1 (ppm)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137.844
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131.169
130.083
129.560
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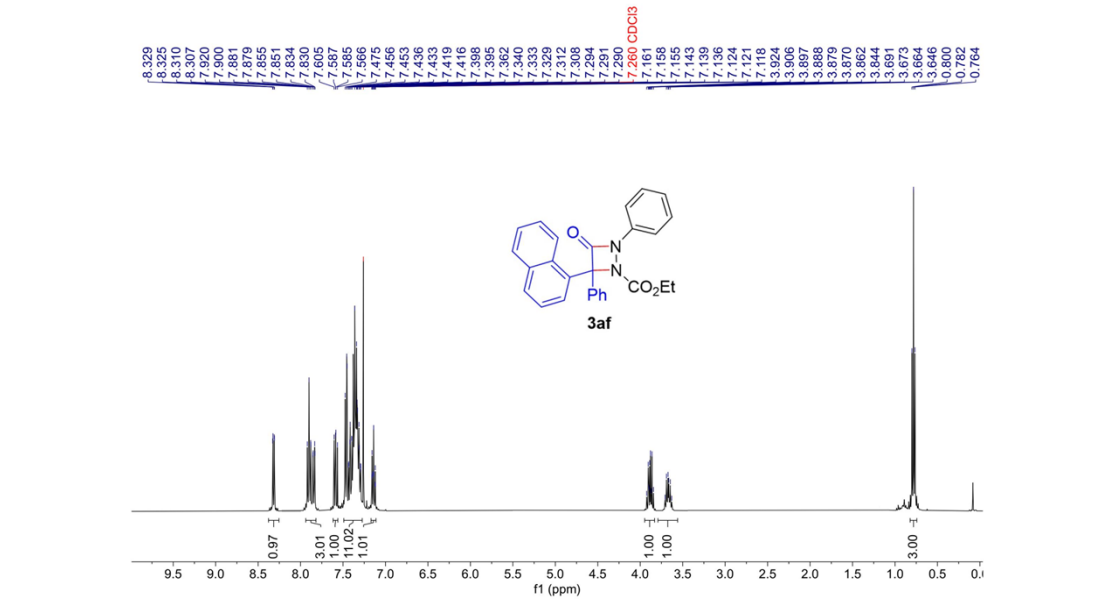
128.730
128.290
126.627
125.012
122.688
116.536
90.765
77.319 CDCl3
77.000 CDCl3
76.683 CDCl3
63.117
13.618
Br
O
N
N
Ph
CO2Et
3ae
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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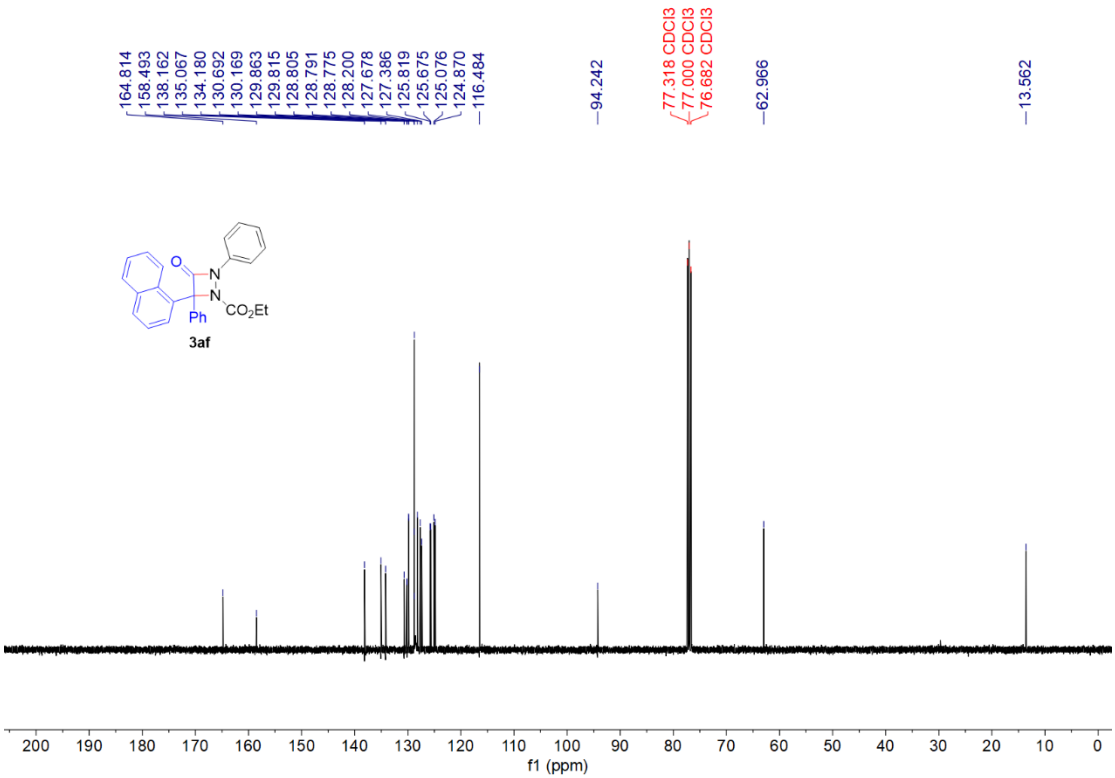
7.881
7.879
7.855
7.851
7.834
7.830
7.605
7.587
7.585
7.566
7.475
7.456
7.453
7.436
7.433
7.419
7.416
7.398
7.395
7.362
7.340
7.333
7.329
7.312
7.308
7.294
7.291
7.280
7.260 CDCl3
7.161
7.158
7.155
7.143
7.139
7.136
7.124
7.121
7.118
3.924
3.906
3.897
3.888
3.879
3.870
3.862
3.844
3.691
3.673
3.664
3.646
0.800
0.782
0.764
O
N
N
Ph
CO2Et
3af
0.97
3.01
1.00
11.02
1.01
1.00
1.00
3.00
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
f1 (ppm)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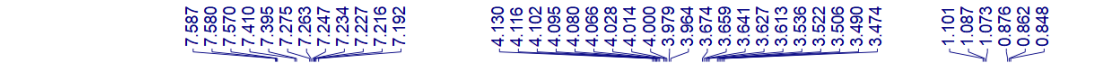
128.805
128.791
128.775
128.200
127.678
127.386
125.819
125.675
125.076
124.870
116.484
94.242
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
62.966
13.562
O
N
N
CO2Et
Ph
3af
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.247
7.234
7.227
7.216
7.192
4.130
4.116
4.102
4.095
4.080
4.066
4.028
4.014
4.000
3.979
3.964
3.674
3.659
3.641
3.627
3.613
3.536
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3.506
3.490
3.474
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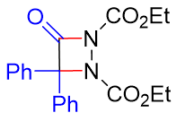
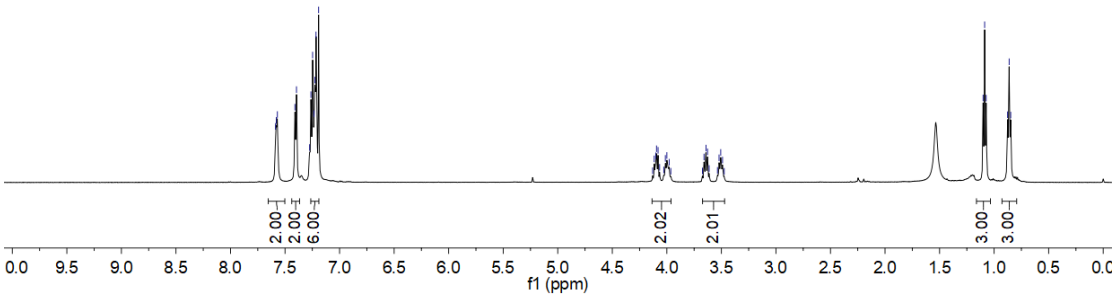
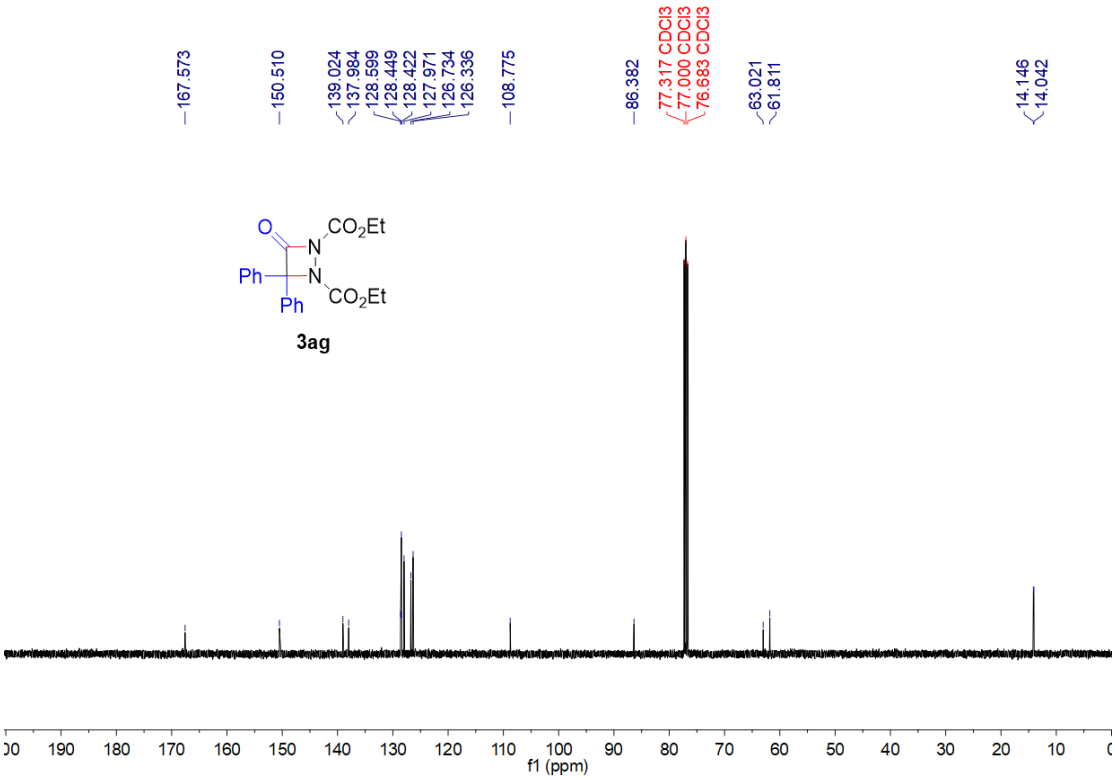
1.087
1.073
0.876
0.862
0.848


O
CO2Et
N
N
Ph
Ph
CO2Et

3ag


2.00
2.00
6.00
2.02
2.01
3.00
3.00
0.0 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)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127.971
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126.336
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86.382
77.317 CDCl3
77.000 CDCl3
76.683 CDCl3
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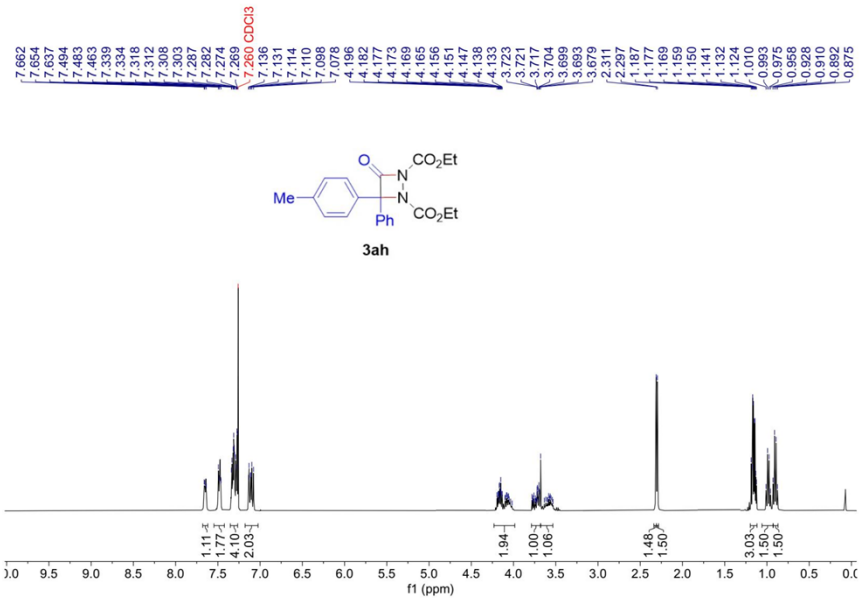
14.146
14.042
O
N
CO2Et
Ph
N
Ph
CO2Et
3ag
f1 (ppm)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7.312
7.308
7.303
7.287
7.282
7.274
7.269
7.260 CDCl3
7.136
7.131
7.114
7.110
7.098
7.078
4.196
4.182
4.177
4.173
4.169
4.165
4.156
4.151
4.147
4.138
4.133
3.723
3.721
3.717
3.704
3.699
3.693
3.679
2.311
2.297
1.187
1.177
1.169
1.159
1.150
1.141
1.132
1.124
1.010
0.993
0.975
0.958
0.928
0.910
0.892
0.875
O
N
CO2Et
Me
N
Ph
CO2Et
3ah
1.11
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1.94
1.00
1.06
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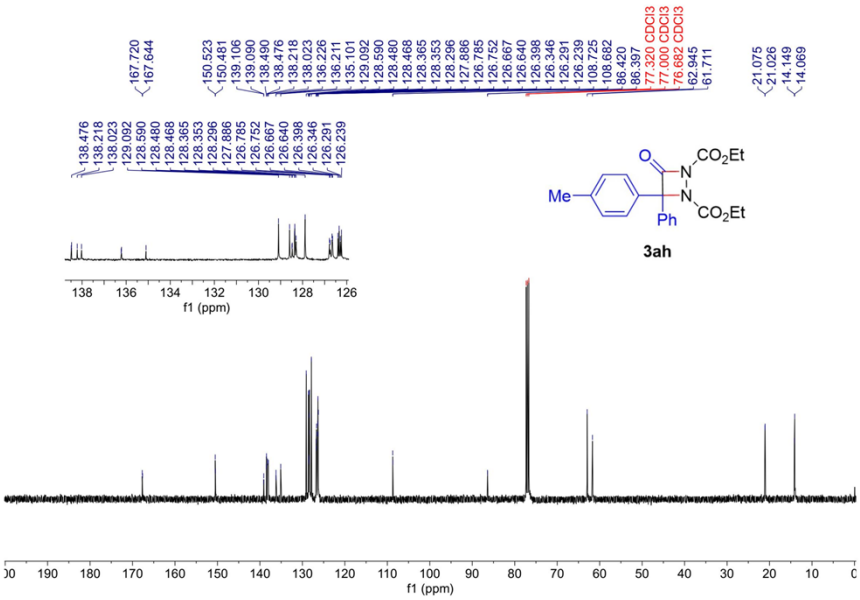
3.03
1.50
1.50
f1 (ppm)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129.092
128.590
128.480
128.468
128.365
128.353
128.296
127.886
126.785
126.752
126.667
126.640
126.398
126.346
126.291
126.239
108.725
108.682
86.420
86.397
77.320 CDCl3
77.000 CDCl3
76.682 CDCl3
62.945
61.711
21.075
21.026
14.149
14.069
138.476
138.218
138.023
129.092
128.590
128.480
128.468
128.365
128.353
128.296
127.886
126.785
126.752
126.667
126.640
126.398
126.346
126.291
126.239
138
136
134
132
130
128
126
f1 (ppm)
O
N
CO2Et
N
CO2Et
Me
Ph
3ah
0
190
180
170
160
150
140
130
120
110
100
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50
40
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20
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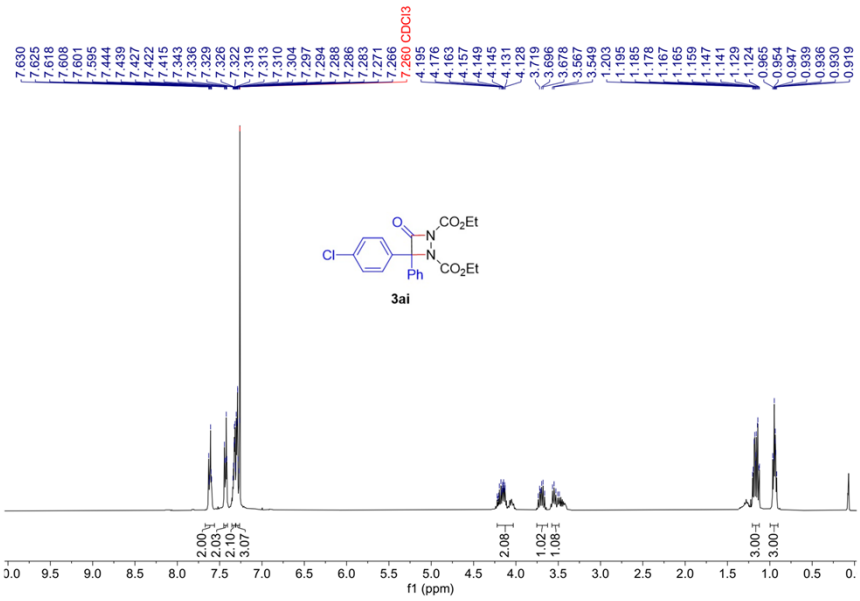
f1 (ppm)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7.326
7.322
7.319
7.313
7.310
7.304
7.297
7.294
7.288
7.286
7.283
7.271
7.266
7.260 CDCl3
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4.176
4.163
4.157
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4.145
4.131
4.128
3.719
3.696
3.678
3.567
3.549
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1.195
1.185
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1.167
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1.159
1.147
1.141
1.129
1.124
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0.947
0.939
0.936
0.930
0.919
O
N
CO2Et
N
CO2Et
Cl
Ph
3ai
2.00
2.03
2.10
3.07
2.08
1.02
1.08
3.00
3.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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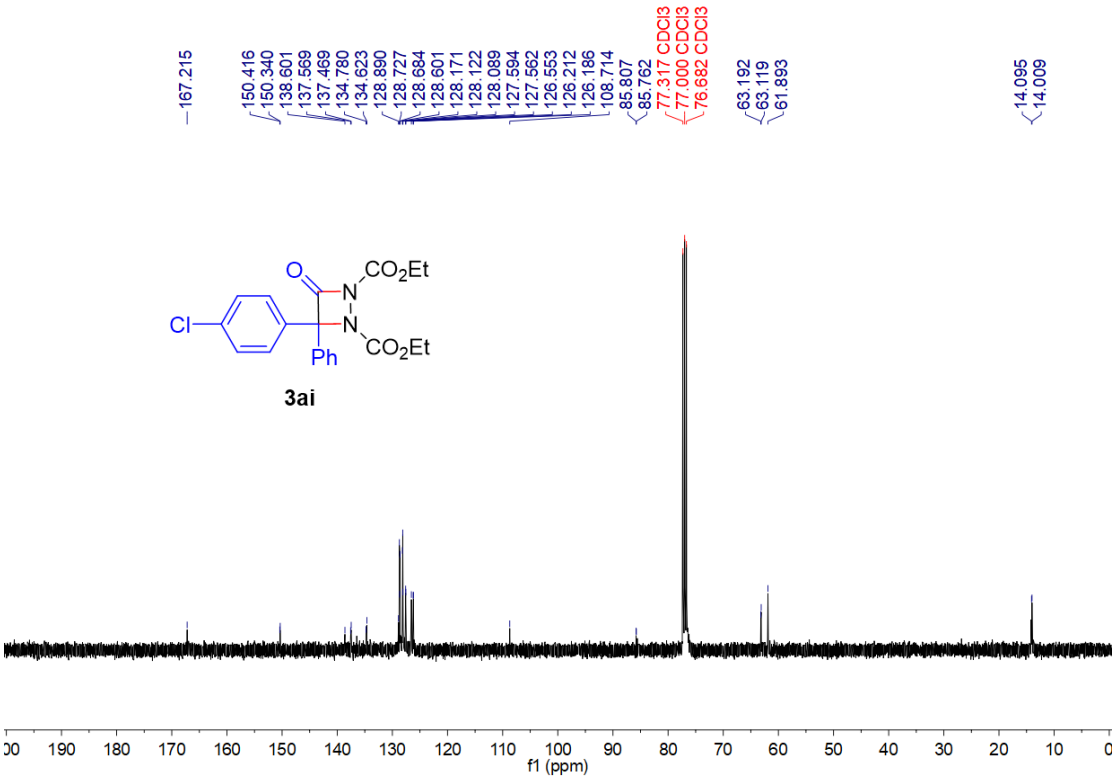
f1 (ppm)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128.601
128.171
128.122
128.089
127.594
127.562
126.553
126.212
126.186
108.714
85.807
85.762
77.317 CDCl3
77.000 CDCl3
76.682 CDCl3
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63.119
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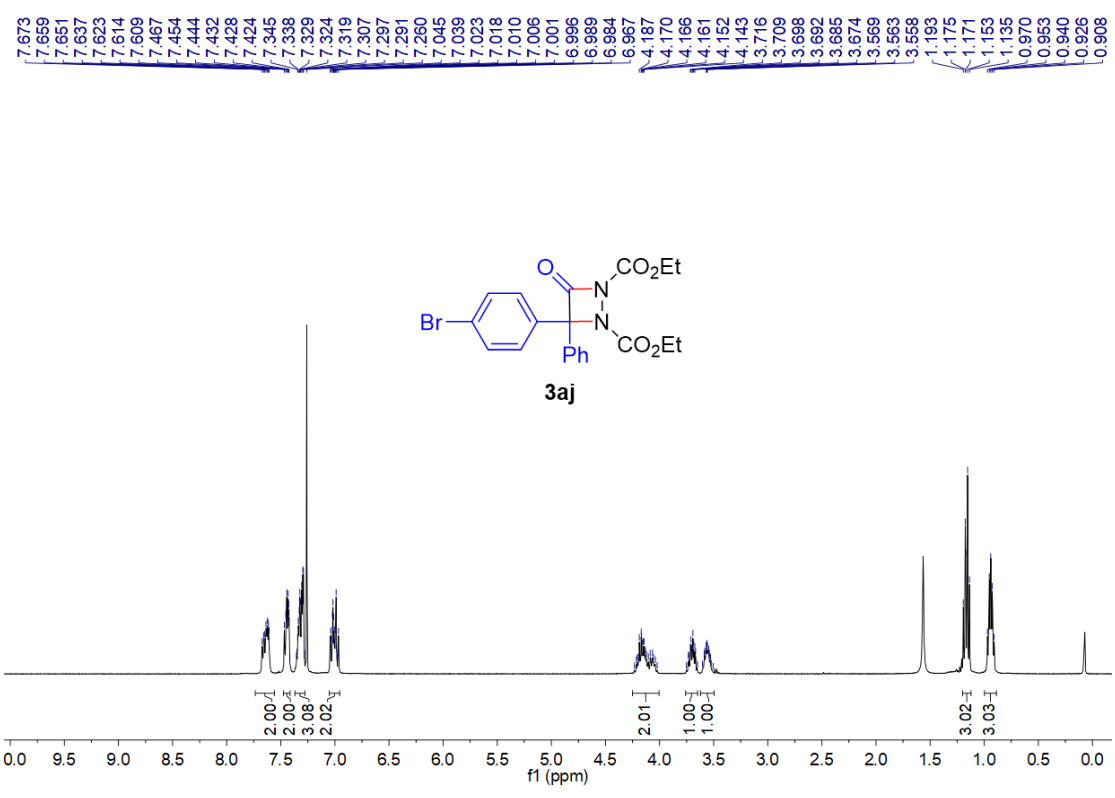
14.095
14.009
O
CO2Et
N
N
CO2Et
Cl
Ph
f1 (ppm)

3ai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7.424
7.345
7.338
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7.324
7.319
7.307
7.297
7.291
7.260
7.045
7.039
7.023
7.018
7.010
7.006
7.001
6.996
6.989
6.984
6.967
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4.170
4.166
4.161
4.152
4.143
3.716
3.709
3.698
3.692
3.685
3.674
3.569
3.563
3.558
1.193
1.175
1.171
1.153
1.135
0.970
0.953
0.940
0.926
0.908
O
CO2Et
N
N
CO2Et
Br
Ph
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2.00
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2.01
1.00
1.00
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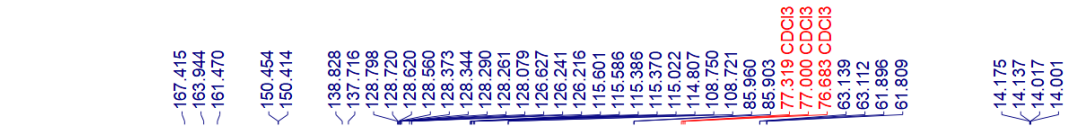
3.03
f1 (ppm)

3aj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128.373
128.344
128.290
128.261
128.079
126.627
126.241
126.216
115.601
115.586
115.386
115.370
115.022
114.807
108.750
108.721
85.960
85.903
77.319 CDCl3
77.000 CDCl3
76.683 CDCl3
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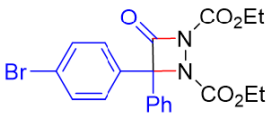
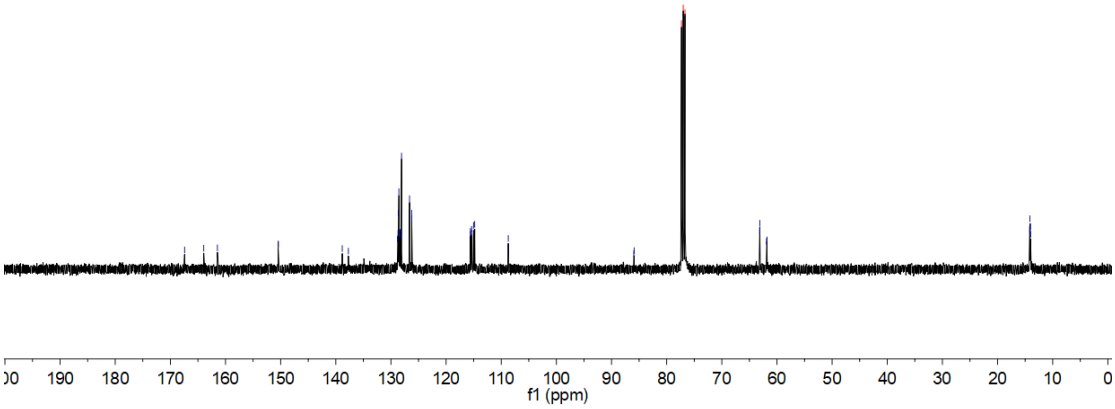
14.001


O
N
N
CO2Et
CO2Et
Br
Ph

3aj


f1 (ppm)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7.173
7.157
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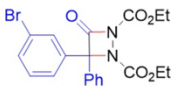
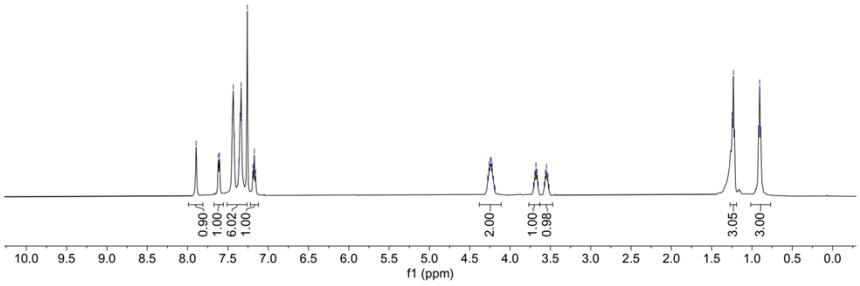

Br
O
N
N
CO2Et
CO2Et
Ph

3ak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0.98
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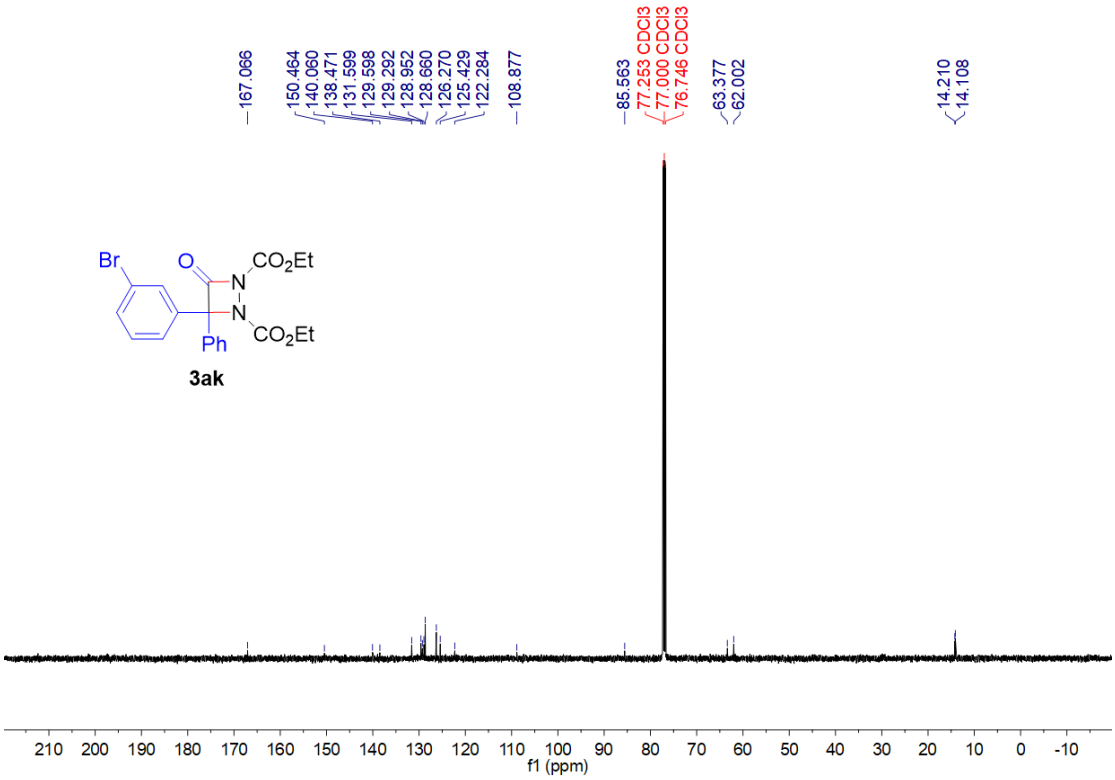
f1 (ppm)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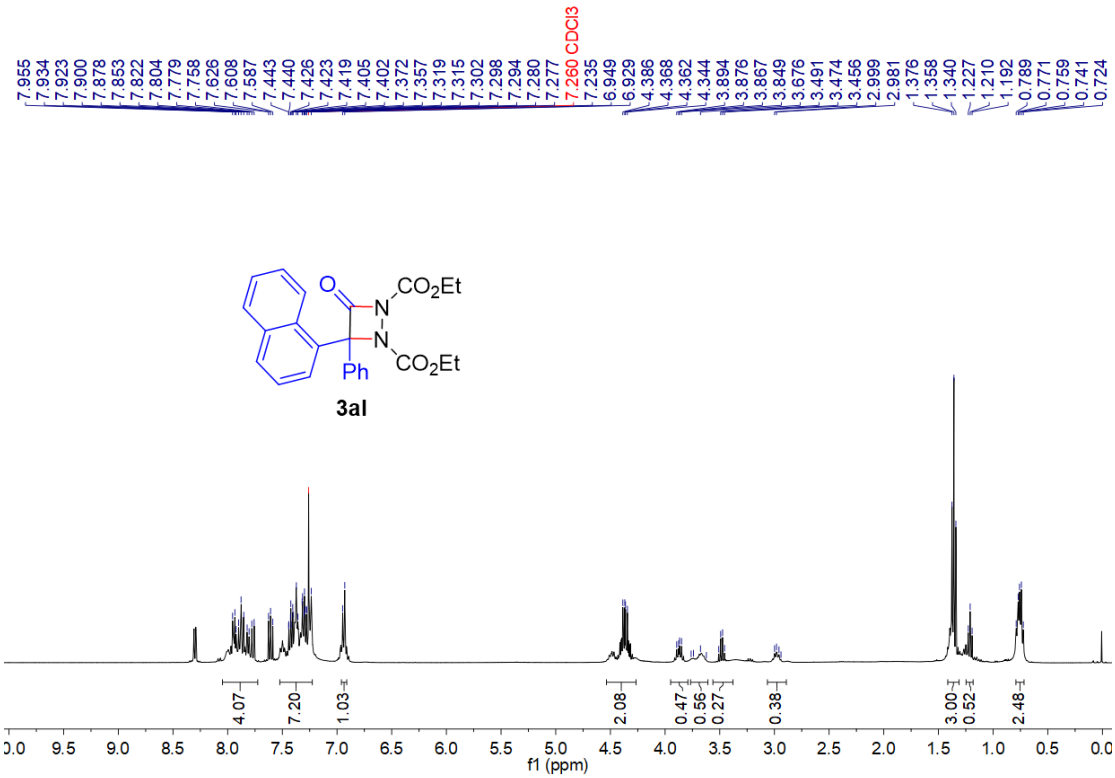
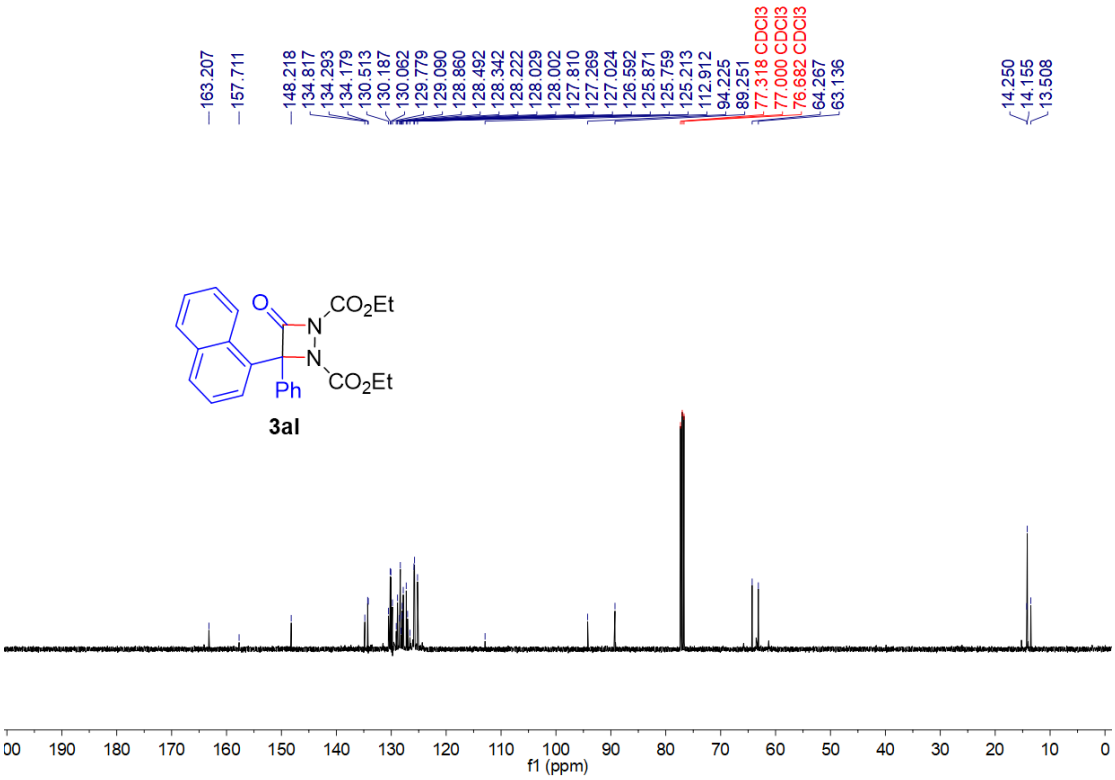
126.270
125.429
122.284
108.877
85.563
77.253 CDCl3
77.000 CDCl3
76.746 CDCl3
63.377
62.002
14.210
14.108
Br
O
N
N
CO2Et
CO2Et
Ph
3ak
f1 (ppm)


7.955
7.934
7.923
7.900
7.878
7.853
7.822
7.804
7.779
7.758
7.626
7.608
7.587
7.443
7.440
7.426
7.423
7.419
7.405
7.402
7.372
7.357
7.319
7.315
7.302
7.298
7.294
7.280
7.277
7.260 CDCl3
7.235
6.949
6.929
4.386
4.368
4.362
4.344
3.894
3.876
3.867
3.849
3.676
3.491
3.474
3.456
2.999
2.981
1.376
1.358
1.340
1.227
1.210
1.192
0.789
0.771
0.759
0.741
0.724
O
N
N
CO2Et
CO2Et
Ph
3al
4.07
7.20
1.03
2.08
0.47
0.56
0.27
0.38
3.00
0.52
2.48
f1 (ppm)


163.207
157.711
148.218
134.817
134.293
134.179
130.513
130.187
130.062
129.779
129.090
128.860
128.492
128.342
128.222
128.029
128.002
127.810
127.269
127.024
126.592
125.871
125.759
125.213
112.912
94.225
89.251
77.318 CDCl3
77.000 CDCl3
76.682 CDCl3
64.267
63.136
14.250
14.155
13.508
O
N
N
CO2Et
CO2Et
Ph
3al
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)