Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Electrochemical dehydroxymethylative functionalization of alkanols for forging C(sp^3)−heteroatom bonds

Lulu Zhao,[a] Jian Tian,[a] Qilin Yuan,[a] Qiwen Zhong,[a] Mengqi Luo,[a] Chao Yang,[a]
Lin Guo,[a*] and Wujiong Xia [a,b*]

[a] *State Key Lab of Urban Water Resource and Environment, School of Science, Harbin Institute of Technology (Shenzhen), Shenzhen, 518055, China.*

**Emails: guolin@hit.edu.cn; xiawj@hit.edu.cn*

[b] *School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang, Henan 453007, China*

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1. General Information

All commercially available reagents were used as supplied without further purification unless stated otherwise. Reactions were monitored by thin-layer chromatography (TLC) on commercial silica gel plates (GF 254) using UV light as a visualizing agent. Products were purified by flash chromatography on 200 – 300 mesh silica gels, SiO_2. Graphite, reticulated vitreous carbon (RVC), glassy carbon (GC) and Pt electrodes were purchased from taobao. Graphite electrodes could be used several times by renewing the top surface of the graphite.

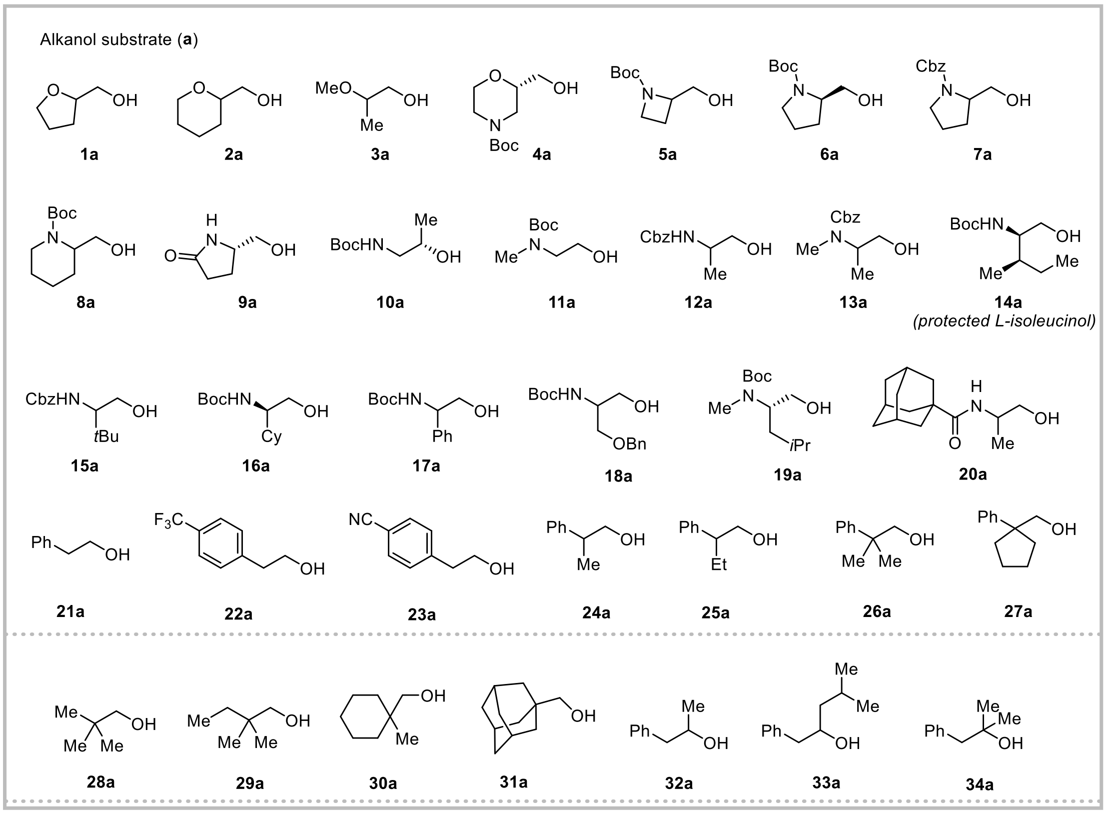
^{1}H NMR (400 MHz), ^{13}C NMR (101 MHz) and ^{19}F NMR (376 MHz) spectra were recorded on a Quantum-I Plus 400 in $CDCl_3$. For ^{1}H NMR, $CDCl_3$ ($\delta = 7.26$ ppm) tetramethylsilane (TMS, $\delta = 0$ ppm) serves as the internal standard; for ^{13}C NMR, $CDCl_3$ ($\delta = 77.16$ ppm) serves as the internal standard. Data are reported as follows: chemical shift (in ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = quintet, h = heptet, m = multiplet, br = broad), coupling constant (in Hz), and integration.

The electrochemical reactions were performed on a DJS-292B potentiostat (made in China) in constant current mode. HR-MS spectra were recorded on a Waters Xevo G2QTOF/UPLC mass spectrometer using electrospray ionization. The cyclic voltammetry measurements were detected by using a CHI 760E electrochemical workstation.

The procedures for the synthesis of substrates are according to the reported method.

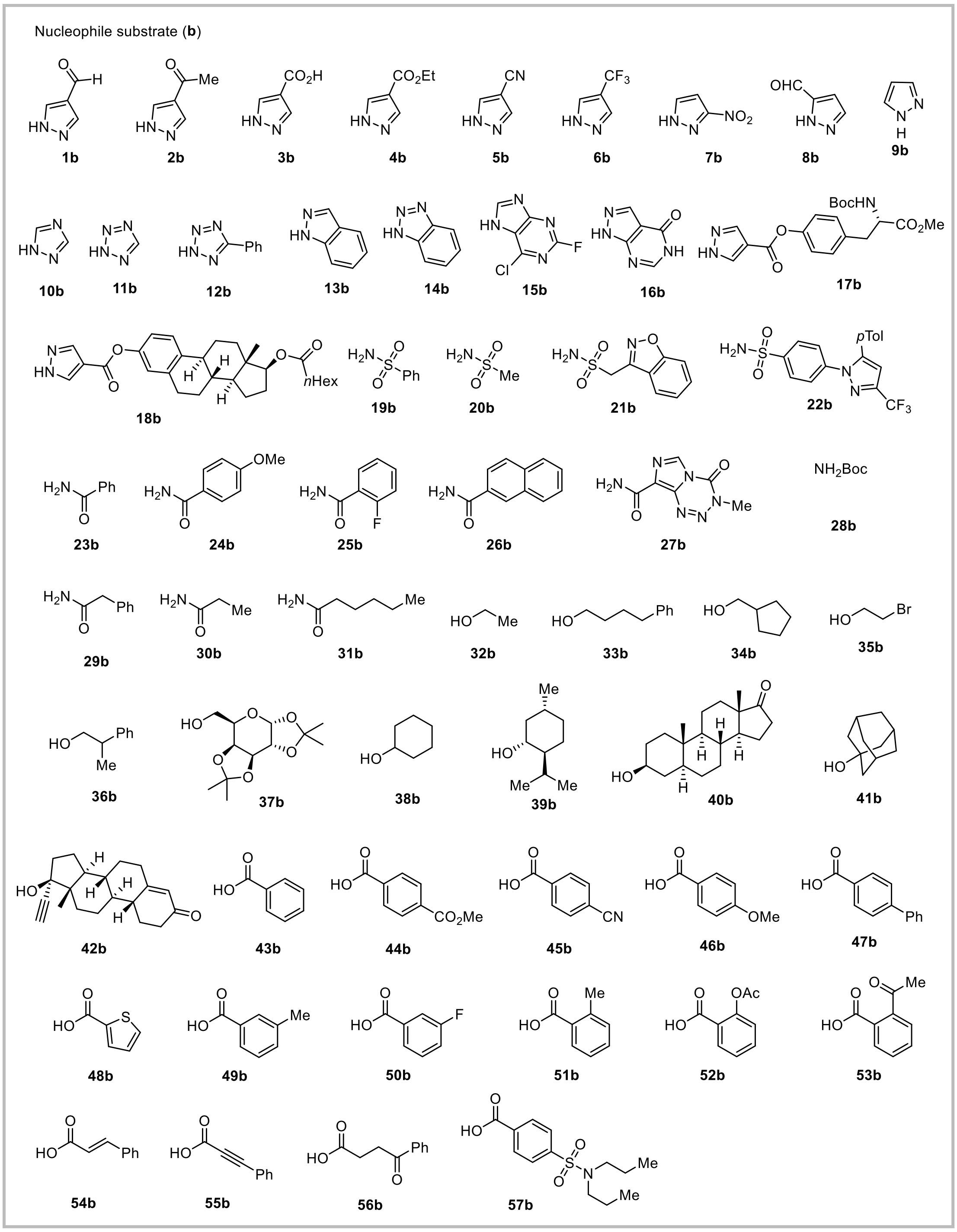
2. Compound Table

2.1 Alkanol substrate (a)

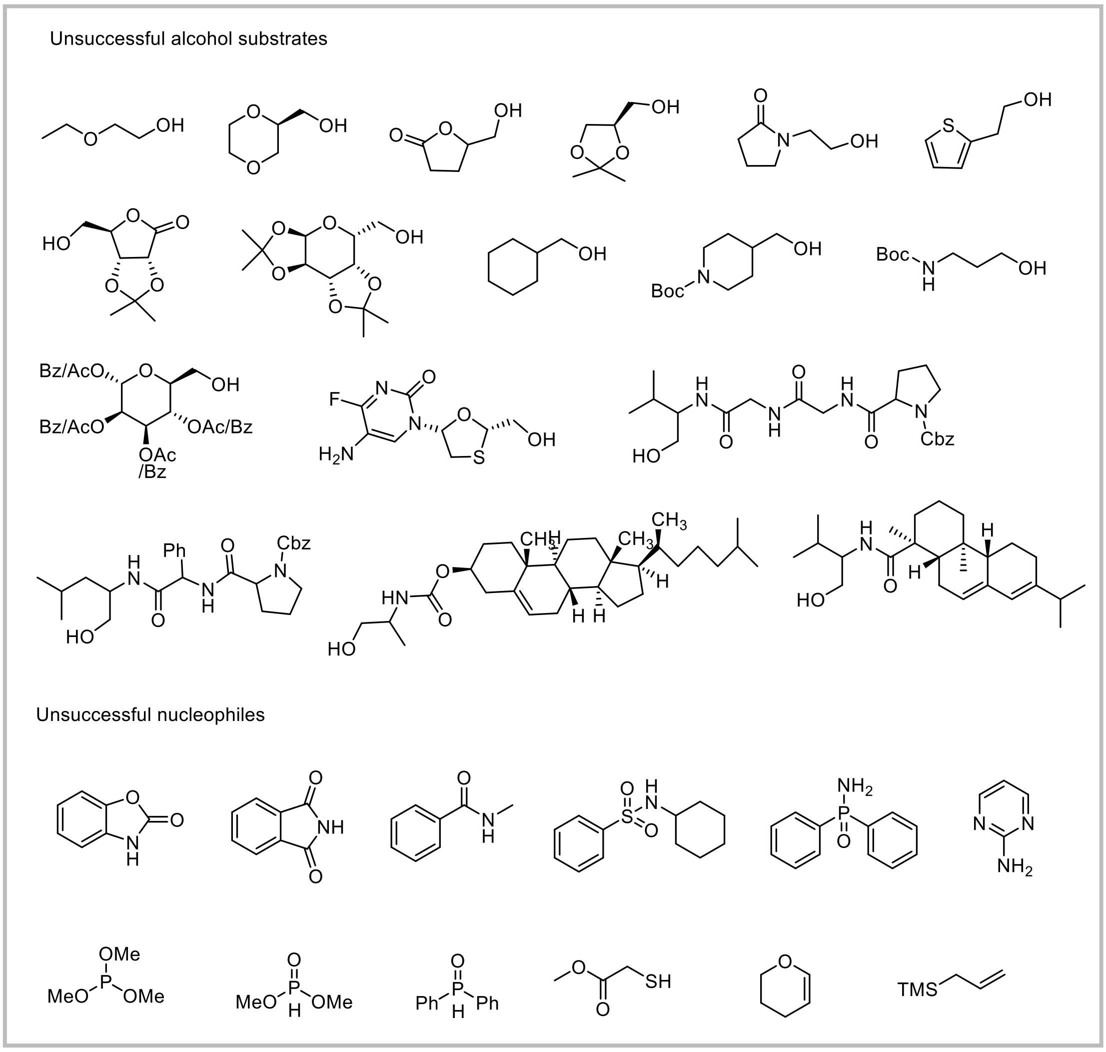


Alkanol substrate (**a**)

2.2 Nucleophile substrate (b)



2.3 Unsuccessful substrates (no desired dehydroxymethylative products were detected)



3. General Procedures

Materials used for set-up:

25 mL three-necked flask, carbon rod (ϕ = 4 mm) anode, platinum plate ($10 \times 15 \times 0.2$ mm^3) cathode, DJS-292B potentiostat.

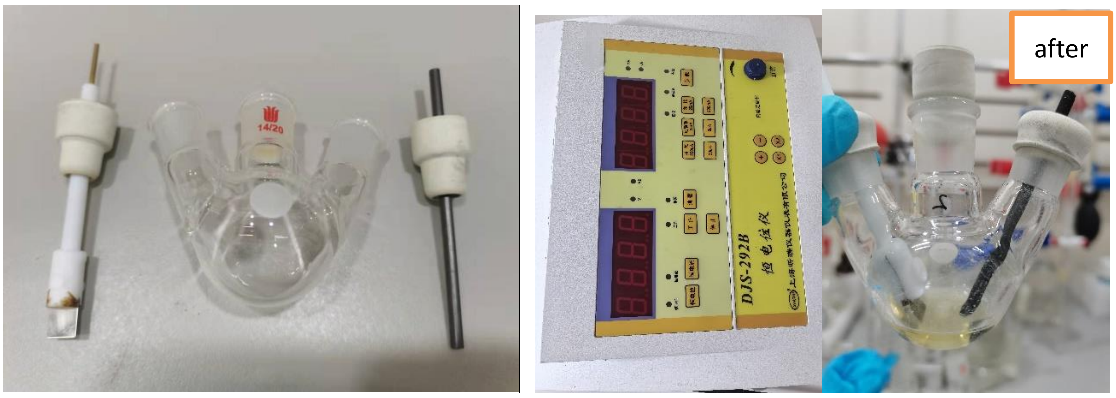
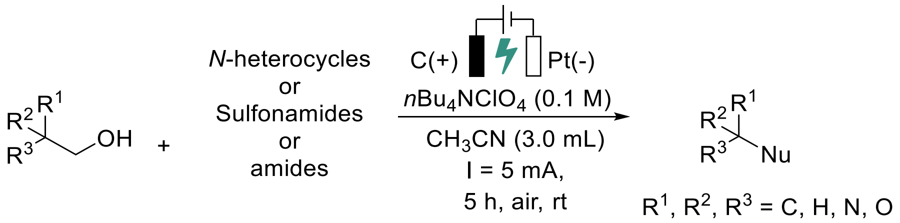


Figure S1. Materials used for set-up

3.1 Method A of construction C-*N* bond

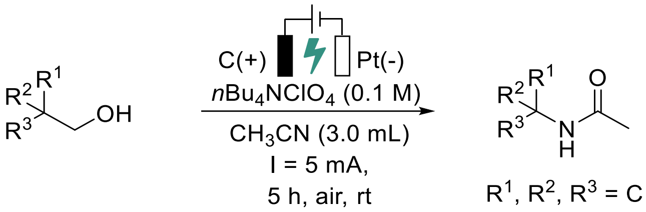
To a three-necked flask equipped with a stir bar, a carbon rod anode, and a platinum plate cathode was added the substrate alkyl alcohol (0.30 mmol) and nucleophiles, including *N*-heterocycles, sulfonamides and amides (0.45 mmol, 1.5 equiv.) and electrolyte nBu$_4$NClO$_4$ (0.1 M), followed by 3.0 mL solvent (CH_3CN). The distance of electrodes was approximately 1.0 cm. The constant current (5.0 mA) electrolysis was then performed at room temperature under air atmosphere with stirring for the indicated time as monitored by TLC or NMR analysis. After completion of the reaction, the reaction mixture was poured into a brine solution and extracted with 20 mL EA for three times, and the combined organic layers were dried over anhydrous Na_2SO_4. The resulting mixture was purified by column chromatography on silica gel (eluted with PE/EA = 5:1) to afford the desired products.



Scheme S1. Reaction for method A

3.2 Method B of construction C-*N* bond

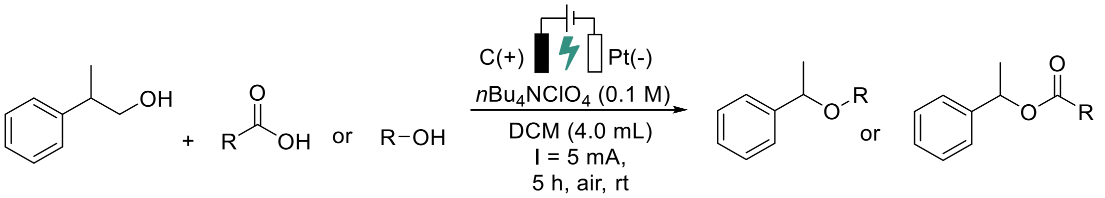
To a three-necked flask equipped with a stir bar, a carbon rod anode, and a platinum plate cathode was added the substrate alkyl alcohol (0.30 mmol) and electrolyte *n*Bu$_4$NClO$_4$ (0.1 M), followed by 3.0 mL solvent (CH$_3$CN). The distance of electrodes was approximately 1.0 cm. The constant current (5.0 mA) electrolysis was then performed at room temperature under air atmosphere with stirring for the indicated time as monitored by TLC or NMR analysis. After completion of the reaction, the reaction mixture was poured into a brine solution and extracted with 20 mL EA for three times, and the combined organic layers were dried over anhydrous Na$_2$SO$_4$. The resulting mixture was purified by column chromatography on silica gel (eluted with PE/EA = 5:1) to afford the desired products.



Scheme S2. Reaction for method B

3.3 Method C of construction C-O bond

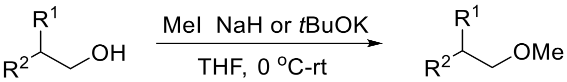
To a three-necked flask equipped with a stir bar, a carbon rod anode, and a platinum plate cathode was added the substrate β-methylphenethyl alcohol (0.30 mmol) and containing O-nucleophiles, including acid and alcohol (0.45 mmol, 1.5 equiv.) and electrolyte *n*Bu$_4$NClO$_4$ (0.075 M), followed by 4.0 mL solvent (DCM). The distance of electrodes was approximately 1.0 cm. The constant current (5.0 mA) electrolysis was then performed at room temperature under air atmosphere with stirring for the indicated time as monitored by TLC or NMR analysis. After completion of the reaction, the reaction mixture was poured into a brine solution and extracted with 20 mL EA for three times, and the combined organic layers were dried over anhydrous Na$_2$SO$_4$. The resulting mixture was purified by column chromatography on silica gel (eluted with PE/EA = 20:1) to afford the desired products.



Scheme S3. Reaction for method C

3.4 Method E of preparation of ether[1].

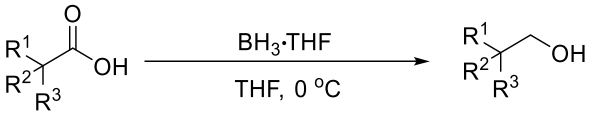
To a 100 mL round bottom flask equipped with a stir bar, was added the primary alcohol (5.0 mmol) and NaH (1.5 equiv.) or *t*BuOK (2.0 equiv.) in THF (10 mL). The reaction mixture was sonicated for 5 mins and MeI (2.0 equiv.) was added dropwise at 0 °C. The reaction was stirred overnight and the solvent was removed. EA was added and washed with water. The organic layer was dried over Na_2SO_4 and purified by column chromatography on silica gel (20:1 → 5:1) to afford pure ether.



Scheme S4. Reaction for method E

3.5 Method F of preparation of alcohols[2].

To a solution of acid (*N*-protected α-amino acid) (0.5 M, 1.0 equiv.) in THF at 0 °C was added slowly borane-THF (2.0 equiv.). The reaction mixture was then allowed to stir at room temperature for 90 min. The reaction mixture was then quenched with saturated $NaHCO_3$ (2.0 mL/mmol) at 0 °C, and extracted with EA. The organic phase was then dried over $MgSO_4$, concentrated under vacuum, and the residue was purified on a silica gel column chromatography to yield alcohols (*N*-Protected amino alcohols).



Scheme S5. Reaction for method F

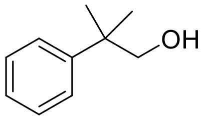
3.6 Method G of preparation of *N*-protected Amines [3].

To a solution of the amine (amino alcohols) in DMF (50 mL) was added NaH (1.0 equiv., 60% dispersion in mineral oil) at 0 °C. After stirring at 0 °C for 15 min and then at rt for 1 h, the reaction mixture was cooled to 0 °C, treated with Boc_2O (1.1 equiv.), and then allowed to stir at rt for 6 -12 h. After the reaction was complete (monitored by TLC), aqueous saturated $NaHCO_3$ was added slowly. The organic layer was separated and the aqueous layer was extracted with EA. The combined organic layers were dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified on a silica gel column chromatography to yield cyclobutanol.

$$\mathrm{R(Ar)NH_2} + \mathrm{Boc_2O} \xrightarrow[\text{DMF, 0 °C - rt}]{\substack{\text{NaH (1.0 equiv.)} \\ \text{Boc}_2\text{O (1.1 equiv.)}}} \mathrm{R(Ar)NHBoc}$$

Scheme S6. Reaction for method G

4. Characterization Data of Substrates

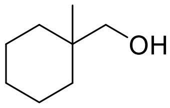


2-methyl-2-phenylpropan-1-ol (26a)

Starting from 2-methyl-2-phenylpropanoic acid (5.0 mmol) with borane-THF (2.0 equiv.) following method F-4 and the product was purified by flash chromatography using PE/EA (10:1 → 5:1) to afford a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.40 – 7.31 (m, 4H), 7.25 – 7.19 (m, 1H), 3.59 (s, 2H), 1.43 (s, 1H), 1.33 (s, 6H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 146.3, 128.4, 126.2, 126.2, 73.0, 40.0, 25.3. (see spectrum)

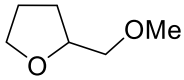


1-methylcyclohexyl)methanol (30a)

Starting from 1-methylcyclohexane-1-carboxylic acid (5.0 mmol) with borane-THF (2.0 equiv.) following method F-4 and the product was purified by flash chromatography using PE/EA (10:1 → 5:1) to afford a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 3.34 (s, 2H), 1.67 (d, J = 9.0 Hz, 1H), 1.47 (qd, J = 10.6, 9.2, 4.2 Hz, 5H), 1.34 – 1.22 (m, 5H), 0.91 (s, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 72.5, 34.9, 34.0, 26.4, 21.9, 21.7. (see spectrum)

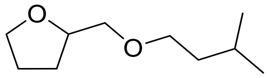


2-(methoxymethyl)tetrahydrofuran (2f)

Starting from tetrahydrofurfuryl alcohol (5.0 mmol) with MeI (2.0 equiv.) following method E and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 4.05 (qd, J = 6.8, 4.2 Hz, 1H), 3.89 (dt, J = 13.2, 6.7 Hz, 1H), 3.81 – 3.74 (m, 1H), 3.45 – 3.35 (m, 5H), 2.00 – 1.84 (m, 3H), 1.60 (ddd, J = 15.0, 11.4, 7.3 Hz, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 77.8, 75.4, 68.4, 59.4, 28.1, 25.8. (see spectrum)

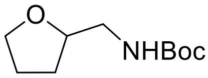


2-((isopentyloxy)methyl)tetrahydrofuran (3f)

Starting from tetrahydrofurfuryl alcohol (5.0 mmol) with 1-iodo-3-methylbutane (2.0 equiv.) following method E and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.04 (p, *J* = 6.0 Hz, 1H), 3.88 (q, *J* = 7.2, 6.6 Hz, 1H), 3.76 (q, *J* = 7.2 Hz, 1H), 3.49 (td, *J* = 7.0, 1.6 Hz, 2H), 3.43 (d, *J* = 5.3 Hz, 2H), 1.99 – 1.82 (m, 3H), 1.69 (tt, *J* = 12.9, 6.4 Hz, 1H), 1.60 (ddd, *J* = 15.2, 10.0, 5.8 Hz, 1H), 1.49 (q, *J* = 6.9 Hz, 2H), 0.90 (d, *J* = 6.6 Hz, 6H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 77.9, 73.5, 70.0, 68.3, 38.4, 28.1, 25.6, 25.0, 22.6, 22.5. (see spectrum)



tert-butyl ((tetrahydrofuran-2-yl)methyl)carbamate (4f)

Starting from (*S*)-(+)-tetrahydrofurfurylamine (5.0 mmol) with Boc_2O (1.1 equiv.) following method G and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.93 (s, 1H), 3.95 (qd, *J* = 6.9, 3.4 Hz, 1H), 3.89 – 3.82 (m, 1H), 3.75 (q, *J* = 7.9, 7.5 Hz, 1H), 3.38 (ddd, *J* = 13.1, 6.2, 3.3 Hz, 1H), 3.06 (dt, *J* = 13.4, 6.2 Hz, 1H), 1.89 (dt, *J* = 12.7, 6.0 Hz, 3H), 1.61 – 1.54 (m, 1H), 1.44 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.1, 79.2, 78.0, 68.0, 44.3, 28.4, 28.3, 25.8. (see spectrum)

5. Reaction Optimizations

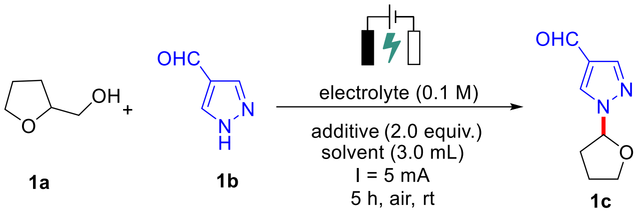


Table S1. Screening of electrolytes

Entry	electrolyte	additive	solvent	**1c** yield (%)
1	$n\mathrm{Bu_4NClO_4}$	Cs_2CO_3	CH_3CN	69
2	$n\mathrm{Bu_4NOAc}$	Cs_2CO_3	CH_3CN	<10
3	$n\mathrm{Bu_4NPF_6}$	Cs_2CO_3	CH_3CN	62
4	$n\mathrm{Bu_4NBF_4}$	Cs_2CO_3	CH_3CN	49
5	$n\mathrm{Bu_4NCl}$	Cs_2CO_3	CH_3CN	trace
6	$n\mathrm{Bu_4PBr}$	Cs_2CO_3	CH_3CN	trace

Table S2. Screening of additives

Entry	electrolyte	additive	solvent	**1c** yield (%)
1	$n\mathrm{Bu_4NClO_4}$	KOAc	CH_3CN	16
2	$n\mathrm{Bu_4NClO_4}$	K_2CO_3	CH_3CN	58
3	$n\mathrm{Bu_4NClO_4}$	Cs_2CO_3	CH_3CN	68
4	$n\mathrm{Bu_4NClO_4}$	*t*BuOK	CH_3CN	62
5	$n\mathrm{Bu_4NClO_4}$	K_3PO_4	CH_3CN	57
6	$n\mathrm{Bu_4NClO_4}$	CsF	CH_3CN	56
7	$n\mathrm{Bu_4NClO_4}$	------	CH_3CN	81

Table S3. Screening of solvents

Entry	electrolyte	solvent	**1c** yield (%)
1	$\boldsymbol{n}\mathbf{Bu_4NClO_4}$	$\mathbf{CH_3CN}$	**81**
2	$n\mathrm{Bu_4NClO_4}$	DCM	40
3	$n\mathrm{Bu_4NClO_4}$	DMF	n.r
4	$n\mathrm{Bu_4NClO_4}$	DMSO	n.d
5	$n\mathrm{Bu_4NClO_4}$	Acetone	35
6	$n\mathrm{Bu_4NClO_4}$	CH_3CN/HFIP (2.6:0.4 mL)	46

Table S4. Screening of electrode

Entry	electrolyte	solvent	electrode	**1c** yield (%)
1	***n*Bu$_4$NClO$_4$**	**CH$_3$CN**	**C(+)/Pt(-)**	**81**
2	*n*Bu$_4$NClO$_4$	CH$_3$CN	C felt (+)/Pt(-)	n.d
3	*n*Bu$_4$NClO$_4$	CH$_3$CN	Pt(+)/Pt(-)	54
4	*n*Bu$_4$NClO$_4$	CH$_3$CN	GC(+)/Pt(-)	trace
5	*n*Bu$_4$NClO$_4$	CH$_3$CN	C(+)/C(-)	70

Table S5. Screening of electric current

Entry	Current (mA)	Time (hour)	**1c y**ield (%)
1	3	8	16
2	**5**	**5**	**81**
3	8	3	27
4	5	4	63
6	5	6	74

6. Mechanistic Studies

6.1 Cyclic voltammetry studies

Substrates redox potentials were determined using CV with the following method in Figure S2-S4. Cyclic voltammetry experiments were performed on a CHI 760E potentiostat using a glassy carbon as working electrode, a platinum wire as counter electrode, and a saturated calomel electrode (SCE) as reference electrode. Samples were prepared with 0.1 mmol of substrate in 10 mL of 0.1 M tetrabutylammonium hexafluorophosphate (*n*Bu_4NPF_6) in dry, degassed acetonitrile. The scan rate is 100 mV/s. Potentials are reported by using half-peak potentials against SCE. The measured values are basically consistent with the literature. [4]

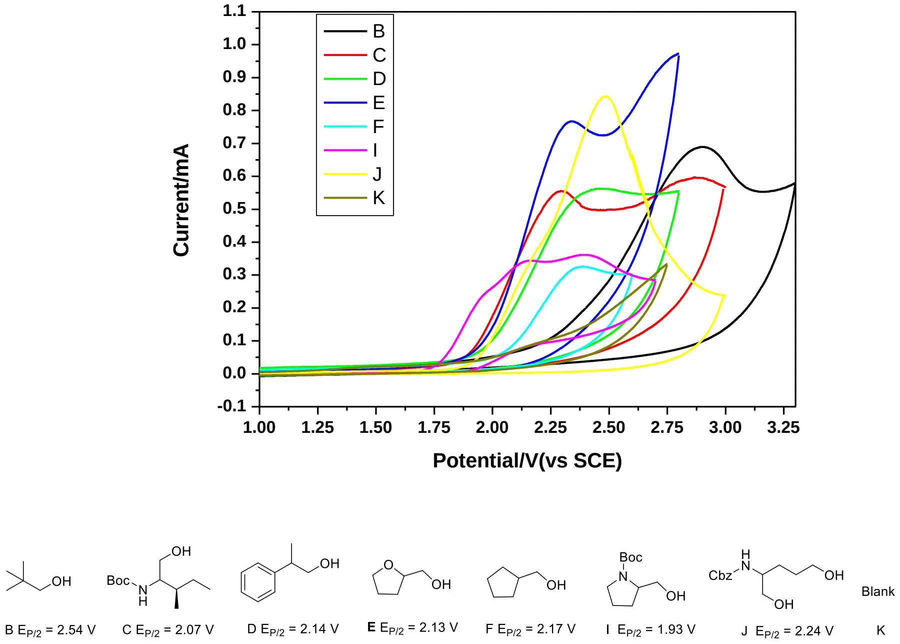


Figure S2. Cyclic voltammetry of alcohols

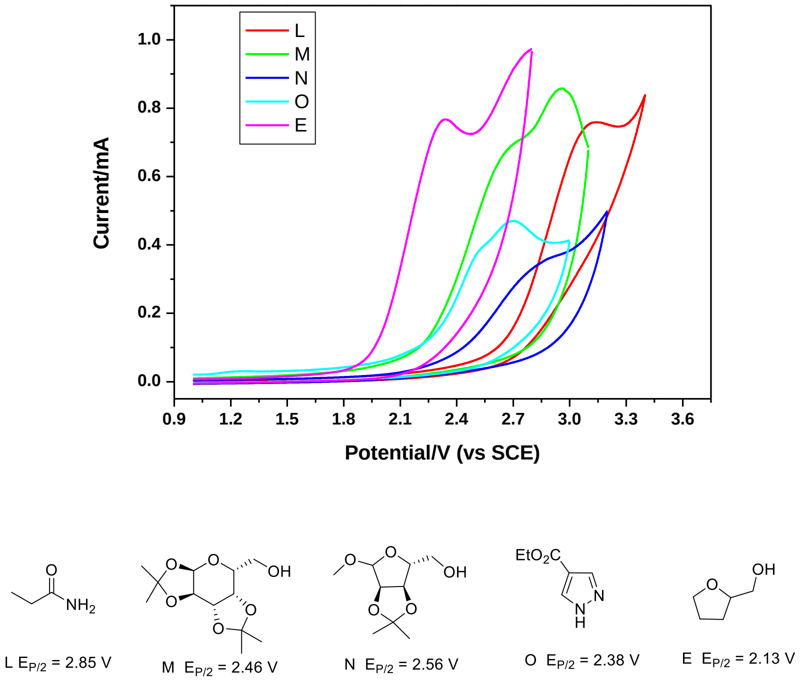


Figure S3. Cyclic voltammogram of nucleophiles.

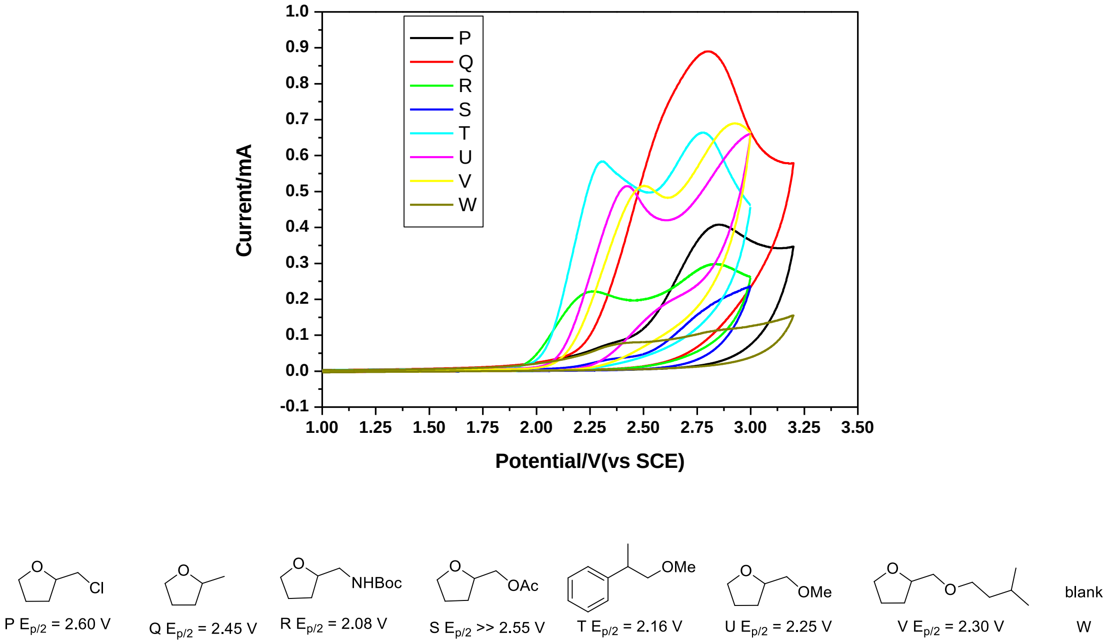
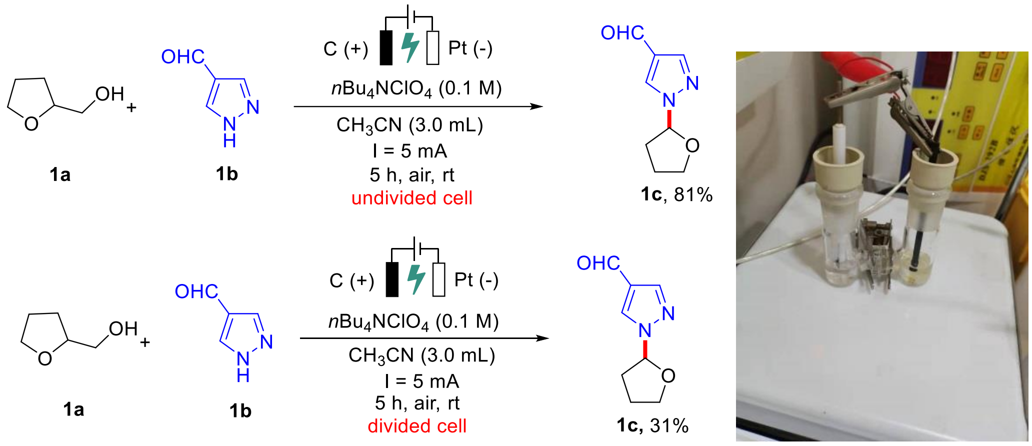


Figure S4. Cyclic voltammogram of substrates

6.2 Comparison of standard conditions with conditions in divided cell

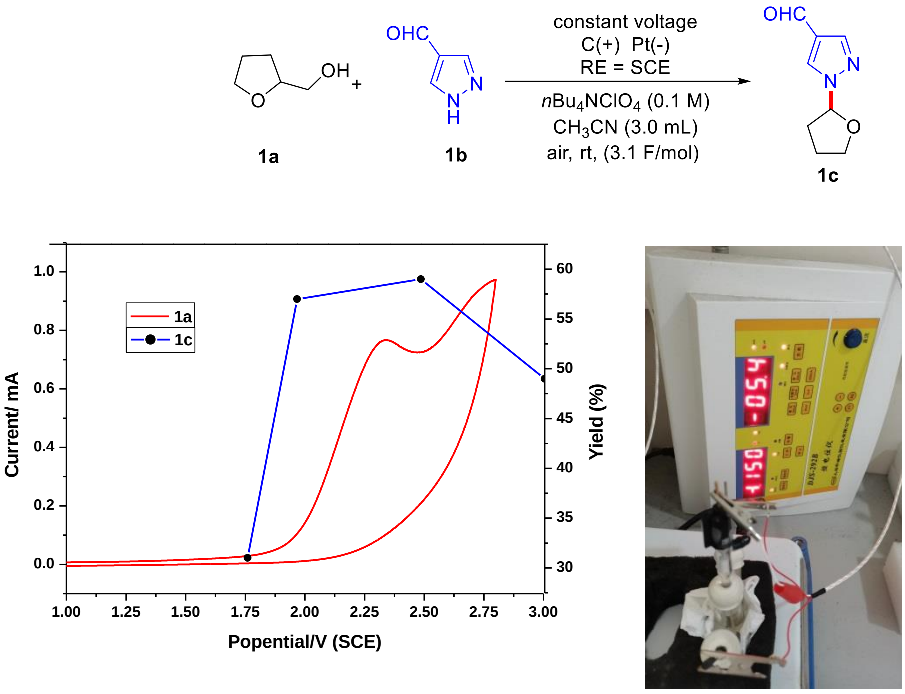
The reaction vessel is an H-type divided electrolytic cell (10 mL+ 10 mL) separated by a AMI-7001-30 membrane. The Part of the anode was added the substrate (tetrahydrofuran-2-yl)methanol (**1a**, 0.30 mmol, 30.6 mg), 1H-pyrazole-4-carbaldehyde (**1b**, 0.45 mmol, 1.5 equiv.) and electrolyte *n*Bu$_4$NClO$_4$ (0.1 M), CH$_3$CN (3.0 mL) and a magnetic stirrer bar. The part of the cathode was added electrolyte1H-pyrazole-4-carbaldehyde (**1b**, 0.30 mmol) and *n*Bu$_4$NClO$_4$ (0.1 M), CH$_3$CN (3.0 mL) and a magnetic stirrer bar. Subsequently, the flask was equipped with a graphite rod anode and a platinum plate cathode, the distance between which was approximately 3.0 cm. The constant current (5.0 mA) electrolysis was then performed at room temperature under air atmosphere (sealed tube) with vigorous stirring for 5 h. The resulting mixture was monitored by TLC, only trace desired product was detected. These results suggest that all of the steps occur in the vicinity of the anode.



Scheme S7. Reaction formula

6.3 Constant potential electrolysis

All the electrochemical measurements were performed DJS-292B potentiostat. For constant potential electrolysis experiments, the reduced electric potential induces significantly longer total reaction time，in order to achieve the constant total charge transfer. Therefore, slightly reduced amount of the total charge (3.1 F/mol) was utilized to manage technical limitations. The three-electrode system was used with graphite rod as the working and a platinum plate as counter electrode and a saturated calomel electrode (SCE) as the reference electrode. The constant potential electrolysis was performed in various potentials and was terminated based on total charge passed through the circuit, 3.1 F/mol. The reaction yield was isolated yield.

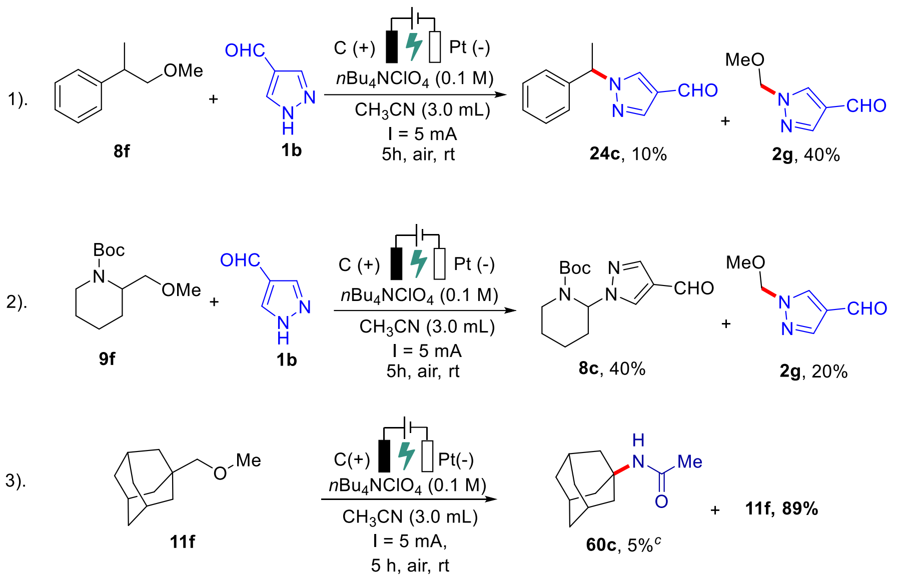


Scheme S8. Reaction formula and set-up

6.4 Effect on leaving groups.

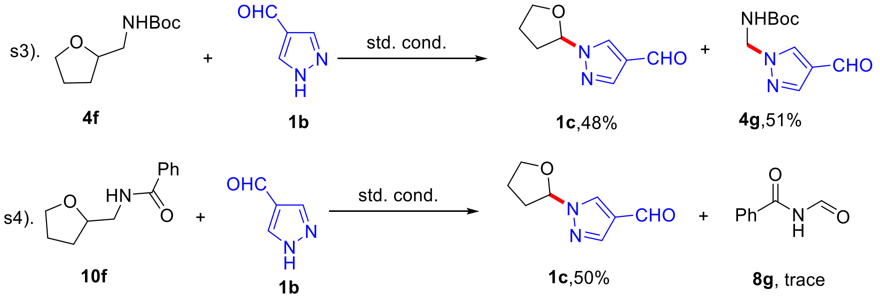
Under standard conditions, we attempted to use alcohols protected by different alkyl groups, methyl ether substrate **8f** and **9f** reacted with **1b**, forming a small amounts of products **24c/8c** and **2g** respectively, which revealed that in the reaction mechanism may involve alkoxy radical intermediates. However, the relevant literatures reported that a variety of aromatic compounds was oxidized at the anode giving radical cation, which undergo side-chain fragmentation involving the C_α-H and/or C_α-C_β bond, and it clearly shows that an OH group is much more effective than an OCH_3 group in promoting C_a-C_b bond cleavage of an alkylaromatic radical cation. However, the relevant literature[5] reported that a variety of aromatic compounds was oxidized at the anode giving radical cation, which undergo side-chain fragmentation involving the C_α-H and/or C_α-C_β bond, and it clearly shows that an OH group is much more effective than an OCH_3 group in promoting C_α-C_β bond cleavage of an alkylaromatic radical cation.

While methyl ether substrate **11f** was employed as the substrate using the electrochemical reaction conditions, gave corresponding Ritter-type products **60c** only 5% with 89% starting material recovered (scheme S9, eq. 3), which revealed that in the reaction mechanism may involve alkoxy radical intermediates.



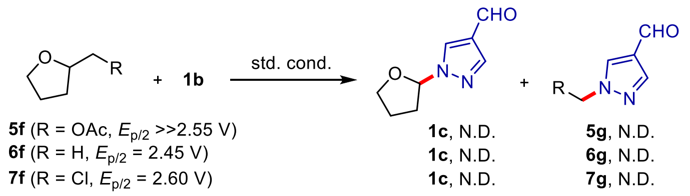
Scheme S9. Reaction formula

When we attempt to use the (tetrahydrofuran-2-yl)methanamine derivatives (**4f** or **10f**) as the substrates, with the pyrazole **1b** as the coupling partner, also providing the product **1c** (Scheme S10).



Scheme S10. Reaction formula

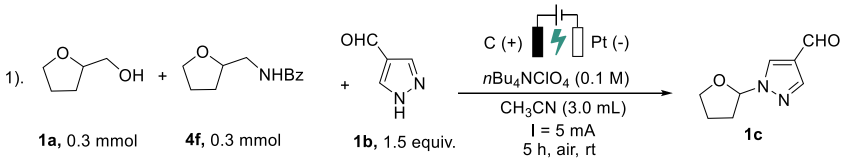
Alternatively, the priority oxidation is not alcohol hydroxyl groups. If the alcohol hydroxyl protection group of alcohol is replaced by acetyl groups (**5f**), 2-methyltetrahydrofuran (**6f**) or 2-(chloromethyl)tetrahydrofuran (**7f**), no product **1c** is generated (Scheme S11). It could be inferred that the electron-withdrawing group has a negative fluence and weak electron-donating groups with higher oxidation potential. Perhaps the alcohol containing N, O or aromatic rings in the hydroxyl β position, are preferentially oxidized.



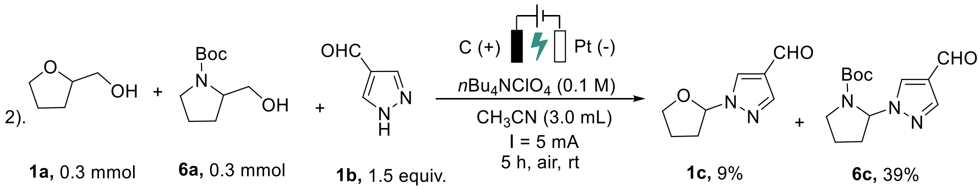
Scheme S11. Reaction formula

6.5 Comparison of N and O atoms.

In order to further explore the oxidation sequence of O and *N*, a series of control experiments were performed. To a three-necked flask equipped with a stir bar, a carbon rode anode, and a platinum wire cathode was added the substrate **1a** (0.3 mmol), **4f** (0.3 mmol) and **1b** (0.45 mmol), continuous electrolysis (5 mA) for 5 hours. It can be found that the **1a** converted 39%, while **4f** converted 98%. Meanwhile, using **1a** and **6a** reacted with **1b** under the standard condition, forming corresponding product **1c** (9%) and **6c** (39%). These results indicated that *N* atom are oxidized preferentially than O atom.



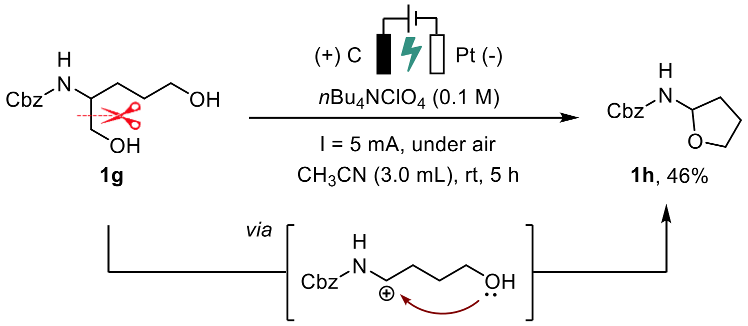
	1a	**4f**
conversion (%)	39%	98%



	1c	**6c**
yield (%)	9%	39%

6.6 Intramolecular reaction.

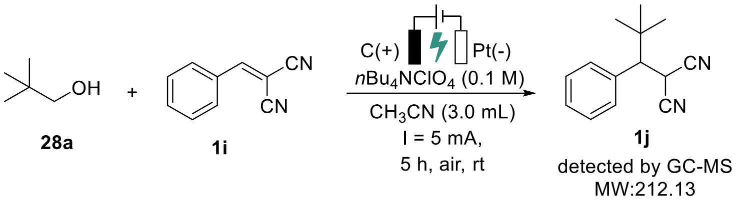
Under standard conditions, when employing the pentadiol as the substrate, the product **1h** via intramolecular nucleophilic reaction was smoothly obtained. This process may involve the carbocation species.



Scheme S12. Reaction formula

6.7 Intramolecular reaction. Radical trapping experiment.

Under standard reaction conditions in the presence of 2-benzylidenemalononitrile **1i** (0.6 mmol, 2.0 equiv.) as a radical receptor and using the **28a** as the substrate, the adduct **1j** was detected (by GC-MS) after 5 hours. This evidence suggested that the reaction involves a radical pathway.



Scheme S13. Reaction formula

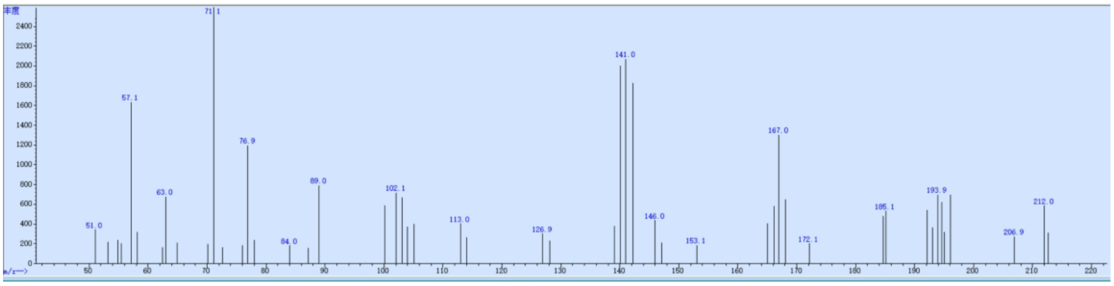
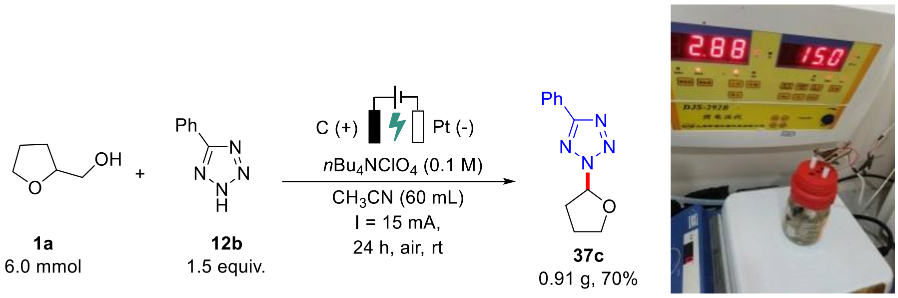


Fig. S5 MS spectrum of possible adduct **1j**

7. Large-Scale Reaction



Scheme S14. Reaction formula

The large scale reaction was carried out using an oven-dried 100-mL beaker, the anode graphite plate (20×20×3.0 mm) and cathode platinum plate (30×30×0.2 mm). To an oven-dried 100 mL beaker equipped with a stir bar, a graphite plate anode, and a platinum plate cathode was added (tetrahydrofuran-2-yl)methanol (**1a,** 6.0 mmol, 0.6171 g), 5-phenyl-2H-tetrazole (**12b,** 9.0 mmol, 1.5 equiv., 1.3200 g) and nBu$_4$NClO$_4$ (0.1 M, 2.0540 g), followed by sequential addition via syringe of CH$_3$CN (60 mL). The reaction mixture was stirred at room temperature at a constant current of 15.0 mA for 24 h. The resulting mixture was purified by column chromatography on silica gel (eluted with PE/EA = 5:1) to afford the desired condensation product **37c** 0.9126 g.

8. Reference

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2. M. Rodriguez, M. Llinares, S. Doulut, A. lleitr and J. Mwtmez, *Tetrahedron Lett.*, 1991, **32**, 923 – 926.
3. C. F. Stanfield, J. E. Parker and P. Kanellls, *J. Org. Chem.*, 1981, **46**, 4797 – 4798.
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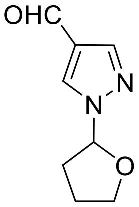
5. (a) E. Baciocchi, M. Bietti, L. Putignani and S. Steenken, *J. Am. Chem. Soc.*, 1996, **118**, 5952 – 5960; (b) E. Baciocchi, M. Bietti and O. Lanzalunga, *Acc. Chem. Res.*, 2000, **33**, 243 – 251; (c) J. Harnedy, H. A. Maashi, A. A. M. A. El Gehani, M. Burns and L. Morrill, C. *Org. Lett.*, 2023*,* **25***,* 1486 –1490.

9. Characterization Data



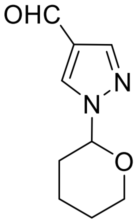
1-(tetrahydrofuran-2-yl)-1H-pyrazole-4-carbaldehyde (1c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 39.85 mg (81% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.12 (s, 1H), 8.00 (s, 1H), 6.01 (dd, *J* = 6.5, 2.1 Hz, 1H), 4.23 – 4.15 (m, 1H), 4.04 (dd, *J* = 15.6, 7.6 Hz, 1H), 2.61 – 2.54 (m, 1H), 2.42 – 2.33 (m, 1H), 2.14 – 2.04 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.2, 141.0, 131.4, 124.1, 90.8, 69.9, 32.3, 23.8. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_8H_{11}N_2O_2$) requires *m/z* 167.0815, found *m/z* 167.0818.



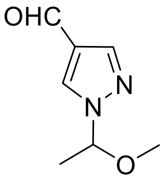
1-(tetrahydro-2H-pyran-2-yl)-1H-pyrazole-4-carbaldehyde (2c)

Starting from tetrahydropyran-2-methanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 34.04 mg (63% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.89 (s, 1H), 8.16 (s, 1H), 8.00 (s, 1H), 5.43 (dd, *J* = 9.1, 2.3 Hz, 1H), 4.08 (d, *J* = 12.4 Hz, 1H), 3.78 – 3.69 (m, 1H), 2.19 – 2.11 (m, 1H), 2.09 – 1.97 (m, 2H), 1.80 – 1.61 (m, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.2, 140.3, 131.5, 124.5, 88.0, 67.9, 30.8, 24.8, 21.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_9H_{13}N_2O_2$) requires *m/z* 181.0972, found *m/z* 181.0971.



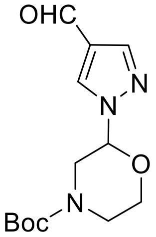
1-(1-methoxyethyl)-1H-pyrazole-4-carbaldehyde (3c)

Starting from 2-methoxy-1-propanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (10:1 → 5:1) to afford 21.26 mg (46% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 8.13 (s, 1H), 8.00 (s, 1H), 5.45 (q, *J* = 5.9 Hz, 1H), 3.28 (s, 3H), 1.69 (d, *J* = 6.0 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.2, 140.1, 129.9, 124.9, 89.9, 56.6, 21.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_7H_{11}N_2O_2$) requires *m/z* 155.0815, found *m/z* 155.0811.



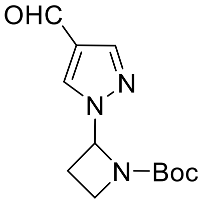
***tert*-butyl 2-(4-formyl-1H-pyrazol-1-yl)morpholine-4-carboxylate (4c)**

Starting from (*S*)-*N*-Boc-2-hydroxymethylmorpholine (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 50.60 mg (60% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.17 (dd, *J* = 20.6, 5.9 Hz, 1H), 8.02 (s, 1H), 6.29 – 6.05 (m, 1H), 4.64 (dd, *J* = 37.6, 12.4 Hz, 1H), 4.01 (dd, *J* = 12.7, 3.1 Hz, 1H), 3.79 – 3.64 (m, 3H), 3.23 – 2.88 (m, 1H), 1.49 – 1.40 (m, 9H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.2, 154.3, 140.7, 132.7&131.8, 82.4, 76.2&75.8, 67.7, 66.4&64.6, 63.2, 41.36&39.97, 28.13&28.07. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{13}H_{20}N_3O_4$) requires *m/z* 282.1448, found *m/z* 282.1449.



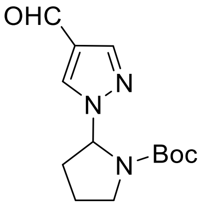
tert-butyl 2-(4-formyl-1H-pyrazol-1-yl)azetidine-1-carboxylate (5c)

Starting from _tert_-butyl 2-(hydroxymethyl)azetidine-1-carboxylate (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 36.16 mg (48% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.87 (s, 1H), 8.11 (s, 1H), 8.01 (s, 1H), 6.09 (dd, _J_ = 6.9, 3.8 Hz, 1H), 4.27 (dd, _J_ = 9.2, 5.7 Hz, 1H), 3.88 (dd, _J_ = 9.2, 3.3 Hz, 1H), 3.01 – 2.95 (m, 1H), 2.45 – 2.35 (m, 1H), 1.46 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.1, 155.2, 141.1, 132.0, 124.4, 89.8, 74.2, 51.0, 38.8, 28.3. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{12}H_{18}N_3O_3$) requires _m/z_ 252.1343, found _m/z_ 252.1342.



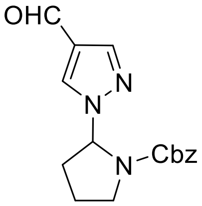
tert-butyl 2-(4-formyl-1H-pyrazol-1-yl)pyrrolidine-1-carboxylate (6c)

Starting from Boc-_L_-prolinol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 51.70 mg (65% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.85 (s, 1H), 8.08 (d, _J_ = 57.7 Hz, 1H), 7.98 (s, 1H), 6.09 – 5.92 (m, 1H), 3.78 – 3.60 (m, 1H), 3.58 – 3.40 (m, 1H), 2.48 – 2.14 (m, 3H), 2.08 – 1.97 (m, 1H), 1.46 – 1.36 (m, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.3&184.1, 154.3&153.2, 141.0&140.6, 133.2&131.4, 123.6, 81.2&81.0, 74.1&73.7, 47.0&46.6, 33.5&32.0, 28.3&28.3, 23.3&22.1. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{13}H_{20}N_3O_3$) requires _m/z_ 266.1499, found _m/z_ 266.1504.



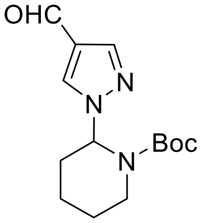
benzyl 2-(4-formyl-1H-pyrazol-1-yl)pyrrolidine-1-carboxylate (7c)

Starting from Cbz-*L*-prolinol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 52.05 mg (58% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.78 (d, *J* = 48.2 Hz, 1H), 7.98 (dd, *J* = 78.9, 72.1 Hz, 2H), 7.35 – 7.31 (m, 5H), 6.12 – 5.97 (m, 1H), 5.21 – 4.99 (m, 2H), 3.84 – 3.48 (m, 2H), 2.56 – 2.20 (m, 3H), 2.04 (d, *J* = 5.8 Hz, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.1&183.9, 154.8&153.7, 140.7&140.5, 135.9&135.5, 133.6&132.2, 128.5, 128.3&128.1, 128.2&127.9, 123.6&123.5, 73.8&73.5, 67.6&67.4, 47.0&46.7, 33.2&31.7, 23.3&22.1. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{16}H_{18}N_3O_3$) requires *m/z* 300.1343, found *m/z* 300.1348



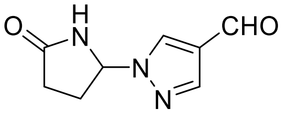
tert-butyl 2-(4-formyl-1H-pyrazol-1-yl)piperidine-1-carboxylate (8c)

Starting from *N*-Boc-piperidine-2-methanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 44.39 mg (53% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.87 (s, 1H), 7.99 (s, 2H), 6.33 (s, 1H), 4.05 (d, *J* = 13.6 Hz, 1H), 2.98 – 2.88 (m, 1H), 2.57 (d, *J* = 14.3 Hz, 1H), 1.99 – 1.90 (m, 1H), 1.76 – 1.73 (m, 2H), 1.63 – 1.52 (m, 2H), 1.47 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.2, 154.6, 140.3, 132.0, 124.0, 81.3, 67.3, 40.1, 28.2, 28.1, 24.1, 18.3. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{14}H_{22}N_3O_3$) requires *m/z* 280.1656, found *m/z* 280.1661.



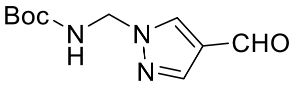
1-(5-oxopyrrolidin-2-yl)-1H-pyrazole-4-carbaldehyde (9c)

Starting from *L*-Pyroglutaminol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 31.70 mg (59% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.84 (s, 1H), 8.13 (s, 1H), 8.01 (s, 1H), 7.80 (s, 1H), 5.94 (d, *J* = 7.4 Hz, 1H), 2.77 – 2.65 (m, 2H), 2.49 – 2.36 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.2, 178.7, 141.7, 130.8, 124.4, 71.3, 28.24, 28.23. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_8H_{10}N_3O_2$) requires *m/z* 180.0768, found *m/z* 180.0770.



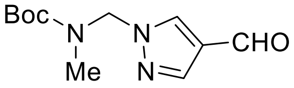
tert-butyl ((4-formyl-1H-pyrazol-1-yl)methyl)carbamate (10c)

Starting from *N*-Boc-(*R*)-1-amino-2-propanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 41.87 mg (62% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.87 (s, 1H), 8.22 (s, 1H), 7.98 (s, 1H), 6.28 (s, 1H), 5.43 (d, *J* = 7.0 Hz, 2H), 1.44 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.1, 155.3, 140.5, 134.1, 124.5, 81.1, 57.2, 28.1. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{10}H_{16}N_3O_3$) requires *m/z* 226.1186, found *m/z* 226.1186.



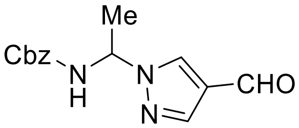
tert-butyl ((4-formyl-1H-pyrazol-1-yl)methyl)(methyl)carbamate (11c)

Starting from (2-hydroxyethyl) methylcarbamic acid 1,1-dimethylethyl ester (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 22.96 mg (32% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.88 (s, 1H), 8.23 – 7.92 (m, 2H), 5.53 (s, 2H), 3.01 (d, *J* = 7.5 Hz, 3H), 1.52 – 1.48 (m, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.1, 155.6&154.1, 140.8&140.1, 133.9&132.1, 124.9, 81.7&81.3, 64.9&64.4, 34.6&34.3, 28.3&28.2. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{11}H_{18}N_3O_3$) requires *m/z* 240.1343, found *m/z* 240.1348.



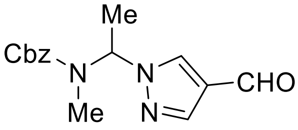
benzyl (1-(4-formyl-1H-pyrazol-1-yl)ethyl)carbamate (12c)

Starting from *N*-benzyloxycarbonyl-*L*-alaninol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 55.71 mg (68% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 (s, 1H), 8.17 (s, 1H), 7.99 (s, 1H), 7.36 – 7.24 (m, 5H), 6.37 (s, 1H), 6.08 – 5.95 (m, 1H), 5.13 (d, *J* = 12.1 Hz, 1H), 5.00 (d, *J* = 12.1 Hz, 1H), 1.76 (d, *J* = 6.4 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.3, 155.3, 140.8, 135.7, 133.1, 128.6, 128.5, 128.2, 123.7, 67.4, 64.7, 20.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{14}H_{16}N_3O_3$) requires *m/z* 274.1186, found *m/z* 274.1188.



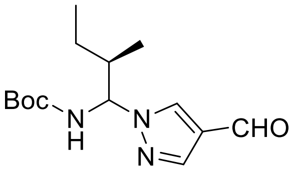
***tert*-butyl (1-(4-formyl-1H-pyrazol-1-yl)ethyl)(methyl)carbamate (13c)**

Starting from *tert*-butyl (1-hydroxypropan-2-yl)(methyl)carbamate (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 45.57 mg (60% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.84 (s, 1H), 8.14 (s, 1H), 7.97 (s, 1H), 7.35 (s, 5H), 6.51 (s, 1H), 5.22 – 5.11 (m, 2H), 2.91 (s, 3H), 1.82 (d, *J* = 6.8 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 156.1, 140.1, 135.8, 133.1, 128.5, 128.2, 127.9, 124.0, 67.8, 67.3, 28.2, 16.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{12}H_{20}N_3O_3$) requires *m/z* 254.1199, found *m/z* 254.1200.



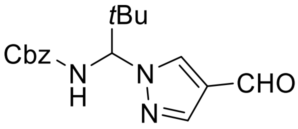
benzyl ((2*R*)-1-(4-formyl-1H-pyrazol-1-yl)-2-methylbutyl)carbamate (14c)

Starting from *N*-Boc-(2*S*,3*S*)-(-)-2-amino-3-methyl-1-pentanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 53.14 mg (63% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.13 (s, 1H), 8.04 (s, 1H), 6.21 (dd, *J* = 14.9, 9.6 Hz, 1H), 5.63 – 5.44 (m, 1H), 2.23 (s, 1H), 1.74 – 1.62 (m, 1H), 1.40 (s, 9H), 1.34 – 1.22 (m, 1H), 1.04 (d, *J* = 6.7 Hz, 2H), 0.94 (t, *J* = 7.5 Hz, 2H), 0.86 (t, *J* = 7.2 Hz, 1H), 0.69 (d, *J* = 6.6 Hz, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 154.9, 140.8&140.7, 133.7&133.4, 123.2&123.1, 80.6&80.5, 72.6&72.4, 38.3&38.1, 28.13&28.08, 24.90&24.85, 14.7&14.5, 10.7&10.6. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{14}H_{24}N_3O_3$) requires *m/z* 282.1812, found *m/z* 282.1809.



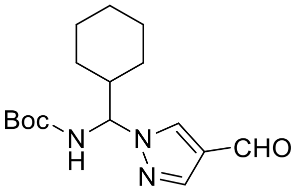
benzyl (1-(4-formyl-1H-pyrazol-1-yl)-2,2-dimethylpropyl)carbamate (15c)

Starting from *N*-carbobenzoxy-*L*-tert-leucinol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 52.95 mg (56% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.85 (s, 1H), 8.02 (s, 1H), 7.98 (s, 1H), 7.33 – 7.32 (m, 5H), 6.03 (d, *J* = 7.4 Hz, 1H), 5.62 (d, *J* = 9.9 Hz, 1H), 5.12 (d, *J* = 12.2 Hz, 1H), 5.04 (d, *J* = 12.1 Hz, 1H), 1.01 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 155.7, 140.4, 135.5, 133.7, 128.6, 128.4, 128.2, 123.2, 75.6, 67.5, 36.1, 25.8. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{22}N_3O_3$) requires *m/z* 316.1656, found *m/z* 316.1658.



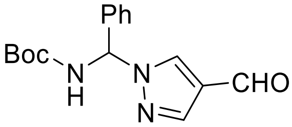
***tert*-butyl (cyclohexyl(4-formyl-1H-pyrazol-1-yl)methyl)carbamate (16c)**

Starting from *tert*-butyl-(*S*)-(1-cyclohexyl-2-hydroxyethyl)carbamate (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 52.53 mg (57% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.86 (s, 1H), 8.05 (d, *J* = 15.6 Hz, 2H), 5.86 (d, *J* = 9.2 Hz, 1H), 5.47 (t, *J* = 9.5 Hz, 1H), 2.13 – 2.03 (m, 1H), 1.94 (d, *J* = 12.2 Hz, 1H), 1.80 (d, *J* = 11.2 Hz, 1H), 1.67 (d, *J* = 7.5 Hz, 2H), 1.40 (s, 9H), 1.27 – 1.06 (m, 6H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.3, 155.1, 141.1, 133.9, 123.3, 80.8, 72.9, 41.3, 29.1, 28.8, 28.3, 26.0, 25.5, 25.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{16}H_{26}N_3O_3$) requires *m/z* 308.1969, found *m/z* 308.1970.



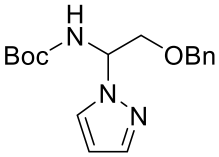
***tert*-butyl ((4-formyl-1H-pyrazol-1-yl)(phenyl)methyl)carbamate (17c)**

Starting from Boc-*L*-phenylglycinol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 45.17 mg (50% yield) as a white solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ 9.83 (s, 1H), 8.71 (d, *J* = 8.3 Hz, 1H), 8.55 (s, 1H), 8.04 (s, 1H), 7.36 (t, *J* = 6.3 Hz, 3H), 7.30 (d, *J* = 7.8 Hz, 2H), 7.02 (d, *J* = 9.1 Hz, 1H), 1.40 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, DMSO-d_6) δ 185.6, 155.2, 140.5, 137.8, 133.6, 129.2, 129.0, 127.2, 124.3, 80.0, 70.7, 28.5. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{16}H_{20}N_3O_3$) requires *m/z* 302.1499, found *m/z* 302.1495.



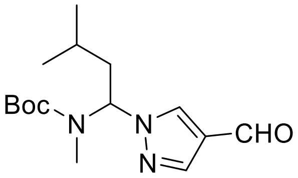
tert-butyl (2-(benzyloxy)-1-(1H-pyrazol-1-yl)ethyl)carbamate (18c)

Starting from *N*-Boc-(*S*)-2-amino-3-benzyloxy-1-propanol (0.3 mmol) with 1H-pyrazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 40.92 mg (43% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.56 (s, 1H), 7.34 – 7.27 (m, 3H), 7.21 (d, *J* = 6.8 Hz, 2H), 6.26 (d, *J* = 1.8 Hz, 1H), 5.99 (s, 1H), 5.77 (s, 1H), 4.45 (s, 2H), 3.97 – 3.84 (m, 2H), 1.41 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 154.7, 140.1, 137.5, 129.3, 128.6, 128.0, 127.8, 105.35, 80.8, 73.5, 70.6, 66.4, 28.3. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{23}N_3O_3$) requires *m/z* 317.1739, found *m/z* 317.1736.



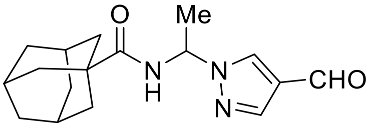
***tert*-butyl (1-(4-formyl-1H-pyrazol-1-yl)-3-methylbutyl)(methyl)carbamate (19c)**

Starting from tert-butyl-(S)-(1-hydroxy-4-methylpentan-2-yl)(methyl)carbamate (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (15:1 → 10:1) to afford 30.09 mg (34% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.12 (s, 1H), 7.98 (s, 1H), 6.37 (d, *J* = 63.1 Hz, 1H), 2.83 (s, 3H), 2.13 – 2.06 (m, 2H), 1.48 (s, 10H), 1.00 (d, *J* = 6.7 Hz, 3H), 0.96 (d, *J* = 6.5 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.2, 155.6, 140.0, 133.7, 123.9, 81.0, 69.0, 39.0, 28.3, 24.4, 22.5, 22.1. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{15}H_{26}N_3O_3$) requires *m/z* 296.1969, found *m/z* 296.1970.



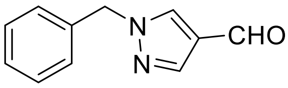
(3*R*,5*R*,7*R*)-*N*-(1-(4-formyl-1H-pyrazol-1-yl)ethyl)adamantane-1-carboxamide (20c)

Starting from (3*R*,5*R*,7*R*)-*N*-(1-hydroxypropan-2-yl)adamantane-1-carboxamide (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 57.83 mg (64% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.84 (s, 1H), 8.15 (s, 1H), 7.98 (s, 1H), 6.56 (d, *J* = 8.4 Hz, 1H), 6.31 – 6.21 (m, 1H), 2.03 (s, 3H), 1.79 (s, 8H), 1.75 – 1.65 (m, 7H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 177.9, 140.4, 133.5, 123.5, 62.0, 40.5, 38.8, 36.2, 27.8, 20.1. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{24}N_3O_2$) requires *m/z* 302.1863, found *m/z* 302.1866.



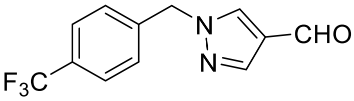
1-benzyl-1H-pyrazole-4-carbaldehyde (21c)

Starting from phenethyl alcohol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 25.12 mg (45% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 8.00 (s, 1H), 7.88 (s, 1H), 7.40 – 7.35 (m, 3H), 7.29 – 7.26 (m, 2H), 5.34 (s, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 141.1, 134.8, 132.6, 129.2, 128.8, 128.2, 124.7, 56.7. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{11}H_{11}N_2O$) requires *m/z* 187.0866, found *m/z* 187.0870.



1-(4-(trifluoromethyl)benzyl)-1H-pyrazole-4-carbaldehyde (22c)

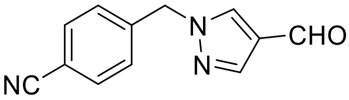
Starting from 2-(4-(trifluoromethyl)phenyl)ethanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv..) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 19.05 mg (25% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.03 (s, 1H), 7.95 (s, 1H), 7.65 (d, J = 8.1 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 5.41 (s, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.1, 141.5, 139.0, 132.9, 129.3, 128.3, 128.1 (q, J = 24.5 Hz) 126.3 (q, J = 245.8 Hz), 126.3 (q, J = 3.6 Hz), 56.1. (see spectrum)

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.71. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{12}H_{10}F_3N_2O$) requires *m/z* 255.0740, found *m/z* 255.0748.



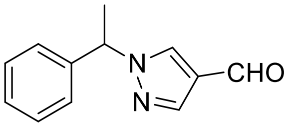
4-((4-formyl-1H-pyrazol-1-yl)methyl)benzonitrile (23c)

Starting from β-methylphenethyl alcohol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 20.26 mg (32% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.88 (s, 1H), 8.04 (s, 1H), 7.98 (s, 1H), 7.68 (d, *J* = 8.2 Hz, 3H), 7.34 (d, *J* = 8.1 Hz, 2H), 5.41 (s, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.0, 141.7, 140.3, 133.0, 129.8, 128.5, 125.1, 118.3, 112.8, 56.0. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{12}H_{10}N_3O$) requires *m/z* 212.0818, found *m/z* 212.0822.



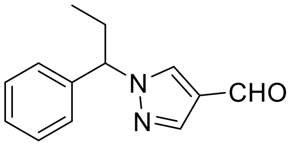
1-(1-phenylethyl)-1H-pyrazole-4-carbaldehyde (24c)

Starting from β-methylphenethyl alcohol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 25.21 mg (42% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.83 (s, 1H), 8.00 (s, 1H), 7.91 (s, 1H), 7.41 – 7.33 (m, 3H), 7.26 (d, *J* = 6.1 Hz, 2H), 5.55 (q, *J* = 7.1 Hz, 1H), 1.93 (d, *J* = 7.1 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.2, 140.8, 140.1, 131.3, 129.1, 128.6, 126.6, 124.2, 62.0, 21.2. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{12}H_{13}N_2O$) requires *m/z* 201.1022, found *m/z* 201.1020.



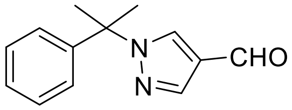
1-(1-phenylpropyl)-1H-pyrazole-4-carbaldehyde(25c)

Starting from 2-phenylbutan-1-ol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 17.99 mg (28% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.83 (s, 1H), 8.00 (s, 1H), 7.96 (s, 1H), 7.36 (dd, *J* = 13.2, 6.0 Hz, 3H), 7.31 (d, *J* = 8.4 Hz, 2H), 5.20 (t, *J* = 7.7 Hz, 1H), 2.54 – 2.41 (m, 1H), 2.28 – 2.17 (m, 1H), 0.93 (t, *J* = 7.3 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.2, 140.9, 139.0, 131.7, 129.0, 128.5, 127.0, 124.1, 68.8, 28.3, 11.1. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{13}H_{15}N_2O$) requires *m/z* 215.1179, found *m/z* 215.1184.



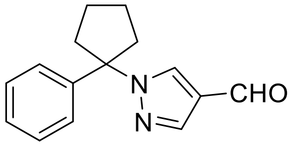
1-(2-phenylpropan-2-yl)-1H-pyrazole-4-carbaldehyde (26c)

Starting from 2-methyl-2-phenylpropan-1-ol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 31.47 mg (49% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.85 (s, 1H), 8.03 (s, 1H), 7.99 (s, 1H), 7.36 – 7.28 (m, 3H), 7.13 – 7.10 (m, 2H), 2.00 (s, 6H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.5, 144.8, 140.6, 131.4, 128.9, 128.0, 125.2, 124.0, 64.9, 29.6. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{13}H_{15}N_2O$) requires *m/z* 215.1179, found *m/z* 215.1187.



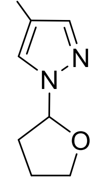
1-(1-phenylcyclopentyl)-1H-pyrazole-4-carbaldehyde (27c)

Starting from (1-Phenylcyclopentyl)methanol (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (10:1 → 5:1) to afford 36.02 mg (50% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 1H), 8.02 (s, 1H), 7.98 (s, 1H), 7.35 – 7.18 (m, 5H), 2.91 – 2.82 (m, 2H), 2.42 – 2.34 (m, 2H), 1.95 – 1.84 (m, 2H), 1.82 – 1.71 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.3, 143.0, 140.4, 131.4, 128.6, 127.8, 125.7, 124.2, 75.9, 38.7, 22.6. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{15}H_{17}N_2O$) requires *m/z* 241.1335, found *m/z* 241.1331.



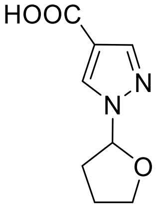
1-(1-(tetrahydrofuran-2-yl)-1H-pyrazol-4-yl)ethan-1-one (28c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1-(1H-pyrazol-4-yl)ethan-1-one (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 36.74 mg (68% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.94 (s, 1H), 5.99 (dd, J = 6.6, 2.2 Hz, 1H), 4.21 – 4.15 (m, 1H), 4.03 (q, J = 7.7 Hz, 1H), 2.61 – 2.54 (m, 1H), 2.43 (s, 3H), 2.41 – 2.31 (m, 1H), 2.15 – 2.02 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 192.3, 140.8, 130.4, 124.0, 90.7, 69.8, 32.2, 27.9, 23.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_9H_{13}N_2O_2$) requires *m/z* 181.0972, found *m/z* 181.0974.



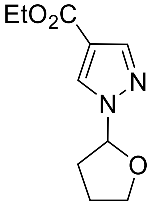
1-(tetrahydrofuran-2-yl)-1H-pyrazole-4-carboxylic acid (29c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-pyrazole-4-carboxylic acid (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 31.68 mg (58% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (s, 1H), 8.00 (s, 1H), 6.00 (dd, J = 6.5, 1.6 Hz, 1H), 4.21 – 4.15 (m, 1H), 4.03 (q, J = 7.7 Hz, 1H), 2.62 – 2.53 (m, 1H), 2.41 – 2.32 (m, 1H), 2.14 – 2.04 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.4, 142.1, 132.1, 113.9, 90.8, 69.8, 32.3, 23.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_8H_{11}N_2O_3$) requires *m/z* 183.9764, found *m/z* 183.9766.



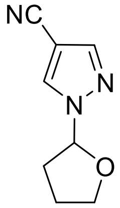
ethyl 1-(tetrahydrofuran-2-yl)-1H-pyrazole-4-carboxylate (30c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with ethyl 1H-pyrazole-4-carboxylate (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 41.60 mg (66% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.06 (s, 1H), 7.93 (s, 1H), 5.98 (dd, *J* = 6.5, 1.7 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 4.20 – 4.14 (m, 1H), 4.02 (q, *J* = 7.5 Hz, 1H), 2.61 – 2.51 (m, 1H), 2.41 – 2.28 (m, 1H), 2.16 – 1.99 (m, 2H), 1.34 (t, *J* = 7.1 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 163.1, 141.4, 131.2, 115.1, 90.6, 69.7, 60.2, 32.1, 23.9, 14.4. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{10}H_{15}N_2O_3$) requires *m/z* 211.1077, found *m/z* 211.1072.



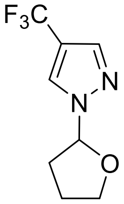
1-(tetrahydrofuran-2-yl)-1H-pyrazole-4-carbonitrile (31c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-pyrazole-4-carbonitrile (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 35.22 mg (72% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.00 (s, 1H), 7.82 (s, 1H), 5.99 (dd, *J* = 6.5, 1.9 Hz, 1H), 4.20 – 4.13 (m, 1H), 4.04 (q, *J* = 7.7 Hz, 1H), 2.62 – 2.54 (m, 1H), 2.43 – 2.31 (m, 1H), 2.12 – 2.02 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 142.6, 133.0, 113.5, 92.2, 91.1, 70.0, 32.3, 23.7. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_8H_{10}N_3O$) requires *m/z* 164.0818, found *m/z* 164.0816.



1-(tetrahydrofuran-2-yl)-4-(trifluoromethyl)-1H-pyrazole (32c)

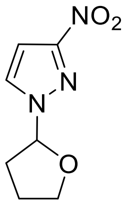
Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 4-(trifluoromethyl)-1H-pyrazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 48.22 mg (78% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.86 (s, 1H), 7.73 (s, 1H), 5.98 (d, *J* = 6.4 Hz, 1H), 4.19 – 4.13 (m, 1H), 4.02 (q, *J* = 7.7 Hz, 1H), 2.62 – 2.56 (m, 1H), 2.40 – 2.31 (m, 1H), 2.16 – 2.02 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.3 (q, *J* = 2.5 Hz), 127.2 (q, *J* = 3.0 Hz), 122.7 (q, *J* = 267.0 Hz), 113.5 (q, *J* = 38.0 Hz), 90.7, 69.7, 32.1, 23.9. (see spectrum)

^{19}F NMR (376 MHz, $CDCl_3$) δ -56.46. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_8H_{10}F_3N_2O$) requires *m/z* 207.0740, found *m/z* 207.0739.



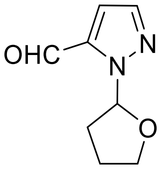
3-nitro-1-(tetrahydrofuran-2-yl)-1H-pyrazole (33c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 3-nitro-1H-pyrazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 28.01 mg (51% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 2.5 Hz, 1H), 6.91 (d, *J* = 2.5 Hz, 1H), 6.03 (dd, *J* = 6.4, 1.7 Hz, 1H), 4.22 – 4.17 (m 1H), 4.06 (q, *J* = 7.7 Hz, 1H), 2.69 – 2.61 (m, 1H), 2.46 – 2.35 (m, 1H), 2.12 – 2.03 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 156.1, 130.1, 102.7, 91.9, 70.2, 32.6, 23.7. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_7H_{10}N_3O_3$) requires *m/z* 184.0717, found *m/z* 184.0714.



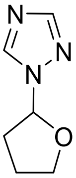
1-(tetrahydrofuran-2-yl)-1H-pyrazole-5-carbaldehyde (34c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-pyrazole-5-carbaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 23.91 mg (48% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 – 9.96 (m, 1H), 7.63 (d, *J* = 2.4 Hz, 1H), 6.81 (d, *J* = 2.5 Hz, 1H), 6.05 (dd, *J* = 6.6, 2.3 Hz, 1H), 4.16 (td, *J* = 8.1, 5.4 Hz, 1H), 4.05 (dd, *J* = 15.5, 7.4 Hz, 1H), 2.68 – 2.60 (m, 1H), 2.44 – 2.35 (m, 1H), 2.22 – 2.03 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 186.8, 151.7, 129.7, 105.7, 90.9, 69.7, 32.1, 24.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_8H_{11}N_2O_2$) requires *m/z* 167.0815, found *m/z* 167.0820.



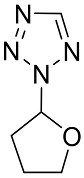
1-(tetrahydrofuran-2-yl)-1H-1,2,4-triazole (35c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-1,2,4-triazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 22.53 mg (54% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.24 (s, 1H), 7.96 (s, 1H), 6.08 (d, J = 6.0 Hz, 1H), 4.18 (dd, J = 13.2, 7.7 Hz, 1H), 4.04 (q, J = 7.6 Hz, 1H), 2.62 – 2.52 (m, 1H), 2.45 – 2.32 (m, 1H), 2.22 – 2.02 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 152.2, 142.2, 88.9, 69.8, 32.1, 23.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_6H_{10}N_3O$) requires *m/z* 140.0818, found *m/z* 140.0819.



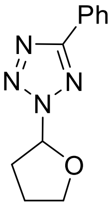
2-(tetrahydrofuran-2-yl)-2H-tetrazole (36c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 2H-tetrazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 28.99 mg (69% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.73 (s, 1H), 6.31 (d, J = 6.4 Hz, 1H), 4.21 – 4.07 (m, 2H), 2.79 – 2.70 (m, 1H), 2.56 – 2.46 (m, 1H), 2.21 – 2.07 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 141.5, 89.1, 70.2, 32.6, 23.5. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_5H_9N_4O$) requires *m/z* 141.0771, found *m/z* 141.0774.



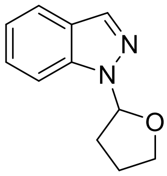
5-phenyl-2-(tetrahydrofuran-2-yl)-2H-tetrazole (37c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 5-phenyl-2H-tetrazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 49.27 mg (76% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 – 8.14 (m, 2H), 7.51 – 7.43 (m, 3H), 6.57 (dd, *J* = 6.5, 2.1 Hz, 1H), 4.30 – 4.23 (m, 1H), 4.16 – 4.09 (m, 1H), 2.73 – 2.64 (m, 1H), 2.56 – 2.44 (m, 2H), 2.21 – 2.10 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.0, 130.3, 128.8, 127.3, 126.8, 91.6, 70.1, 31.7, 23.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{11}H_{13}N_4O$) requires *m/z* 217.1084, found *m/z* 217.1080.



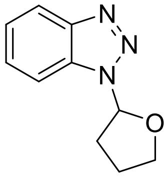
1-(tetrahydrofuran-2-yl)-1H-indazole (38c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-indazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 23.70 mg (42% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 1H), 7.71 (d, *J* = 8.7 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.32 – 7.24 (m, 1H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.32 – 6.25 (m, 1H), 4.30 – 4.25 (m, 1H), 4.14 – 4.05 (m, 1H), 2.78 – 2.67 (m, 1H), 2.51 – 2.41 (m, 1H), 2.15 – 2.04 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 149.1, 126.2, 121.7, 121.4, 121.1, 120.5, 117.7, 91.7, 70.0, 33.0, 24.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{11}H_{13}N_2O$) requires *m/z* 189.1022, found *m/z* 189.1025.



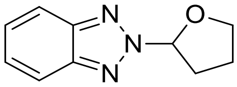
1-(tetrahydrofuran-2-yl)-1H-benzo[d][1,2,3]triazole (39c-1)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-benzo[d][1,2,3]triazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 20.99 mg (37% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.06 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 8.3 Hz, 1H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 6.51 (dd, *J* = 6.8, 2.4 Hz, 1H), 4.14 – 4.00 (m, 2H), 3.21 – 3.11 (m, 1H), 2.57 – 2.46 (m, 1H), 2.46 – 2.33 (m, 1H), 2.24 – 2.12 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 146.4, 132.8, 127.5, 124.1, 119.9, 110.4, 87.9, 69.3, 30.8, 24.4. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{10}H_{12}N_3O$) requires *m/z* 190.0975, found *m/z* 190.0973



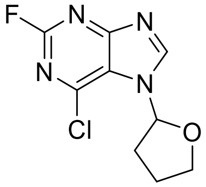
2-(tetrahydrofuran-2-yl)-2H-benzo[d][1,2,3]triazole (39c-2)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1H-benzo[d][1,2,3]triazole (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 11.35 mg (20% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.88 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.39 (dd, *J* = 6.6, 3.1 Hz, 2H), 6.60 (dd, *J* = 6.4, 2.2 Hz, 1H), 4.38 – 4.30 (m, 1H), 4.15 (q, *J* = 7.8 Hz, 1H), 2.80 – 2.70 (m, 1H), 2.59 – 2.46 (m, 2H), 2.23 – 2.09 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 144.3, 126.6, 118.5, 94.2, 70.3, 32.4, 24.3. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{10}H_{12}N_3O$) requires *m/z* 190.0975, found *m/z* 190.0978.



6-chloro-2-fluoro-7-(tetrahydrofuran-2-yl)-7H-purine (40c)

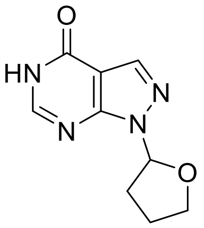
Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 6-chloro-2-fluoro-7H-purine (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 32.68 mg (45% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.22 (s, 1H), 6.28 (dd, *J* = 5.7, 3.8 Hz, 1H), 4.35 – 4.28 (m, 1H), 4.10 (q, *J* = 7.4 Hz, 1H), 2.58 – 2.53 (m, 2H), 2.19 (dd, *J* = 13.4, 6.4 Hz, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 156.9 (d, *J* = 220.8 Hz), 152.6 (d, *J* = 7.3 Hz), 152.4 (d, *J* = 6.7 Hz), 144.1 (d, *J* = 3.1 Hz), 130.9 (d, *J* = 5.0 Hz), 86.7, 70.0, 32.4, 24.1. (see spectrum)

^{19}F NMR (376 MHz, CDCl$_3$) δ -49.31. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_9H_9ClFN_4O$) requires *m/z* 243.0443, found *m/z* 243.0441.



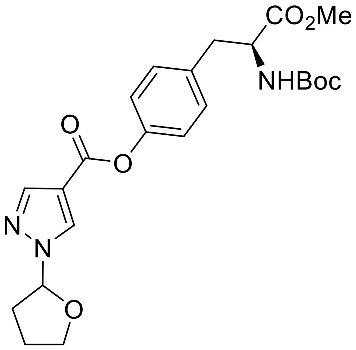
1-(tetrahydrofuran-2-yl)-1,5-dihydro-4H-pyrazolo[3,4-d]pyrimidin-4-one (41c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 1,5-dihydro-4H-pyrazolo[3,4-d]pyrimidin-4-one (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (1:1 →EA) to afford 51.93 mg (84% yield) as a white solid.

^{1}H NMR (400 MHz, DMSO-d_6) δ 11.76 (s, 1H), 8.63 (s, 1H), 7.97 (d, *J* = 3.5 Hz, 1H), 6.19 (dd, *J* = 6.0, 3.1 Hz, 1H), 4.18 – 4.13 (m, 1H), 3.94 (q, *J* = 7.4 Hz, 1H), 2.43 – 2.33 (m, 2H), 2.21 – 2.10 (m, 1H), 2.05 – 1.93 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, DMSO-d_6) δ 159.5, 159.2, 147.7, 128.1, 107.3, 91.3, 69.9, 32.3, 24.3. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_9H_{11}N_4O_2$) requires *m/z* 207.0877, found *m/z* 207.0876.



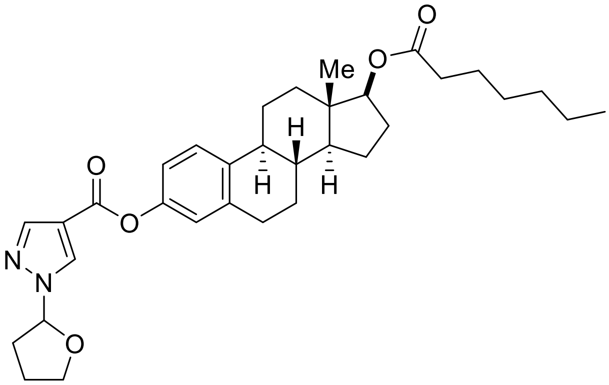
4-((*S*)-2-((tert-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)phenyl 1-(tetrahydrofuran-2-yl)-1H-pyrazole-4-carboxylate (42c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with (*S*)-4-(2-((tert-butoxycarbonyl)amino)-3-methoxy-3-oxopropyl)phenyl 1H-pyrazole-4-carboxylate (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 63.37 mg (46 % yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (s, 1H), 8.07 (s, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 8.5 Hz, 2H), 6.02 (dd, *J* = 6.5, 2.1 Hz, 1H), 5.10 (d, *J* = 8.1 Hz, 1H), 4.59 (dd, *J* = 13.4, 6.1 Hz, 1H), 4.20 (td, *J* = 8.0, 5.4 Hz, 1H), 4.04 (q, *J* = 7.6 Hz, 1H), 3.72 (s, 3H), 3.16 – 3.04 (m, 2H), 2.64 – 2.54 (m, 1H), 2.43 – 2.31 (m, 1H), 2.18 – 2.02 (m, 2H), 1.43 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 161.1, 155.0, 149.4, 141.8, 133.5, 131.9, 130.1, 121.7, 113.8, 90.6, 79.9, 69.7, 54.3, 52.1, 37.5, 32.1, 28.2, 23.8. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{23}H_{30}N_3O_7$) requires *m/z* 460.2079, found *m/z* 460.2081.



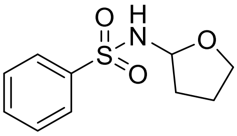
(8*R*,9*S*,13*S*,14*S*,17*S*)-17-(heptanoyloxy)-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl 1-(tetrahydrofuran-2-yl)-1H-pyrazole-4-carboxylate (43c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with (8*R*,9*S*,13*S*,14*S*,17*S*)-17-(heptanoyloxy)-13-methyl-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl 1H-pyrazole-4-carboxylate (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 70.73 mg (43 % yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.07 (s, 1H), 7.30 (d, *J* = 8.5 Hz, 1H), 6.92 (dd, *J* = 8.4, 2.5 Hz, 1H), 6.88 (d, *J* = 2.3 Hz, 1H), 6.02 (dd, *J* = 6.5, 2.2 Hz, 1H), 4.74 – 4.67 (m, 1H), 4.20 (td, *J* = 8.0, 5.3 Hz, 1H), 4.04 (q, *J* = 7.5 Hz, 1H), 2.88 (dd, *J* = 10.0, 4.9 Hz, 2H), 2.64 – 2.55 (m, 1H), 2.42 – 2.35 (m, 1H), 2.35 – 2.19 (m, 5H), 2.17 – 2.02 (m, 2H), 1.89 (d, *J* = 12.7 Hz, 2H), 1.80 – 1.69 (m, 1H), 1.68 – 1.58 (m, 2H), 1.57 – 1.29 (m, 13H), 0.89 (t, *J* = 6.9 Hz, 3H), 0.84 (s, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 173.9, 161.5, 148.2, 141.9, 138.1, 137.8, 131.8, 126.4, 121.6, 118.7, 114.1, 90.7, 82.3, 69.7, 49.8, 44.0, 42.9, 38.2, 36.9, 34.6, 32.2, 31.4, 29.5, 28.8, 27.6, 27.0, 26.0, 25.1, 23.8, 23.2, 22.5, 14.0, 12.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{33}H_{45}N_2O_5$) requires *m/z* 549.3323, found *m/z* 549.3325.



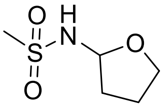
N-(tetrahydrofuran-2-yl)benzenesulfonamide (44c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with benzenesulfonamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 34.74 mg (51 % yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.94 – 7.90 (m, 2H), 7.57 (t, J = 7.3 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 2H), 5.41 – 5.37 (m, 1H), 3.69 (t, *J* = 6.9 Hz, 2H), 2.22 – 2.14 (m, 1H), 1.94 – 1.83 (m, 3H), 1.82 – 1.74 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 141.4, 132.6, 128.9, 127.1, 85.0, 67.3, 32.7, 24.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{10}H_{14}NO_3S$) requires *m/z* 228.0689, found *m/z* 228.0690.



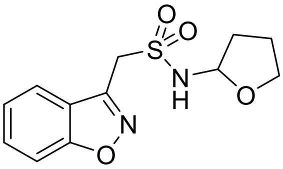
N-(tetrahydrofuran-2-yl)methanesulfonamide (45c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with methanesulfonamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 34.66 mg (70 % yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 5.73 (d, *J* = 9.4 Hz, 1H), 5.39 – 5.31 (m, 1H), 3.93 (q, *J* = 7.2 Hz, 1H), 3.87 – 3.82 (m, 1H), 3.08 (s, 3H), 2.29 – 2.19 (m, 1H), 2.02 – 1.85 (m, 2H), 1.83 – 1.74 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 84.9, 67.3, 42.8, 32.1, 24.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_5H_{12}NO_3S$) requires *m/z* 166.0532, found *m/z* 166.0536.



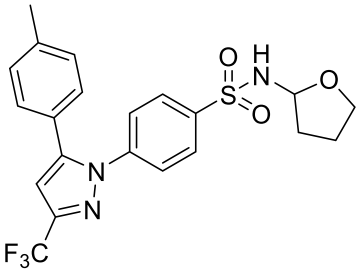
1-(benzo[d]isoxazol-3-yl)-N-(tetrahydrofuran-2-yl)methanesulfonamide (46c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with benzo[d]isoxazole-3-sulfonamide (1.5 equiv..) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 42.31 mg (50 % yield) as a colorless oil.

^{1}H NMR (400 MHz, Acetone-d_6) δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.70 – 7.64 (m, 2H), 7.42 (ddd, *J* = 8.0, 5.9, 2.0 Hz, 1H), 5.42 (td, *J* = 6.7, 3.4 Hz, 1H), 4.93 (dd, *J* = 34.1, 14.1 Hz, 2H), 4.10 (dd, *J* = 14.8, 7.6 Hz, 1H), 3.91 – 3.82 (m, 1H), 2.93 – 2.90 (m, 1H), 2.29 – 2.17 (m, 1H), 2.12 – 2.01 (m, 1H), 1.96 – 1.85 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, Acetone-d_6) δ 163.4, 150.5, 130.3, 123.8, 123.3, 121.4, 109.4, 85.2, 66.9, 51.0, 31.3, 24.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_2O_4S$) requires *m/z* 283.0747, found *m/z* 283.0750.



***N*-(tetrahydrofuran-2-yl)-4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (47c)**

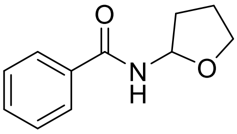
Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 4-(5-(p-tolyl)-3-(trifluoromethyl)-1H-pyrazol-1-yl)benzenesulfonamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 56.84 mg (42 % yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.7 Hz, 2H), 7.44 (d, *J* = 8.7 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 6.74 (s, 1H), 5.44 (d, *J* = 9.1 Hz, 1H), 5.40 – 5.34 (m, 1H), 3.71 – 3.61 (m, 2H), 2.37 (s, 3H), 2.23 – 2.13 (m, 1H), 1.92 – 1.83 (m, 2H), 1.81 – 1.68 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.2, 144.0 (q, *J* = 38.4 Hz), 142.3, 140.9, 139.7, 129.7, 128.7, 128.2, 125.7, 125.3, 121.0 (q, *J* = 269.7 Hz), 106.2, 85.0, 67.2, 32.6, 24.0, 21.3. (see spectrum)

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.39. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{21}H_{21}F_3N_3O_3S$) requires *m/z* 452.1250, found *m/z* 452.1249.



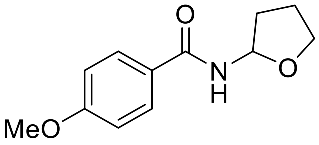
***N*-(tetrahydrofuran-2-yl)benzamide (48c)**

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with benzamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 24.08 mg (42 % yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 6.64 (s, 1H), 5.91 (dd, *J* = 11.6, 6.9 Hz, 1H), 3.99 (dd, *J* = 14.8, 6.8 Hz, 1H), 3.85 (dd, *J* = 14.7, 7.3 Hz, 1H), 2.35 – 2.22 (m, 1H), 2.06 – 1.95 (m, 2H), 1.92 – 1.85 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 167.3, 134.1, 131.7, 128.5, 127.0, 81.8, 67.6, 32.2, 24.6. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{11}H_{14}NO_2$) requires *m/z* 192.1019, found *m/z* 192.1017.



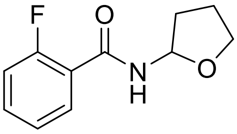
4-methoxy-*N*-(tetrahydrofuran-2-yl)benzamide (49c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 4-methoxybenzamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 23.88 mg (36 % yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.8 Hz, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.44 (d, *J* = 6.8 Hz, 1H), 5.90 (q, *J* = 6.7 Hz, 1H), 4.00 (q, *J* = 6.7 Hz, 1H), 3.89 – 3.82 (m, 4H), 2.33 – 2.24 (m, 1H), 2.05 – 1.96 (m, 2H), 1.91 – 1.83 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3, 129.1, 128.9, 126.3, 113.7, 81.8, 67.6, 55.4, 32.3, 24.7. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{12}H_{16}NO_3$) requires *m/z* 222.1125, found *m/z* 222.1122.



2-fluoro-*N*-(tetrahydrofuran-2-yl)benzamide (50c)

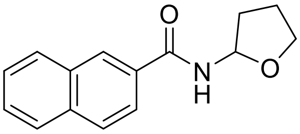
Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 2-fluorobenzamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 28.85 mg (46 % yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.09 (td, *J* = 7.9, 1.8 Hz, 1H), 7.51 – 7.44 (m, 1H), 7.28 – 7.23 (m, 1H), 7.14 – 7.07 (m, 1H), 7.05 (s, 1H), 5.98 – 5.91 (m, 1H), 4.05 – 3.97 (m, 1H), 3.91 – 3.84 (m, 1H), 2.34 – 2.25 (m, 1H), 2.05 – 1.97 (m, 2H), 1.94 – 1.85 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 162.9 (d, *J* = 3.1 Hz, 1H), 161.6 (d, *J* = 248.4 Hz, 1H), 133.5 (d, *J* = 9.4 Hz, 1H), 132.1 (d, *J* = 1.7 Hz, 1H), 124.8 (d, *J* = 3.2 Hz, 1H), 120.7 (d, *J* = 11.2 Hz, 1H), 115.9 (d, *J* = 24.9 Hz, 1H), 81.6, 67.7, 32.3, 24.6. (see spectrum)

^{19}F NMR (376 MHz, CDCl$_3$) δ -113.46. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{11}H_{13}FNO_2$) requires *m/z* 210.0925, found *m/z* 210.0923.



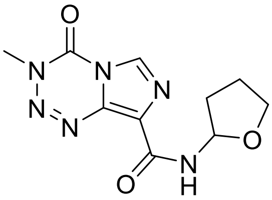
N-(tetrahydrofuran-2-yl)-2-naphthamide (51c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 2-naphthamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 26.76 mg (37 % yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.28 (s, 1H), 7.93 – 7.81 (m, 4H), 7.59 – 7.51 (m, 2H), 6.66 (d, *J* = 6.4 Hz, 1H), 5.98 (q, *J* = 6.7 Hz, 1H), 4.07 – 4.01 (m, 1H), 3.92 – 3.85 (m, 1H), 2.38 – 2.28 (m, 1H), 2.10 – 1.99 (m, 2H), 1.99 – 1.89 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 167.3, 134.8, 132.5, 131.3, 128.9, 128.4, 127.7, 127.6, 126.8, 123.6, 81.9, 67.8, 32.4, 24.7. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{15}H_{16}NO_2$) requires *m/z* 242.1176, found *m/z* 242.1181.



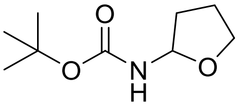
3-methyl-4-oxo-N-(tetrahydrofuran-2-yl)-3,4-dihydroimidazo[5,1-d][1,2,3,5]tetrazine-8-carboxamide (52c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 3-methyl-4-oxo-3,4-dihydroimidazo[5,1-d][1,2,3,5]tetrazine-8-carboxamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 28.52 mg (36 % yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.39 (s, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 6.02 – 5.97 (m, 1H), 4.07 – 4.00 (m, 4H), 3.89 (dd, *J* = 14.5, 7.3 Hz, 1H), 2.36 – 2.27 (m, 1H), 2.12 – 1.91 (m, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 159.0, 138.7, 134.1, 131.5, 127.7, 81.0, 67.8, 36.6, 32.3, 24.7. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{10}H_{13}N_6O_3$) requires *m/z* 265.1044, found *m/z* 265.1049.



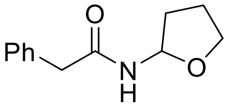
tert-butyl (tetrahydrofuran-2-yl)carbamate (53c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with tert-butyl carbamate (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 35.93 mg (64 % yield) as a colorless oil.

1H NMR (400 MHz, CDCl$_3$) δ 5.50 (s, 1H), 5.10 (d, *J* = 7.3 Hz, 1H), 3.90 (q, *J* = 7.3 Hz, 1H), 3.80 (q, *J* = 7.3 Hz, 1H), 2.21 – 2.12 (m, 1H), 1.97 – 1.87 (m, 2H), 1.71 – 1.60 (m, 1H), 1.46 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 154.8, 82.4, 79.9, 66.8, 31.6, 28.3, 24.6. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_9H_{18}NO_3$) requires *m/z* 188.1281, found *m/z* 188.1284.



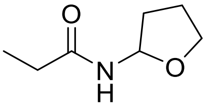
2-phenyl-*N*-(tetrahydrofuran-2-yl)acetamide (54c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with 2-phenylacetamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 16.61 mg (27 % yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.35 (t, *J* = 7.2 Hz, 2H), 7.28 (dd, *J* = 15.1, 5.0 Hz, 3H), 5.81 (s, 1H), 5.71 – 5.65 (m, 1H), 3.84 (dd, *J* = 14.2, 7.6 Hz, 1H), 3.76 (dd, *J* = 14.7, 7.5 Hz, 1H), 3.57 (s, 2H), 2.19 – 2.08 (m, 1H), 1.92 – 1.79 (m, 2H), 1.63 – 1.55 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 170.9, 134.4, 129.4, 129.0, 127.4, 81.2, 67.4, 43.8, 31.9, 24.5. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{12}H_{16}NO_2$) requires *m/z* 206.1176, found *m/z* 206.1176.



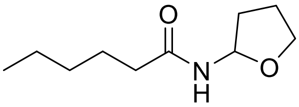
N-(tetrahydrofuran-2-yl)propionamide (55c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with propionamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 16.31 mg (38 % yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 5.95 (s, 1H), 5.73 (dd, J = 12.8, 6.4 Hz, 1H), 3.94 (dd, J = 14.5, 7.2 Hz, 1H), 3.81 (dd, J = 14.7, 7.2 Hz, 1H), 2.20 (dt, J = 11.3, 5.7 Hz, 3H), 1.95 (dt, J = 13.9, 6.8 Hz, 2H), 1.72 (dt, J = 12.6, 6.6 Hz, 1H), 1.15 (t, J = 7.5 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 173.7, 80.9, 67.4, 32.0, 29.7, 24.6, 9.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_7H_{14}NO_2$) requires m/z 144.1019, found m/z 144.1016.



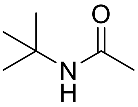
N-(tetrahydrofuran-2-yl)hexanamide (56c)

Starting from tetrahydrofurfuryl alcohol (0.3 mmol) with hexanamide (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 22.22 mg (40% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 5.86 (s, 1H), 5.77 – 5.69 (m, 1H), 3.98 – 3.88 (m, 1H), 3.80 (q, J = 7.0 Hz, 1H), 2.26 – 2.13 (m, 3H), 1.99 – 1.90 (m, 2H), 1.75 – 1.68 (m, 1H), 1.67 – 1.59 (m, 2H),1.34 – 1.27 (m, 4H), 0.89 (t, J = 6.9 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 173.1, 81.0, 67.4, 36.8, 32.0, 31.4, 25.1, 24.6, 22.3, 13.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{10}H_{20}NO_2$) requires m/z 186.1489, found m/z 186.1490.



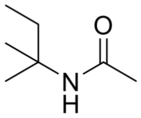
N-(tert-butyl)acetamide (57c)

Starting from neopentyl alcohol (5.0 mmol) following method B and the product was purified by flash chromatography using PE/EA (3:1 → 1:1) to afford 11.05 mg (32%) yield as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 5.34 (s, 1H), 1.93 (s, 3H), 1.35 (s, 9H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 183.6, 51.3, 28.7, 24.6. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_6H_{14}NO$) requires *m/z* 116.1070, found *m/z* 116.1074.



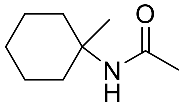
***N*-(tert-pentyl)acetamide (58c)**

Starting from 2,2-dimethylbutan-1-ol following method B and the product was purified by flash chromatography using PE/EA (3:1 → 1:1) to afford 15.88 mg (41%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.18 (s, 1H), 1.93 (s, 3H), 1.72 (dd, *J* = 15.0, 7.5 Hz, 2H), 1.29 (s, 6H), 0.84 (t, *J* = 7.5 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.7, 54.1, 32.8, 26.6, 24.7, 8.5. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_7H_{16}NO$) requires *m/z* 130.1226, found *m/z* 130.1221.



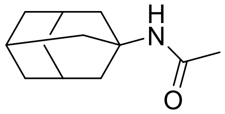
***N*-(1-methylcyclohexyl)acetamide (59c)**

Starting from (1-methylcyclohexyl)methanol (0.3 mmol) following method B and the product was purified by flash chromatography using PE/EA (3:1 → 1:1) to afford 20.94 mg (45 %) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.17 (s, 1H), 2.00 – 1.93 (m, 4H), 1.53 – 1.48 (m, 3H), 1.46 – 1.37 (m, 4H), 1.36 (s, 3H), 1.34 – 1.24 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 53.6, 36.8, 26.3, 25.7, 24.8, 22.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_9H_{18}NO$) requires *m/z* 156.1383, found *m/z* 156.1380.



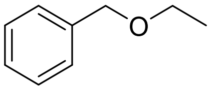
***N*-(adamantan-1-yl)acetamide (60c)**

Starting from 1-Adamantanemethanol (0.3 mmol) following method B and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 24.92 mg (43% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.16 (s, 1H), 2.07 (s, 2H), 1.99 (d, *J* = 2.6 Hz, 6H), 1.91 (s, 3H), 1.67 (s, 7H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 51.8, 41.6, 36.3, 29.4, 24.7. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{12}H_{20}NO$) requires *m/z* 194.1539, found *m/z* 194.1541.



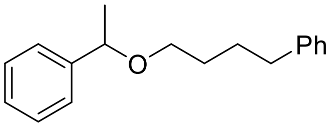
(ethoxymethyl)benzene (61c)

Starting from phenethyl alcohol (0.3 mmol) with ethanol (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 10.61 mg (26% yield) as a colorless oil.

^1H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 5H), 4.49 (s, 2H), 3.52 (q, *J* = 7.0 Hz, 2H), 1.23 (t, *J* = 7.0 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.5, 128.3, 127.6, 127.4, 72.6, 65.6, 15.1. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_9H_{13}O$) requires *m/z* 137.0961, found *m/z* 137.0965.



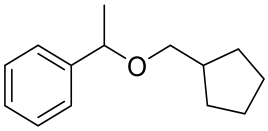
(1-(4-phenylbutoxy)ethyl)benzene (62c)

Starting from phenethyl alcohol (0.3 mmol) with 4-phenylbutanol (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 36.6 mg (48% yield) as a colorless oil.

^1H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 5H), 7.25 (d, *J* = 7.3 Hz, 2H), 7.16 (t, *J* = 7.6 Hz, 3H), 4.36 (q, *J* = 6.4 Hz, 1H), 3.30 (t, *J* = 6.1 Hz, 2H), 2.59 (t, *J* = 7.3 Hz, 2H), 1.69 – 1.58 (m, 4H), 1.42 (d, *J* = 6.5 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.2, 142.5, 128.4, 128.3, 128.2, 127.3, 126.1, 125.6, 78.0, 68.5, 35.7, 29.5, 28.1, 24.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{18}H_{23}O$) requires *m/z* 255.1743, found *m/z* 255.1746.



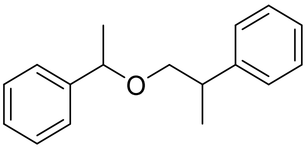
(1-(cyclopentylmethoxy)ethyl)benzene (63c)

Starting from phenethyl alcohol (0.3 mmol) with cyclopentanemethanol (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 31.24 mg (51% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.28 (m, 4H), 7.28 – 7.22 (m, 1H), 4.37 (q, *J* = 6.5 Hz, 1H), 3.16 (p, *J* = 9.0 Hz, 2H), 2.21 – 2.08 (m, 1H), 1.78 – 1.68 (m, 2H), 1.56 – 1.47 (m, 4H), 1.43 (d, *J* = 6.5 Hz, 3H), 1.24 – 1.13 (m, 2H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 144.4, 128.3, 127.2, 126.1, 78.0, 73.4, 39.6, 29.8, 29.5, 25.2, 25.3, 24.3. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{14}H_{21}O$) requires *m/z* 205.1587, found *m/z* 205.1584.



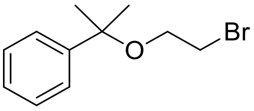
(1-(1-phenylethoxy)propan-2-yl)benzene (64c)

Starting from phenethyl alcohol (0.3 mmol) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 33.14 mg (46% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.36 – 7.25 (m, 6H), 7.18 (q, *J* = 6.0 Hz, 4H), 4.30 – 4.32 (m, 1H), 3.42 (dd, *J* = 14.6, 8.4 Hz, 1H), 3.32 (dd, *J* = 15.5, 7.5 Hz, 1H), 3.07– 2.96 (m, 1H), 1.40 (d, *J* = 6.3, 3H), 1.29 (d, *J* = 6.9 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 144.6&144.5, 144.1&144.0, 128.33&128.27, 128.22&128.19, 127.4&127.3, 127.26&127.21, 126.20&126.17, 126.14&126.09, 78.2&78.0, 74.6&74.5, 40.2&40.1, 24.2&24.1, 18.5&18.3. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{17}H_{21}O$) requires *m/z* 241.1587, found *m/z* 241.1582.



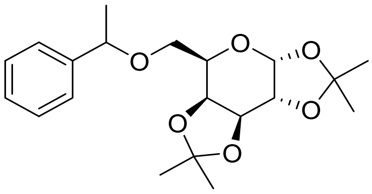
(2-(2-bromoethoxy)propan-2-yl)benzene (65c)

Starting from 2-methyl-2-phenylpropan-1-ol (0.3 mmol) with 2-bromoethan-1-ol (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (20:1 → 10:1) to afford 28.52 mg (35 % yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.46 – 7.41 (m, 2H), 7.35 (t, *J* = 7.5 Hz, 2H), 7.26 (t, *J* = 7.2 Hz, 1H), 3.49 – 3.44 (m, 2H), 3.44 – 3.39 (m, 2H), 1.56 (s, 7H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 145.8, 128.4, 127.3, 125.9, 63.4, 31.5, 28.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{11}H_{16}BrO$) requires *m/z* 243.0379, found *m/z* 243.0377.



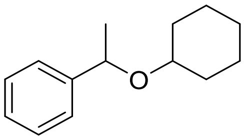
(3aS,5S,5aR,8aR,8bS)-2,2,7,7-tetramethyl-5-((1-phenylethoxy)methyl)tetrahydro-3aH-bis([1,3]dioxolo)[4,5-b:4',5'-d]pyran (66c)

Starting from phenethyl alcohol (0.3 mmol) with 1,2:3,4-di-O-isopropylidene-*D*-galactopyranose (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 42.59 mg (39% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.32 (d, *J* = 4.4 Hz, 8H), 7.28 – 7.24 (d, *J* = 4.7 Hz, 2H), 5.54 (d, *J* = 5.0 Hz, 1H), 5.50 (d, *J* = 5.0 Hz, 1H), 4.58 (td, *J* = 7.7, 2.3 Hz, 2H), 4.49 (qd, *J* = 6.4, 4.3 Hz, 2H), 4.30 (dq, *J* = 7.3, 3.3, 2.5 Hz, 3H), 4.22 (dd, *J* = 7.9, 1.7 Hz, 1H), 4.00 – 3.95 (m, 1H), 3.92 (td, *J* = 6.7, 1.5 Hz, 1H), 3.51 (td, *J* = 7.3, 3.4 Hz, 4H), 1.58 (s, 3H), 1.51 (s, 3H), 1.45 (d, *J* = 6.5 Hz, 6H), 1.42 (s, 3H), 1.39 (s, 3H), 1.34 (d, *J* = 1.7 Hz, 6H), 1.31 (d, *J* = 5.8 Hz, 6H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 143.7&143.6, 128.3&128.2, 127.3&127.2, 126.2&126.1, 109.1, 108.5&108.4, 96.3&96.2, 78.3&78.3, 71.1&70.9, 70.6, 70.5, 67.3&67.2, 66.9&66.3, 26.1&26.0, 25.95&25.91, 24.94&24.92, 24.4&24.3, 24.0&23.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{20}H_{29}O_6$) requires *m/z* 365.1959, found *m/z* 265.1959.



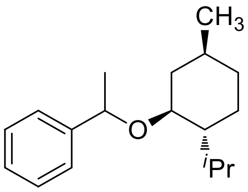
(1-(cyclohexyloxy)ethyl)benzene (67c)

Starting from phenethyl alcohol (0.3 mmol) with cyclohexanol (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 25.70 mg (42% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.30 (m, 4H), 7.29 – 7.23 (m, 1H), 4.59 (q, *J* = 6.5 Hz, 1H), 3.20 – 3.11 (m, 1H), 1.98 (d, *J* = 11.8 Hz, 1H), 1.70 (dt, *J* = 10.7, 9.4 Hz, 3H), 1.52 – 1.45 (m, 1H), 1.40 (d, *J* = 6.5 Hz, 3H), 1.33 – 1.24 (m, 2H), 1.20 – 1.09 (m, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.1, 128.3, 127.1, 126.1, 74.8, 74.2, 33.5, 31.8, 25.8, 24.9, 24.4, 24.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{14}H_{21}O$) requires *m/z* 205.1587, found *m/z* 205.1590.



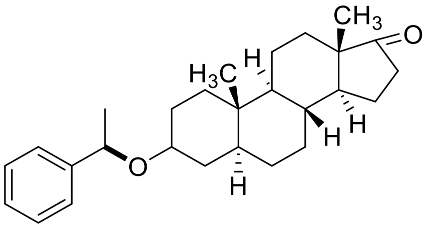
(1-(((1S,2R,5S)-2-isopropyl-5-methylcyclohexyl)oxy)ethyl)benzene (68c)

Starting from phenethyl alcohol (0.3 mmol) with *L*-Menthol (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 35.10 mg (45% yield) as a colorless oil.

^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.22 (m, 10H), 4.56 (q, *J* = 6.5 Hz, 1H), 4.49 (q, *J* = 6.4 Hz, 1H), 3.16 (td, *J* = 10.5, 4.2 Hz, 1H), 2.91 (td, *J* = 10.4, 4.1 Hz, 1H), 2.36 – 2.18 (m, 1H), 2.27 – 2.17 (m, 2H), 1.77 – 1.69 (m, 1H), 1.64 – 1.58 (m, 3H), 1.56 – 1.51 (m, 1H), 1.43 (dd, *J* = 9.1, 6.5 Hz, 6H), 1.29 – 1.16 (m, 5H), 0.93 (dd, *J* = 9.0, 6.9 Hz, 6H), 0.88 – 0.82 (m, 8H), 0.82 – 0.75 (m, 6H), 0.26 (d, *J* = 6.9 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.4&144.1, 128.2, 127.4&127.1, 126.9&126.2, 78.0&76.8, 75.1&73.8, 49.0&48.4, 42.0&40.3, 34.5&34.4, 31.6&31.4, 25.3&24.7, 24.5&23.6, 23.1&22.7, 22.4&21.3, 16.2&15.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{18}H_{29}O$) requires *m/z* 261.2213, found *m/z* 261.2209.



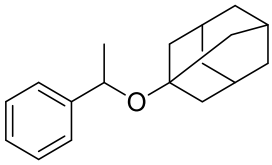
(5S,8R,9S,10S,13S,14S)-10,13-dimethyl-3-((R)-1-phenylethoxy)tetradecahydro-1H-cyclopenta[a]phenanthren-17(2H)-one (69c)

Starting from phenethyl alcohol (0.3 mmol) with epiandrosterone (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (20:1 → 10:1) to afford 37.82 mg (32% yield) as a colorless oil.

^1H NMR (400 MHz, $CDCl_3$) δ 7.32 (s, 4H), 7.26 (d, *J* = 3.4 Hz, 2H), 4.61 (qd, *J* = 6.5, 2.7 Hz, 1H), 3.19 – 3.10 (m, 1H), 2.42 (dd, *J* = 19.4, 9.2 Hz, 1H), 2.10 – 1.85 (m, 3H), 1.81 – 1.56 (m, 6H), 1.54 – 1.43 (m, 3H), 1.40 (d, *J* = 6.5 Hz, 3H), 1.35 – 1.15 (m, 7H), 1.00 – 0.94 (m, 1H), 0.83 (d, *J* = 7.8 Hz, 6H), 0.64 – 0.56 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 221.41&221.38, 144.9, 128.3, 127.1, 126.04&126.01, 75.5&75.4, 74.38&74.32, 54.44&54.43, 51.4, 47.8, 44.9&44.6, 37.0&36.8, 35.8&35.7, 35.81, 35.0, 34.4, 31.5, 30.9&30.8, 29.1&28.9, 28.3&27.7, 24.9&24.8, 21.7, 20.4&20.3, 13.8, 12.3&12.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{27}H_{39}O_2$) requires *m/z* 395.2945, found *m/z* 395.2948.



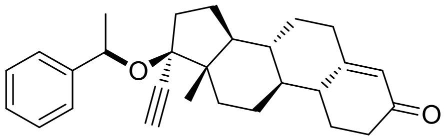
1-(1-phenylethoxy)adamantane (70c)

Starting from phenethyl alcohol (0.3 mmol) with adamantan-1-ol (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (20:1 → 10:1) to afford 29.18 mg (38% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.21 (m, 5H), 4.58 (q, *J* = 6.5 Hz, 1H), 3.37 (s, 1H), 2.14 (d, *J* = 12.0 Hz, 2H), 2.08 (s, 1H), 1.88 – 1.71 (m, 5H), 1.69 (s, 2H), 1.50 (dd, *J* = 23.5, 9.0 Hz, 4H), 1.42 (d, *J* = 6.5 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 145.2, 128.2, 126.9, 126.0, 78.6, 73.5, 37.6, 36.7, 36.3, 33.1, 31.8, 31.6, 30.9, 27.5, 27.4, 24.8. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{18}H_{25}O$) requires *m/z* 257.1900, found *m/z* 257.1196.



(8R,9S,10R,13S,14S,17R)-17-ethynyl-13-methyl-17-((R)-1-phenylethoxy)-6,7,8,9,10,11,12,13,14,15,16,17-dodecahydro-1H-cyclopenta[a]phenanthren-3(2H)-one (71c)

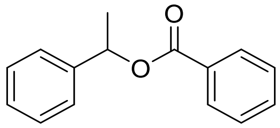
Starting from phenethyl alcohol (0.3 mmol) with norethindrone (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (20:1 → 10:1) to afford 42.21 mg (35% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 4H), 7.25 – 7.19 (m, 1H), 5.82 (s, 1H), 4.95 (p, *J* = 6.5 Hz, 1H), 2.54 – 2.37 (m, 3H), 2.34 – 2.20 (m, 4H), 2.12 – 2.02 (m, 2H), 1.95 – 1.86 (m, 1H), 1.86 – 1.78 (m, 2H), 1.76 – 1.70 (m, 2H), 1.61 – 1.57 (m, 2H), 1.57 – 1.50 (m, 2H), 1.41 (dd, *J* =

6.5, 3.1 Hz, 3H), 1.33 (d, *J* = 12.1 Hz, 1H), 1.23 – 1.14 (m, 1H), 1.13 – 1.00 (m, 1H), 0.94 – 0.83 (m, 2H), 0.91 – 0.87 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.2, 167.0&166.9, 146.7&146.1, 128.2&128.0, 126.9&126.7, 126.2&126.0, 124.7, 86.1&85.8, 85.5&85.4, 76.24&76.20, 75.3&75.2, 74.2&73.9, 49.4&49.3, 48.6&48.3, 47.8, 42.8&42.7, 41.0&40.9, 38.1&36.7, 36.5&35.6, 33.2&33.0, 30.9, 26.7&26.6, 26.4, 25.9&25.8, 23.3&23.2, 13.4&13.3. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{28}H_{35}O_2$) requires *m/z* 403.2632, found *m/z* 403.2635.



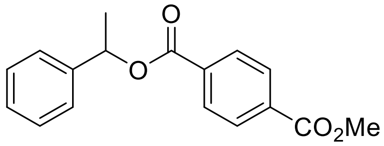
1-phenylethyl benzoate (72c)

Starting from phenethyl alcohol (0.3 mmol) with benzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 23.74 mg (35% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.7 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 8.4 Hz, 4H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.31 – 7.27 (m, 1H), 6.14 (q, *J* = 6.6 Hz, 1H), 1.67 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.8, 141.8, 132.9, 130.5, 129.6, 128.5, 128.3, 127.9, 126.0, 72.9, 22.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{15}H_{15}O_2$) requires *m/z* 227.1067, found *m/z* 227.1068.



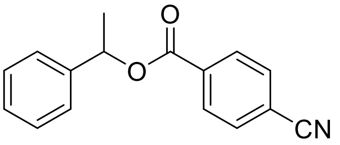
methyl (1-phenylethyl) terephthalate (73c)

Starting from phenethyl alcohol (0.3 mmol) with 4-(methoxycarbonyl)benzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 31.54 mg (37% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (q, *J* = 8.3 Hz, 4H), 7.45 (d, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 1H), 6.14 (q, *J* = 6.6 Hz, 1H), 3.94 (s, 3H), 1.69 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.3, 165.0, 141.4, 134.3, 133.9, 129.6, 129.5, 128.6, 128.0, 126.1, 73.5, 52.4, 22.3. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{17}O_4$) requires *m/z* 285.1121, found *m/z* 285.1125.



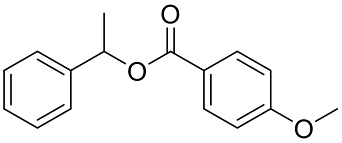
1-phenylethyl 4-cyanobenzoate (74c)

Starting from phenethyl alcohol (0.3 mmol) with 4-cyanobenzoic acid 1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 25.61 mg (34% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, J = 8.4 Hz, 2H), 7.74 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 7.1 Hz, 2H), 7.38 (t, J = 7.3 Hz, 2H), 7.33 (d, J = 8.4 Hz, 1H), 6.14 (q, J = 6.6 Hz, 1H), 1.69 (d, J = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.2, 141.0, 134.3, 132.2, 130.1, 128.7, 128.2, 126.1, 118.0, 116.3, 74.0, 22.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{16}H_{14}NO_2$) requires *m/z* 252.1019, found *m/z* 252.1021.



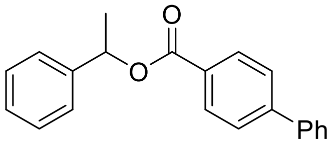
1-phenylethyl 4-methoxybenzoate (75c)

Starting from phenethyl alcohol (0.3 mmol) with 4-methoxybenzoic acid 1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 27.66 mg (36% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.8 Hz, 2H), 7.44 (d, J = 7.3 Hz, 2H), 7.36 (t, J = 7.4 Hz, 2H), 7.29 (t, J = 7.2 Hz, 1H), 6.92 (d, J = 8.9 Hz, 2H), 6.11 (q, J = 6.6 Hz, 1H), 3.85 (s, 3H), 1.65 (d, J = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 163.3, 142.0, 131.6, 128.5, 127.8, 126.0, 122.9, 113.6, 72.5, 55.4, 22.5. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{16}H_{17}O_3$) requires *m/z* 257.1172, found *m/z* 257.1168.



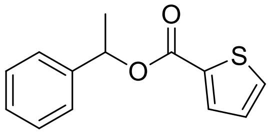
1-phenylethyl [1,1'-biphenyl]-4-carboxylate (76c)

Starting from phenethyl alcohol (0.3 mmol) with [1,1'-biphenyl]-4-carboxylic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 29.00 mg (32% yield) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 8.2 Hz, 2H), 7.62 (d, *J* = 7.2 Hz, 2H), 7.47 (dt, *J* = 7.4, 3.6 Hz, 4H), 7.39 (dd, *J* = 13.6, 6.6 Hz, 3H), 7.31 (t, *J* = 7.3 Hz, 1H), 6.16 (q, *J* = 6.6 Hz, 1H), 1.69 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 145.6, 141.8, 140.0, 130.2, 129.2, 128.9, 128.5, 128.1, 127.9, 127.3, 127.0, 126.0, 73.0, 22.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{21}H_{19}O_2$) requires *m/z* 303.1380, found *m/z* 303.1382.



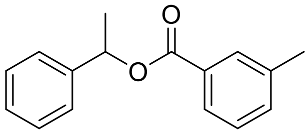
1-phenylethyl thiophene-2-carboxylate (77c)

Starting from phenethyl alcohol (0.3 mmol) with thiophene-2-carboxylic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 34.81 mg (50% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 3.7 Hz, 1H), 7.55 (d, *J* = 5.0 Hz, 1H), 7.43 (d, *J* = 7.4 Hz, 2H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.33 – 7.27 (m, 1H), 7.09 (t, *J* = 4.3 Hz, 1H), 6.09 (q, *J* = 6.6 Hz, 1H), 1.66 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.5, 141.5, 134.1, 133.5, 132.3, 128.5, 127.9, 127.7, 126.0, 73.1, 22.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{13}H_{13}O_2S$) requires *m/z* 233.0631, found *m/z* 233.0630.



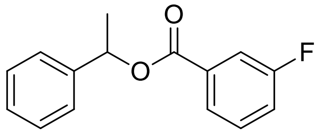
1-phenylethyl 3-methylbenzoate (78c)

Starting from phenethyl alcohol (0.3 mmol) with 3-methylbenzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 18.73 mg (26% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 5.4 Hz, 2H), 7.45 (d, *J* = 7.3 Hz, 2H), 7.36 (dd, *J* = 12.7, 5.3 Hz, 3H), 7.30 (dd, *J* = 12.0, 5.1 Hz, 2H), 6.13 (q, *J* = 6.6 Hz, 1H), 2.40 (s, 3H), 1.67 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.0, 141.8, 138.1, 133.7, 130.4, 130.1, 128.5, 128.2, 127.8, 126.8, 126.0, 72.8, 22.4, 21.3. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{16}H_{17}O_2$) requires *m/z* 241.1223, found *m/z* 241.1223.



1-phenylethyl 3-fluorobenzoate (79c)

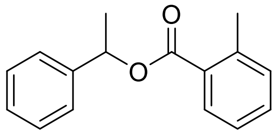
Starting from phenethyl alcohol (0.3 mmol) with 3-fluorobenzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 24.16 mg (33% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 7.7 Hz, 1H), 7.77 – 7.72 (m, 1H), 7.46 – 7.35 (m, 5H), 7.34 – 7.28 (m, 1H), 7.28 – 7.22 (m, 1H), 6.12 (q, *J* = 6.6 Hz, 1H), 1.68 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7(d, *J* = 3.0 Hz), 161.5 (d, *J* = 248.1 Hz), 141.4, 132.7(d, *J* = 7.5 Hz), 130.0 (d, *J* = 7.8 Hz), 128.6, 128.0, 126.0, 125.4 (d, *J* = 2.9 Hz), 120.0 (d, *J* = 21.4 Hz), 116.5(d, *J* = 23.1 Hz), 73.4, 22.3. (see spectrum)

^{19}F NMR (376 MHz, $CDCl_3$) δ -112.37, -112.39, -112.41, -112.43. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{15}H_{14}FO_2$) requires *m/z* 245.0972, found *m/z* 245.0970.



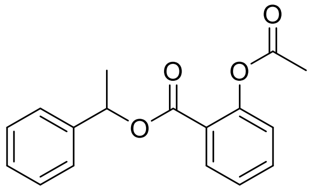
1-phenylethyl 2-methylbenzoate (80c)

Starting from phenethyl alcohol (0.3 mmol) with 2-methylbenzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 25.21 mg (35% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.96 (d, *J* = 7.9 Hz, 1H), 7.45 (d, *J* = 7.3 Hz, 2H), 7.42 – 7.34 (m, 3H), 7.30 (t, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 7.8 Hz, 2H), 6.11 (q, *J* = 6.6 Hz, 1H), 2.59 (s, 3H), 1.67 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 166.8, 141.8, 140.2, 131.9, 131.6, 130.6, 129.9, 128.5, 127.8, 126.1, 125.7, 72.8, 22.5, 21.8. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{16}H_{17}O_2$) requires *m/z* 241.1223, found *m/z* 241.1225.



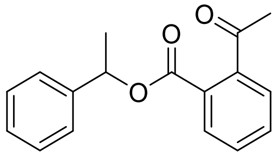
1-phenylethyl 2-acetoxybenzoate (81c)

Starting from phenethyl alcohol (0.3 mmol) with 2-acetoxybenzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 26.62 mg (31% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.05 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.55 (td, *J* = 7.8, 1.6 Hz, 1H), 7.44 – 7.34 (m, 4H), 7.34 – 7.29 (m, 2H), 7.09 (d, *J* = 8.1 Hz, 1H), 6.10 (q, *J* = 6.6 Hz, 1H), 2.21 (s, 3H), 1.65 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 169.6, 163.8, 150.5, 141.2, 133.7, 131.8, 128.6, 128.0, 126.2, 126.0, 123.8, 73.1, 22.1, 20.9. (see spectrum)

HRMS (ESI): exact mass calculated for [M+H]$^+$ ($C_{17}H_{17}O_4$) requires *m/z* 285.1121, found *m/z* 285.1121.



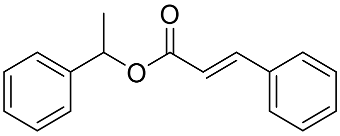
1-phenylethyl 2-acetylbenzoate (82c)

Starting from phenethyl alcohol (0.3 mmol) with 2-acetylbenzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 34.59 mg (43% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.90 (d, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.1 Hz, 1H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.43 – 7.35 (m, 5H), 7.31 (t, *J* = 7.1 Hz, 1H), 6.11 (q, *J* = 6.6 Hz, 1H), 2.48 (s, 3H), 1.67 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 203.2, 166.0, 143.1, 141.0, 132.1, 129.8, 128.8, 128.5, 128.1, 127.3, 126.2, 121.5, 74.0, 30.3, 21.9. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{17}O_3$) requires *m/z* 269.1172, found *m/z* 269.1171.



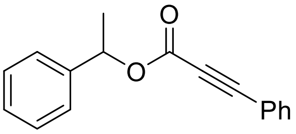
1-phenylethyl cinnamate (83c)

Starting from phenethyl alcohol (0.3 mmol) with cinnamic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 36.30 mg (48% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 16.0 Hz, 1H), 7.52 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.44 – 7.34 (m, 7H), 7.33 – 7.27 (m, 1H), 6.48 (d, *J* = 16.0 Hz, 1H), 6.03 (q, *J* = 6.6 Hz, 1H), 1.62 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.2, 144.9, 141.7, 134.4, 130.2, 128.8, 128.5, 128.1, 127.9, 126.1, 118.3, 72.4, 22.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{17}O_2$) requires *m/z* 253.1223, found *m/z* 253.1223.



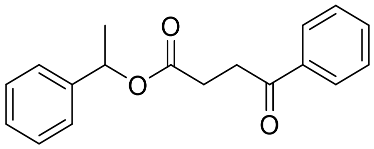
1-phenylethyl 3-phenylpropiolate (84c)

Starting from phenethyl alcohol (0.3 mmol) with 3-phenylpropiolic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 35.26 mg (47% yield) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.4 Hz, 2H), 7.46 – 7.29 (m, 8H), 6.02 (q, *J* = 6.6 Hz, 1H), 1.64 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.4, 140.6, 133.0, 130.6, 128.6, 128.5, 128.2, 126.3, 119.6, 86.3, 80.8, 74.3, 22.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{17}H_{15}O_2$) requires *m/z* 251.1067, found *m/z* 251.1071.



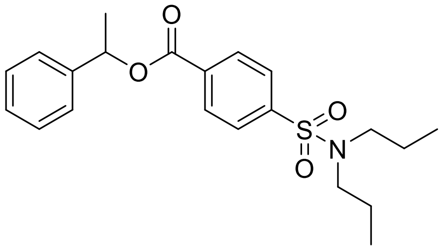
1-phenylethyl 4-oxo-4-phenylbutanoate (85c)

Starting from phenethyl alcohol (0.3 mmol) with 4-oxo-4-phenylbutanoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 36.39 mg (43% yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.99 – 7.94 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.31 (m, 4H), 7.29 (dt, *J* = 6.8, 2.9 Hz, 1H), 5.91 (q, *J* = 6.6 Hz, 1H), 3.39 – 3.22 (m, 2H), 2.90 – 2.71 (m, 2H), 1.55 (d, *J* = 6.6 Hz, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 198.0, 172.1, 141.6, 136.6, 133.2, 128.6, 128.5, 128.0, 127.8, 126.0, 72.6, 33.3, 28.5, 22.2. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{18}H_{19}O_3$) requires *m/z* 283.1329, found *m/z* 283.1330.



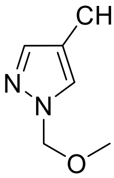
1-phenylethyl 4-(*N,N*-dipropylsulfamoyl)benzoate (86c)

Starting from phenethyl alcohol (0.3 mmol) with 4-(*N,N*-dibutylsulfamoyl)benzoic acid (1.5 equiv.) following method C and the product was purified by flash chromatography using PE/EA (50:1 → 20:1) to afford 45.53 mg (39% yield) as a white solid.

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.18 (d, *J* = 8.4 Hz, 2H), 7.87 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 7.2 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 2H), 7.32 (t, *J* = 7.2 Hz, 1H), 6.15 (q, *J* = 6.6 Hz, 1H), 3.12 – 3.05 (m, 4H), 1.70 (d, *J* = 6.6 Hz, 3H), 1.60 – 1.49 (m, 4H), 0.87 (t, *J* = 7.4 Hz, 6H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 164.5, 144.2, 141.2, 133.8, 130.2, 128.6, 128.2, 127.0, 126.1, 73.8, 49.9, 22.2, 21.9, 11.1. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{21}H_{28}NO_4S$) requires *m/z* 390.1734, found *m/z* 390.1739.



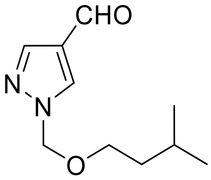
1-(methoxymethyl)-1H-pyrazole-4-carbaldehyde (2g)

Starting from (1-methoxypropan-2-yl)benzene (0.3 mmol) with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 16.80 mg (40 % yield) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.92 (s, 1H), 8.12 (s, 1H), 8.03 (s, 1H), 5.44 (s, 2H), 3.39 (s, 3H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.2, 140.9, 133.3, 125.4, 82.7, 57.4. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_6H_9N_2O_2$) requires *m/z* 141.0659, found *m/z* 141.0660.



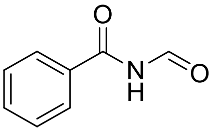
1-((isopentyloxy)methyl)-1H-pyrazole-4-carbaldehyde (3g)

Starting from 2-((isopentyloxy)methyl)tetrahydrofuran with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (3:1 → 1:1) to afford 5.88 mg (10 %) as a colorless oil.

^{1}H NMR (400 MHz, CDCl$_3$) δ 9.92 (s, 1H), 8.12 (s, 1H), 8.02 (s, 1H), 5.48 (s, 2H), 3.54 (t, *J* = 6.3 Hz, 2H), 1.64 (dt, *J* = 20.0, 6.7 Hz, 1H), 1.45 (q, *J* = 6.8 Hz, 2H), 0.86 (dd, *J* = 6.6, 1.0 Hz, 6H). (see spectrum)

^{13}C NMR (101 MHz, CDCl$_3$) δ 184.3, 140.8, 133.2, 125.4, 81.5, 68.6, 38.1, 25.0, 22.6. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{10}H_{17}N_2O_2$) requires *m/z* 197.1285, found *m/z* 197.1282.



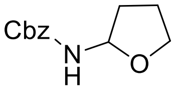
***N*-formylbenzamide (8g)**

Starting from *N*-((tetrahydrofuran-2-yl)methyl)benzamide with 1H-pyrazole-4-carboxaldehyde (1.5 equiv.) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford trace yield as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.38 (d, *J* = 9.7 Hz, 1H), 9.25 (s, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 2H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 163.7, 134.1, 131.2, 129.3, 128.0. (see spectrum)

HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_8H_8NO_2$) requires *m/z* 150.0550, found *m/z* 150.0554.



benzyl (tetrahydrofuran-2-yl)carbamate (1h)

Starting from benzyl (1,5-dihydroxypentan-2-yl)carbamate (0.3 mmol) following method A and the product was purified by flash chromatography using PE/EA (5:1 → 3:1) to afford 30.8 mg (46% yield) as a colorless oil.

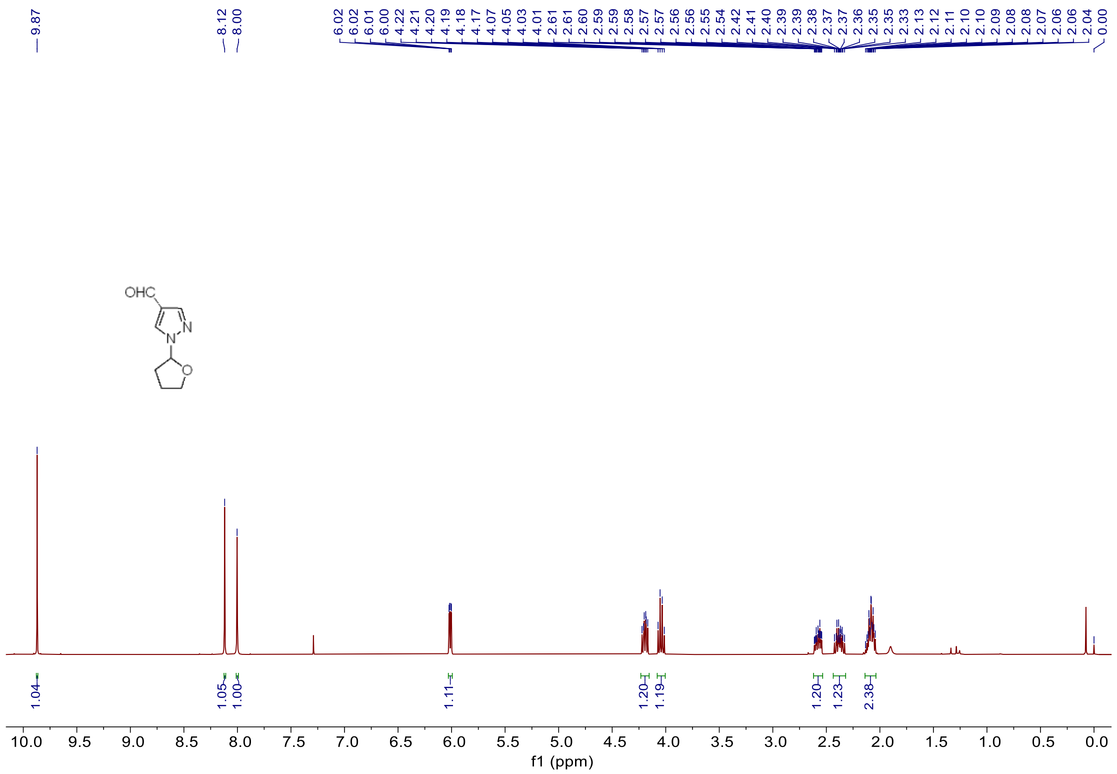
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (dd, *J* = 10.1, 3.8 Hz, 5H), 5.57 (d, *J* = 2.9 Hz, 1H), 5.32 (d, *J* = 8.1 Hz, 1H), 5.10 (d, *J* = 3.4 Hz, 2H), 3.91 (dd, *J* = 8.3, 7.0 Hz, 1H), 3.85 – 3.77 (m, 1H), 2.17 (td, *J* = 13.9, 7.1 Hz, 1H), 1.98 – 1.86 (m, 2H), 1.75 – 1.62 (m, 1H). (see spectrum)

^{13}C NMR (101 MHz, $CDCl_3$) δ 155.6, 136.3, 128.6, 128.3, 128.2, 82.9, 67.2, 67.0, 31.9, 24.7. (see spectrum)

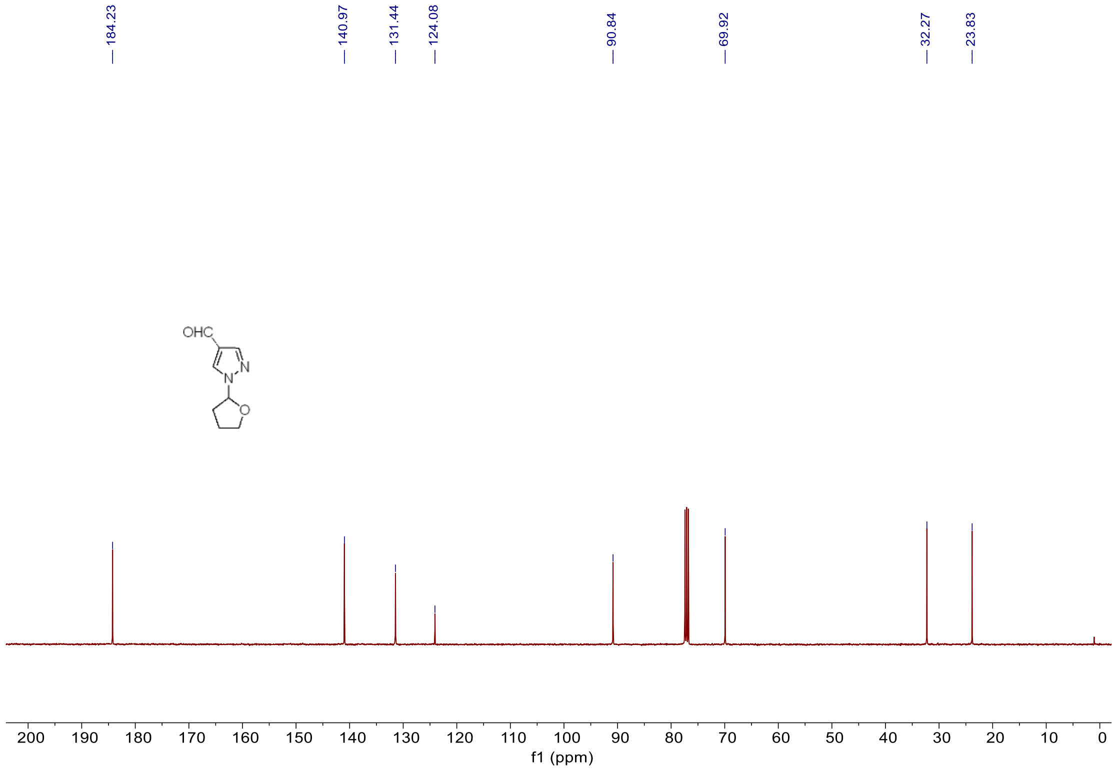
HRMS (ESI): exact mass calculated for $[M+H]^+$ ($C_{12}H_{16}NO_3$) requires *m/z* 222.1125, found *m/z* 222.1128.

10. NMR Spectra

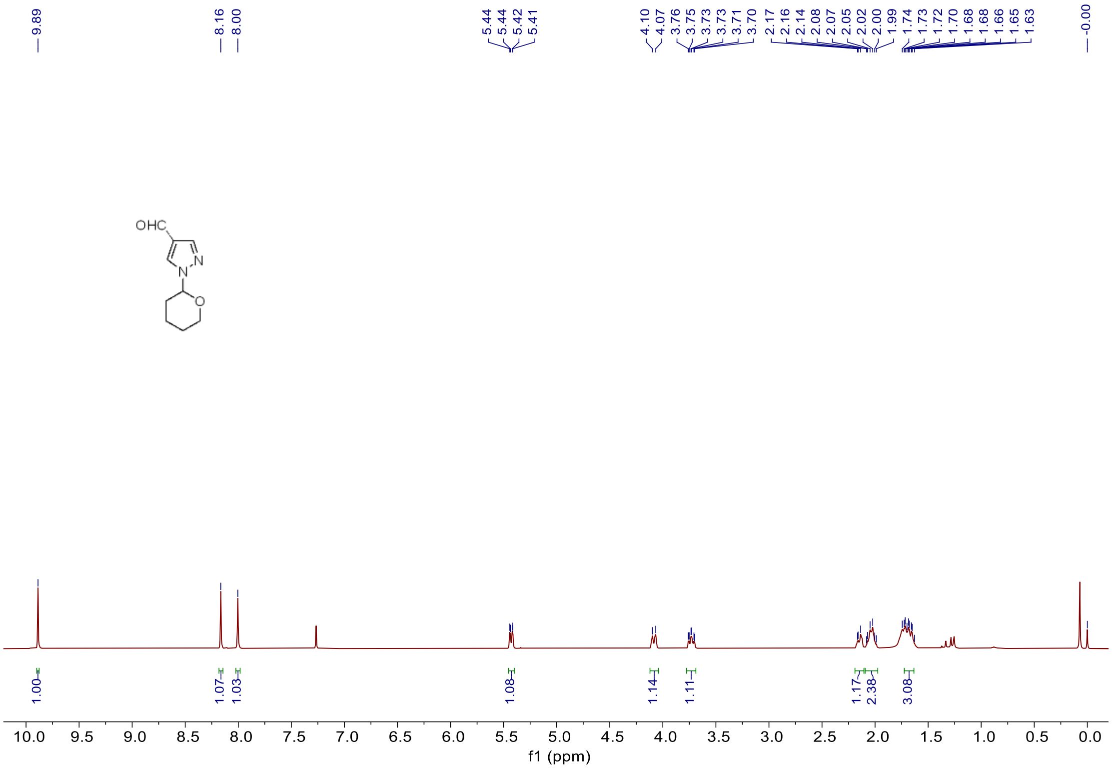
^{1}H NMR of spectrum of (400 MHz) of **1c** (see procedure)



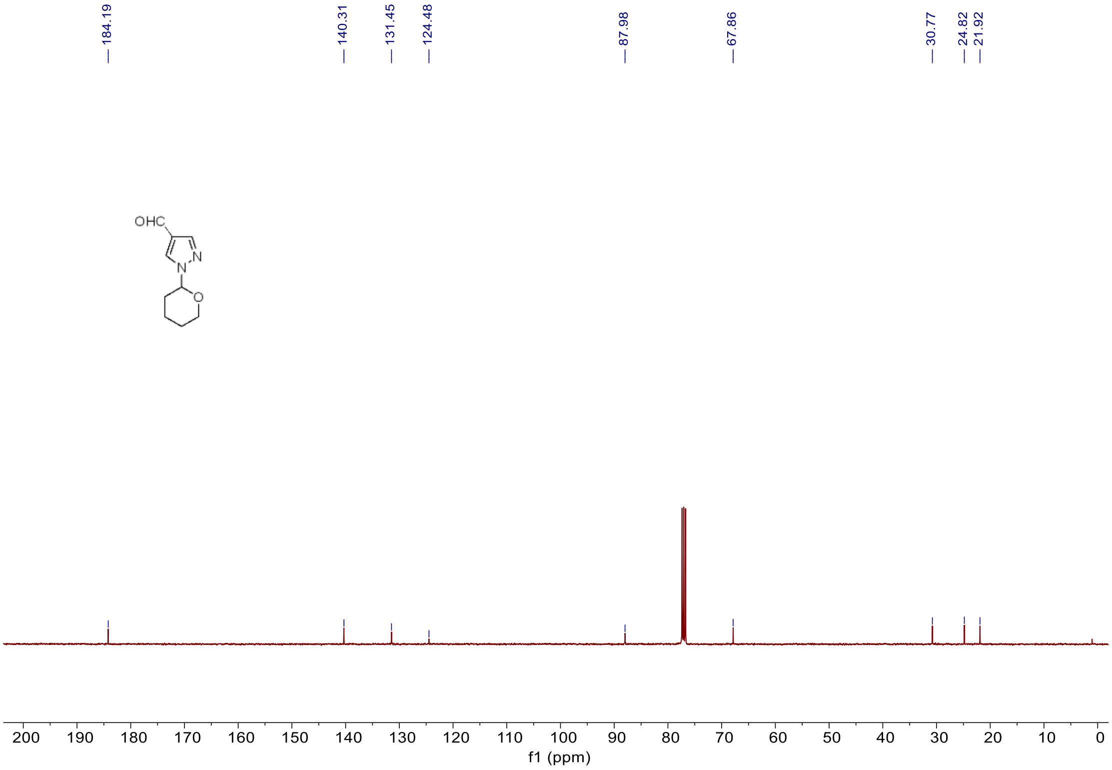
^{13}C NMR spectrum (101 MHz) of **1c** (see procedure)



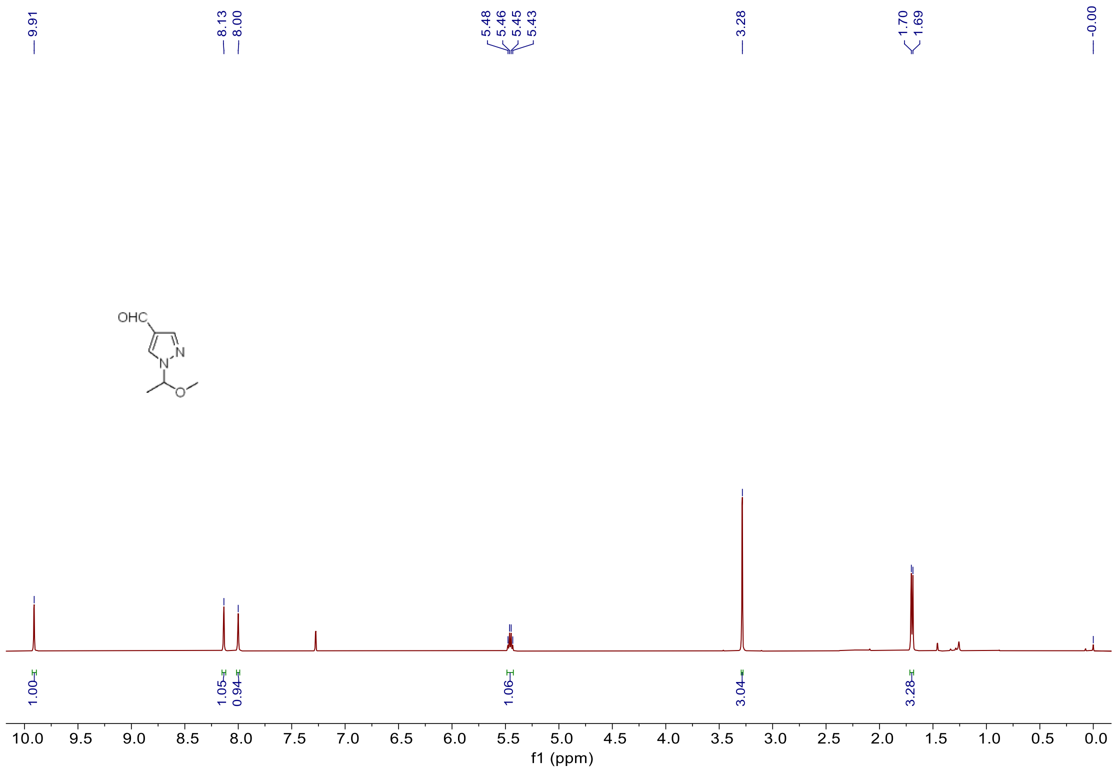
^{1}H NMR (400 MHz) spectrum (101 MHz) of **2c** (see procedure)



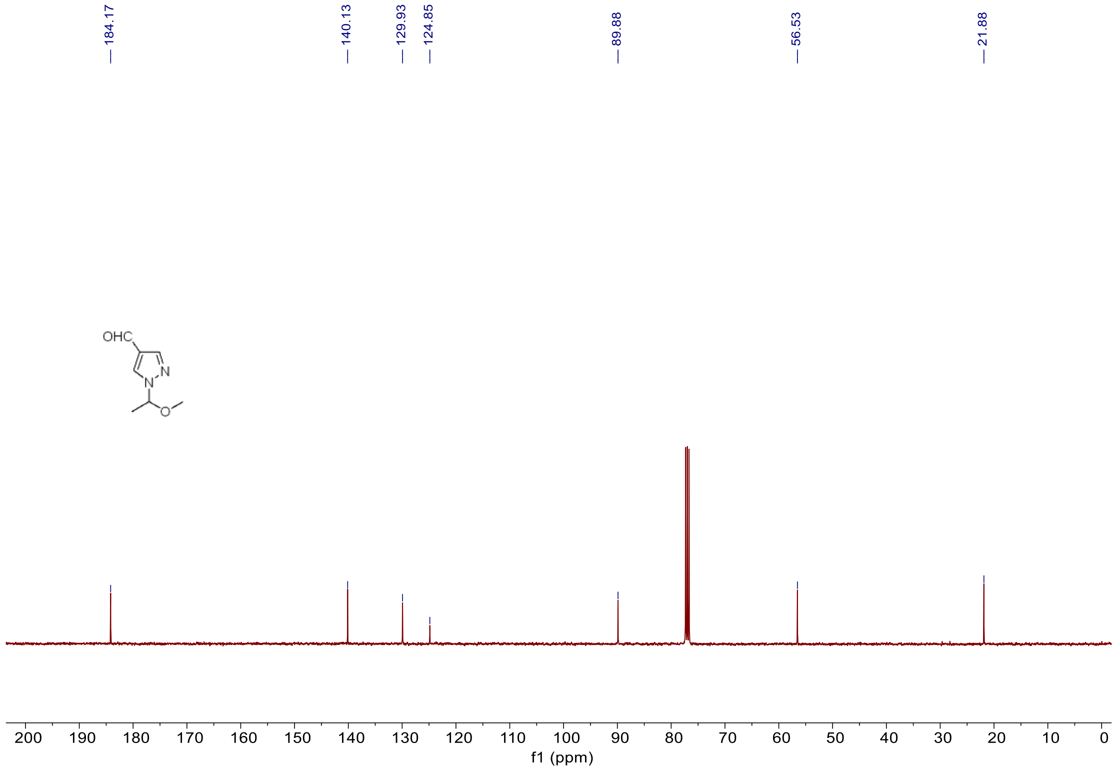
^{13}C NMR spectrum (101 MHz) of **2c** (see procedure)



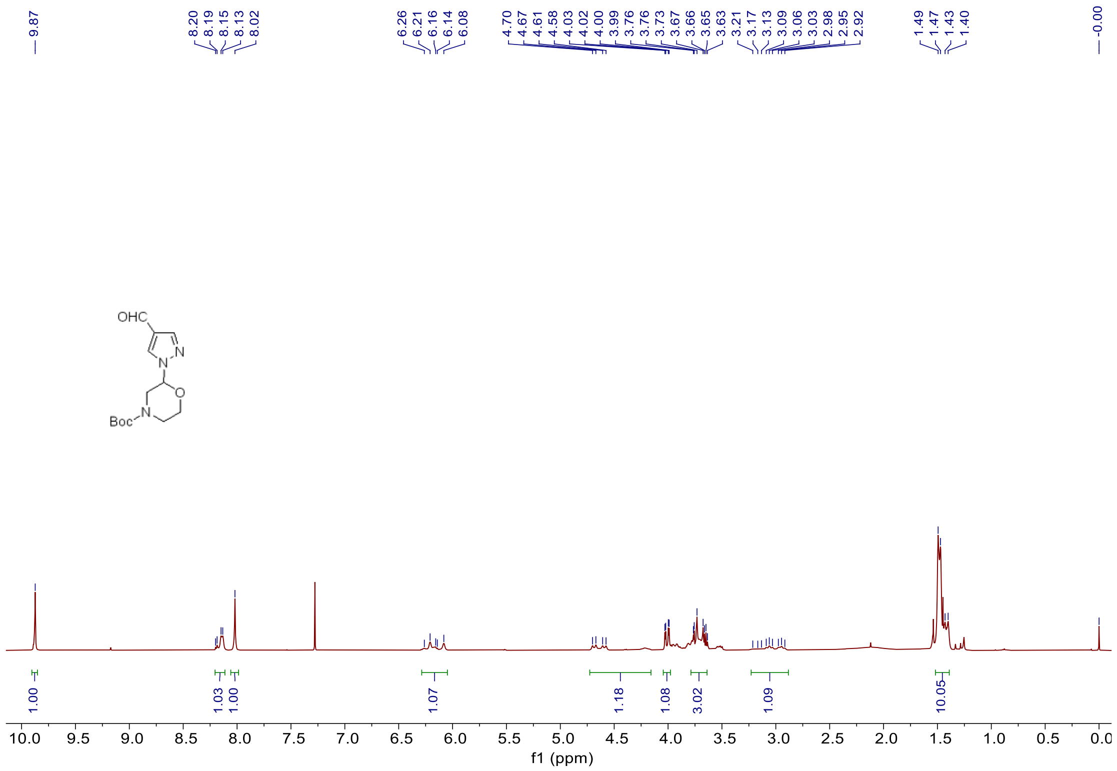
^{1}H NMR spectrum (400 MHz) of **3c** (see procedure)



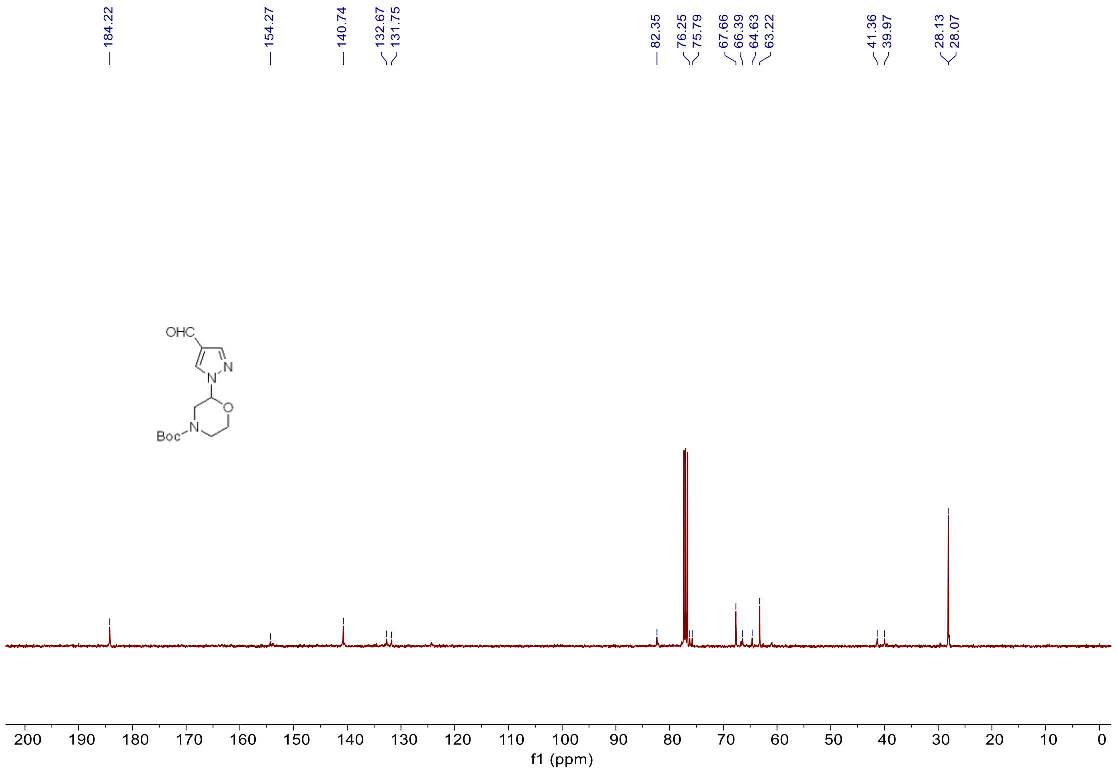
^{13}C NMR spectrum (101 MHz) of **3c** (see procedure)



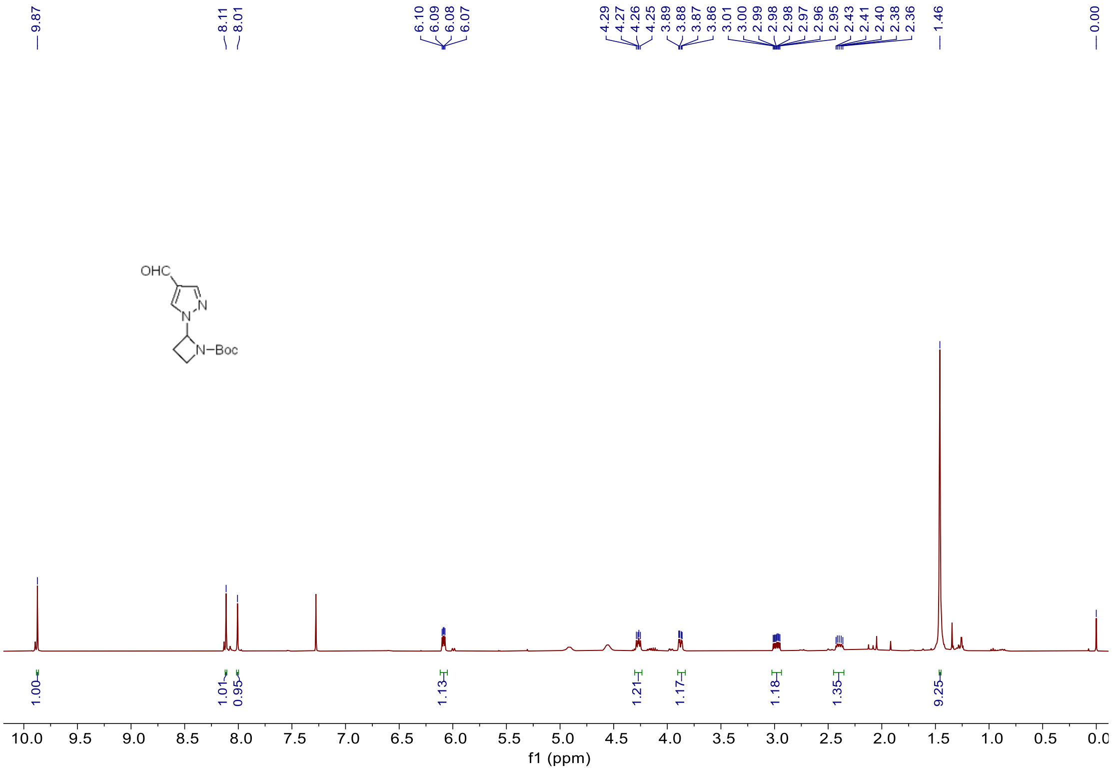
^{1}H NMR spectrum (400 MHz) of **4c** (see procedure)



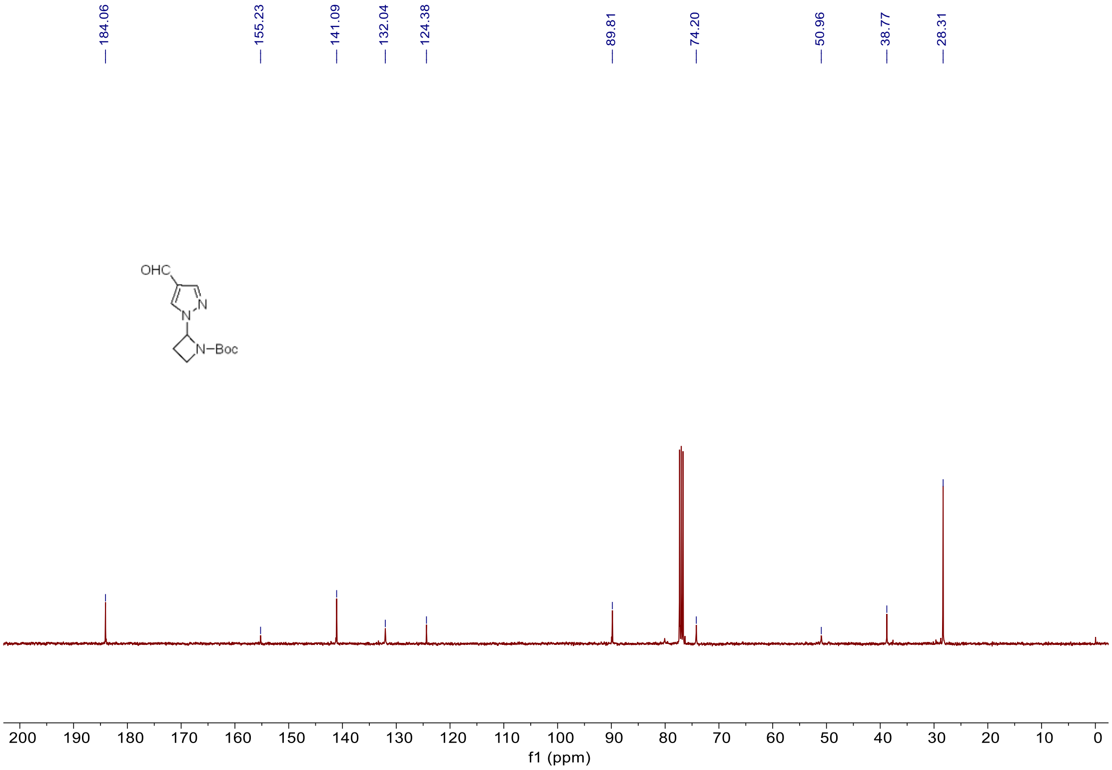
^{13}C NMR spectrum (101 MHz) of **4c** (see procedure)



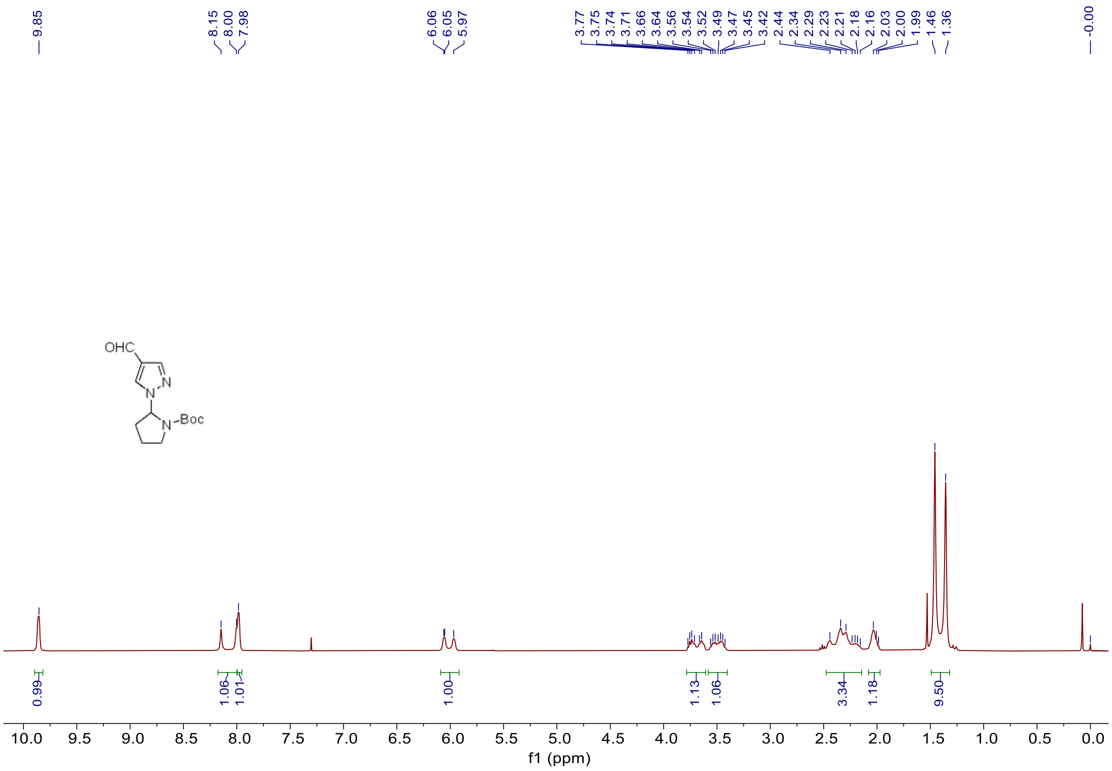
^{1}H NMR spectrum (400 MHz) of **5c** (see procedure)



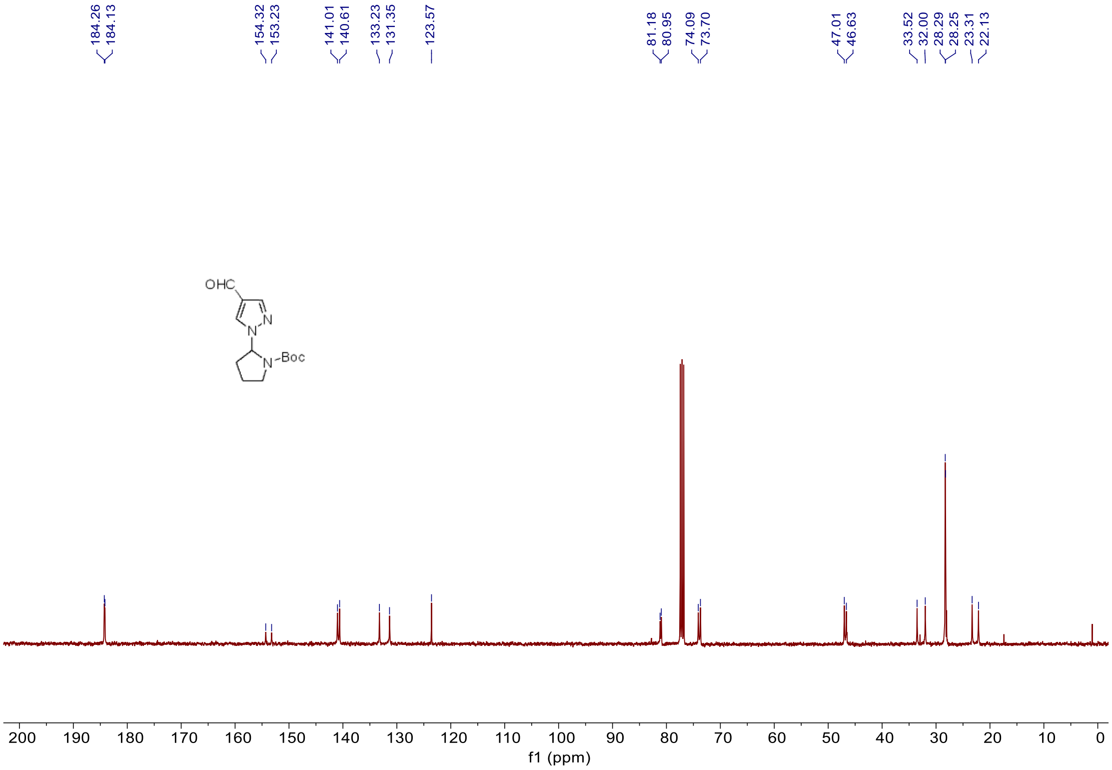
^{13}C NMR spectrum (101 MHz) of **5c** (see procedure)



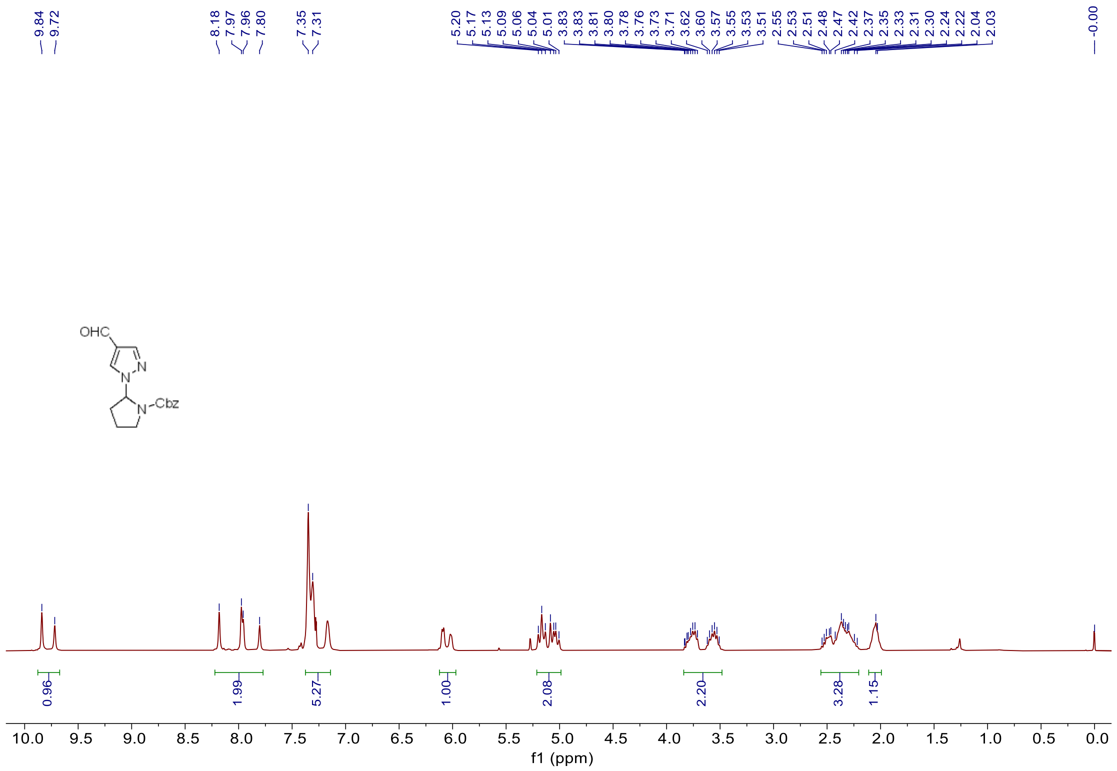
^{1}H NMR spectrum (400 MHz) of **6c** (see procedure)



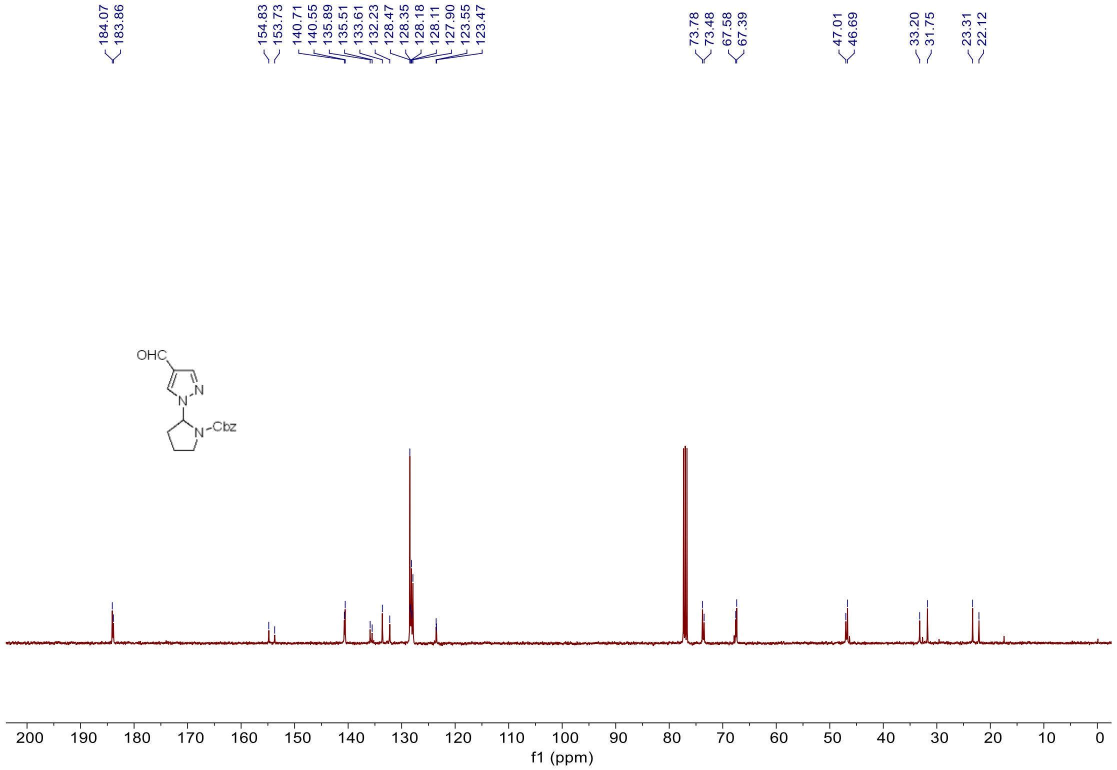
^{13}C NMR spectrum (101 MHz) of **6c** (see procedure)



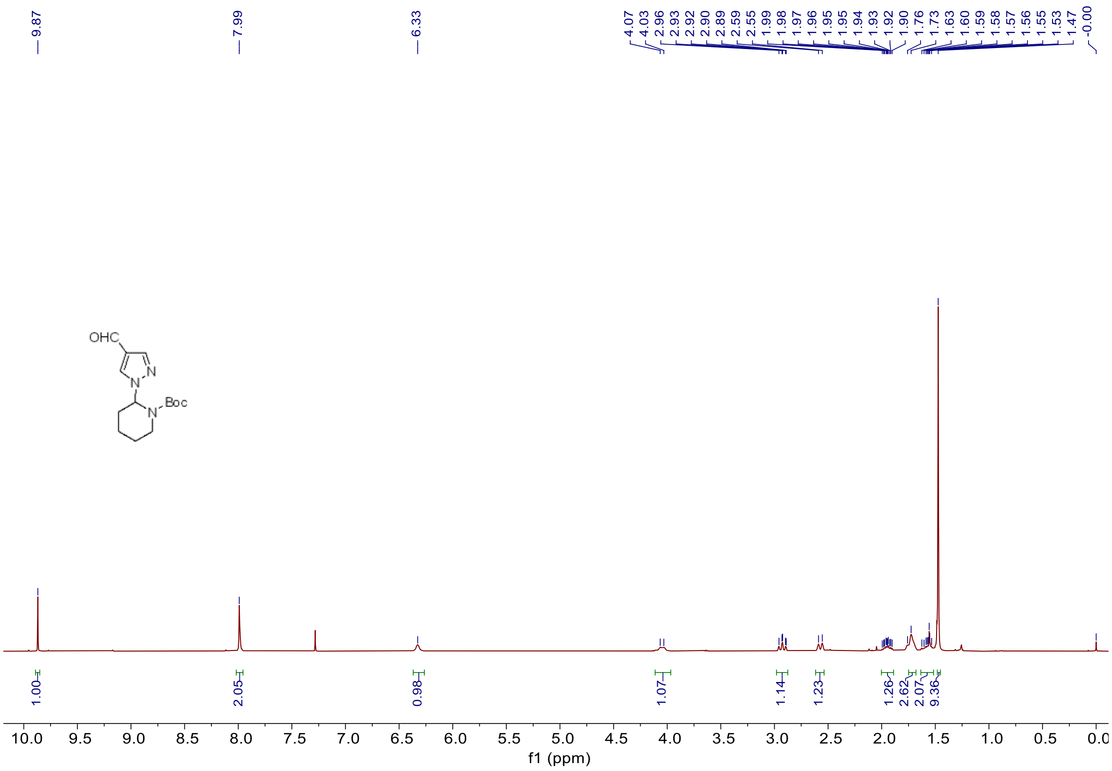
^{1}H NMR spectrum (400 MHz) of **7c** (see procedure)



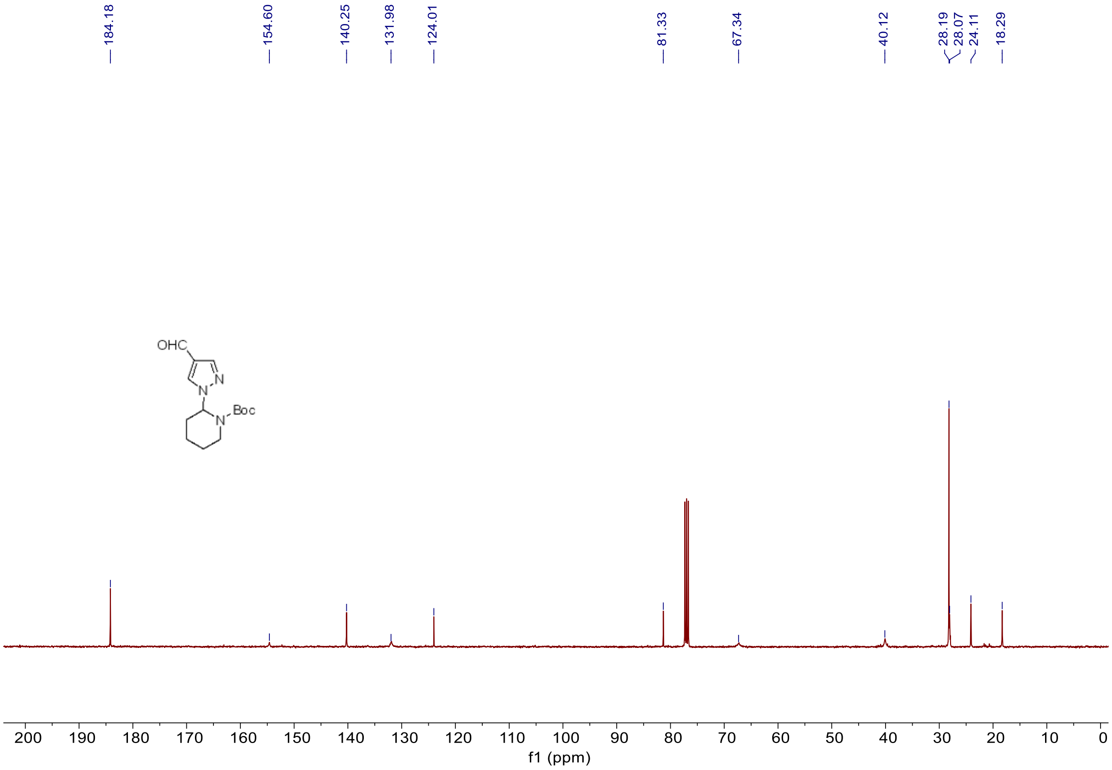
^{13}C NMR spectrum (101 MHz) of **7c** (see procedure)



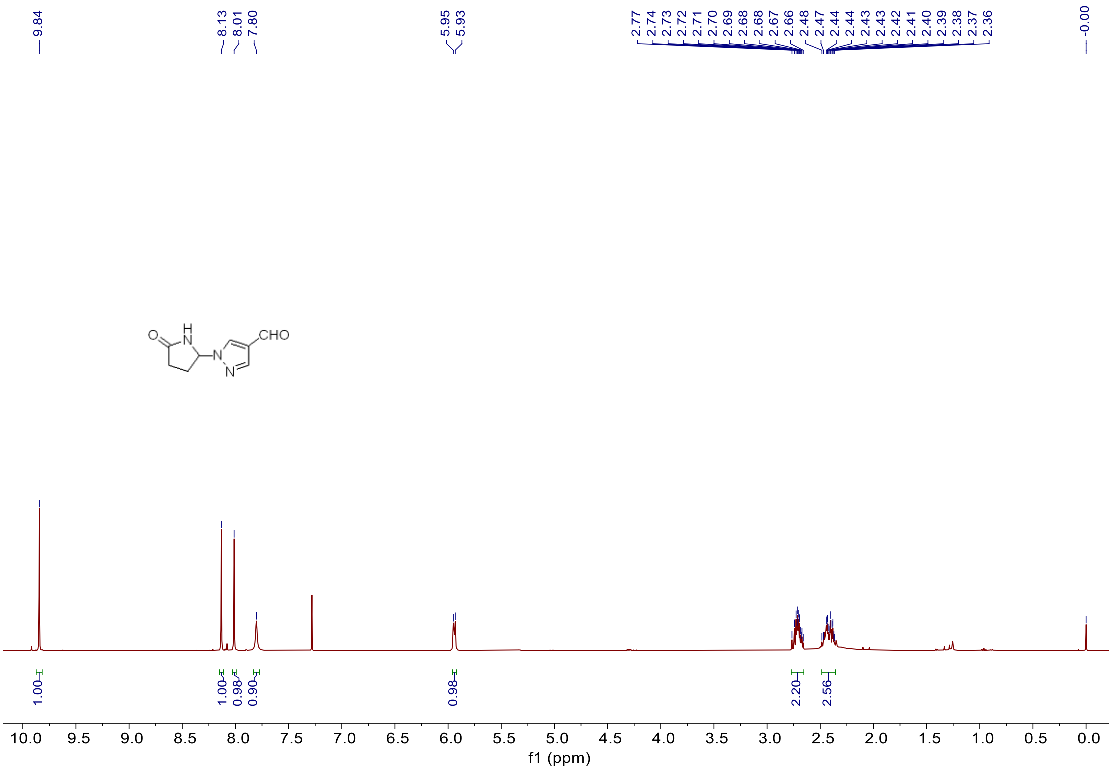
^{1}H NMR spectrum (400 MHz) of **8c** (see procedure)



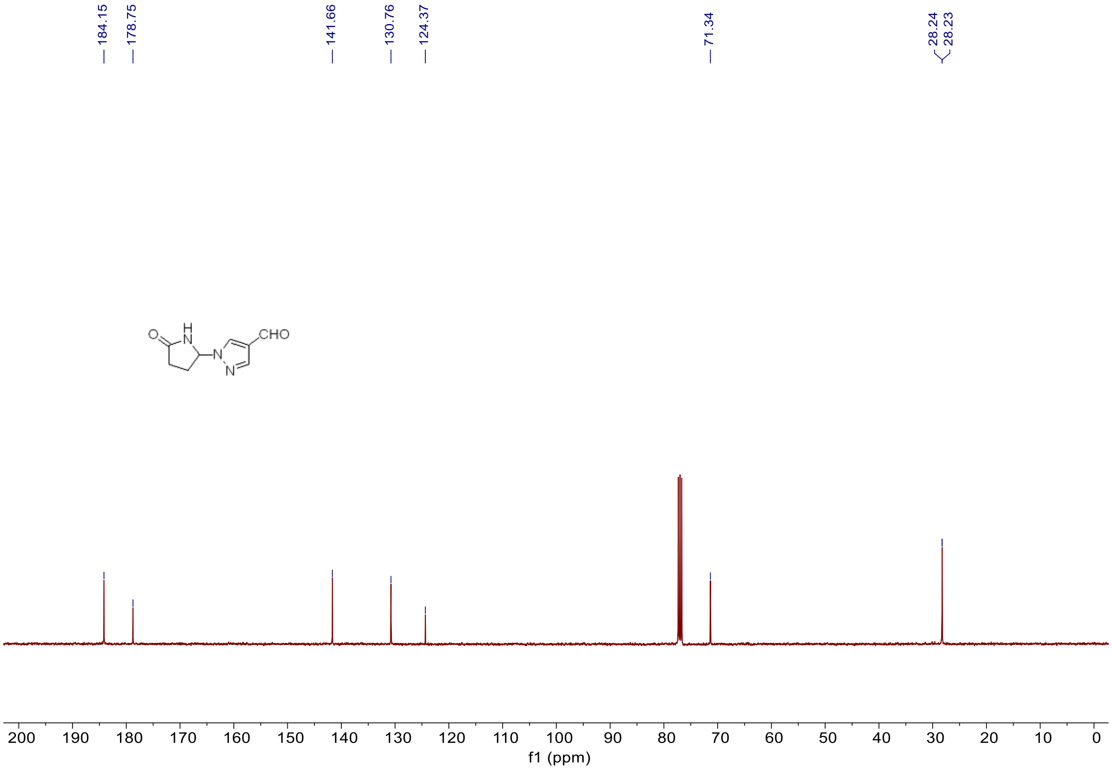
^{13}C NMR spectrum (101 MHz) of **8c** (see procedure)



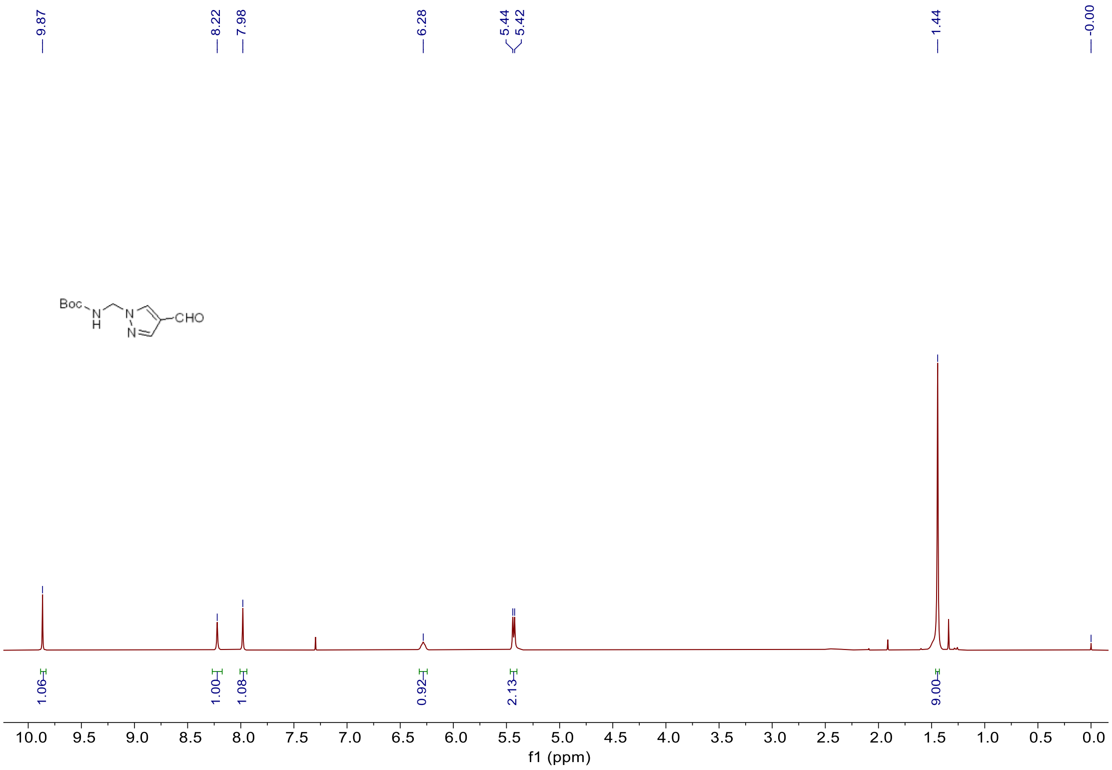
^{1}H NMR spectrum (400 MHz) of **9c** (see procedure)



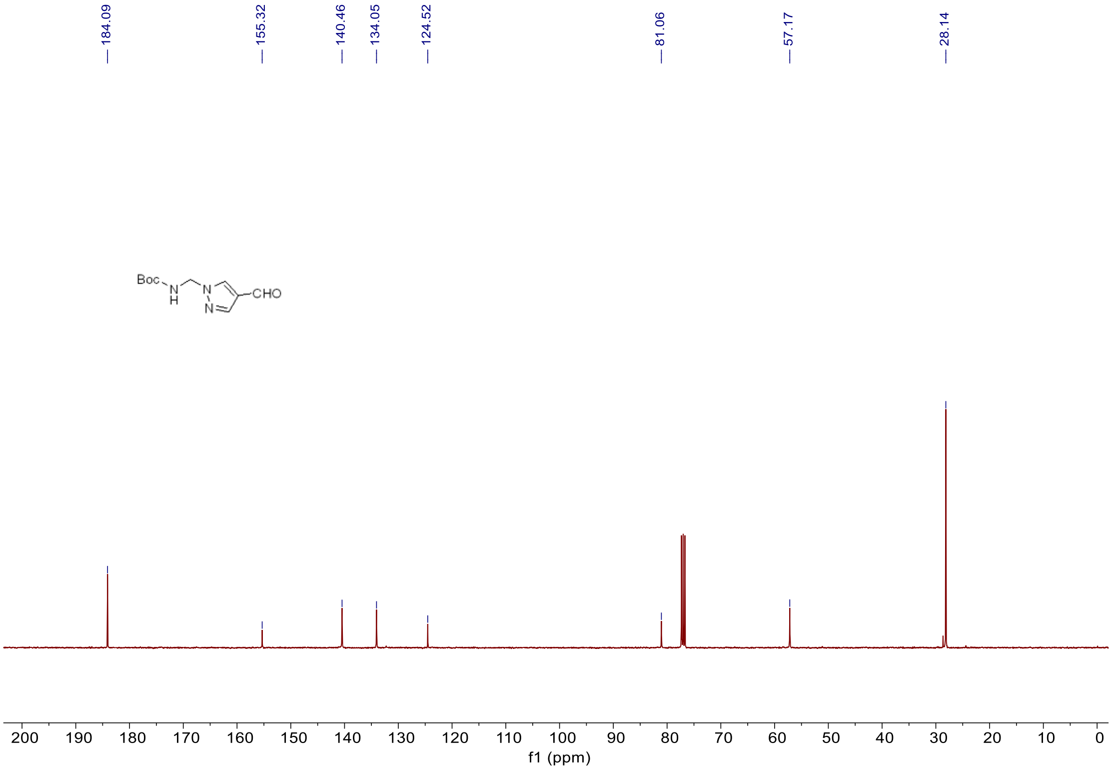
^{13}C NMR spectrum (101 MHz) of **9c** (see procedure)



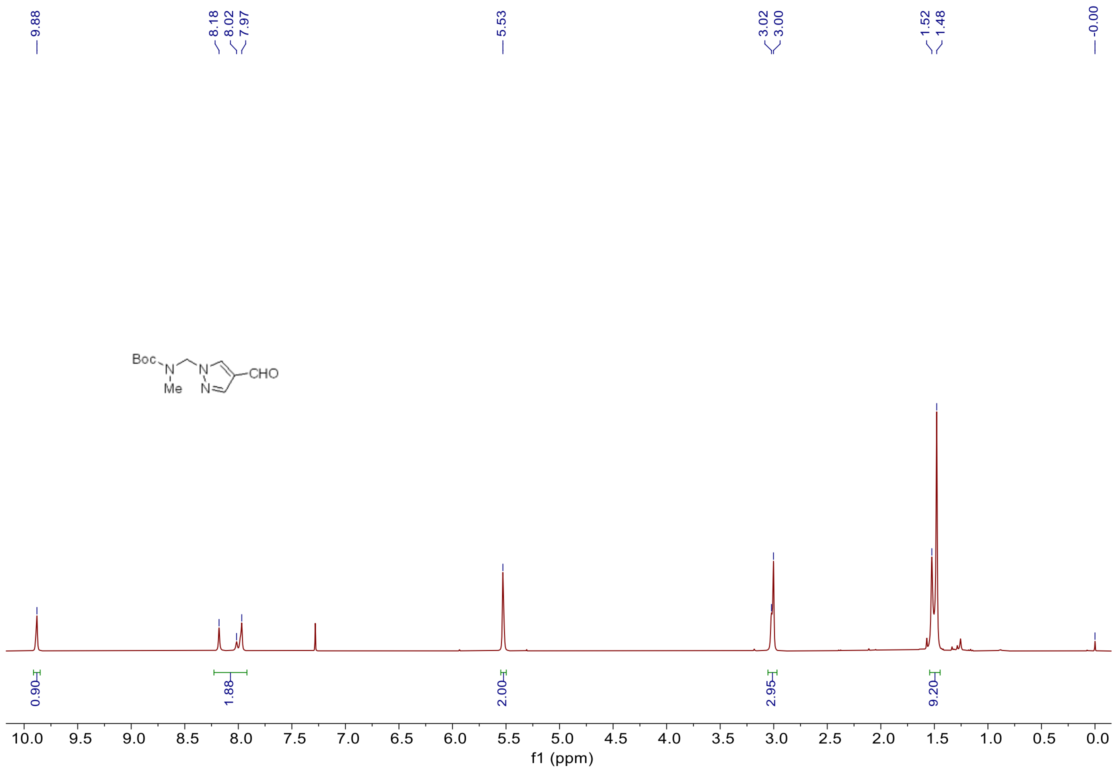
^{1}H NMR spectrum (400 MHz) of **10c** (see procedure)



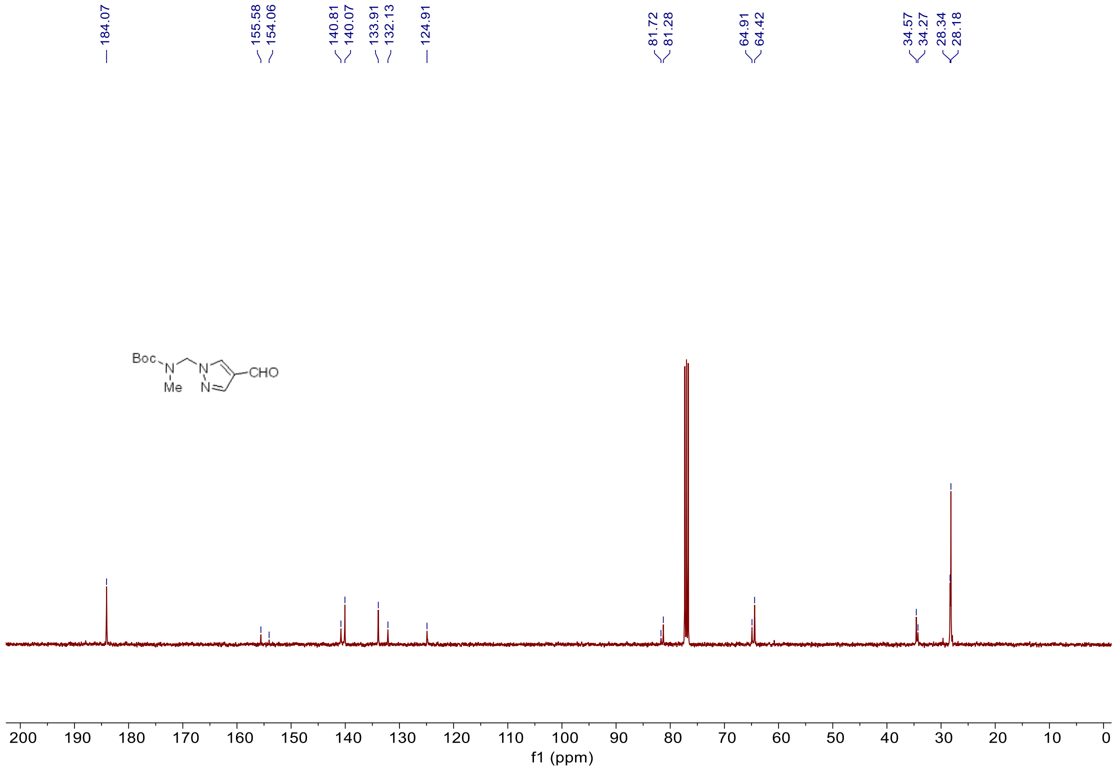
^{13}C NMR spectrum (101 MHz) of **10c** (see procedure)



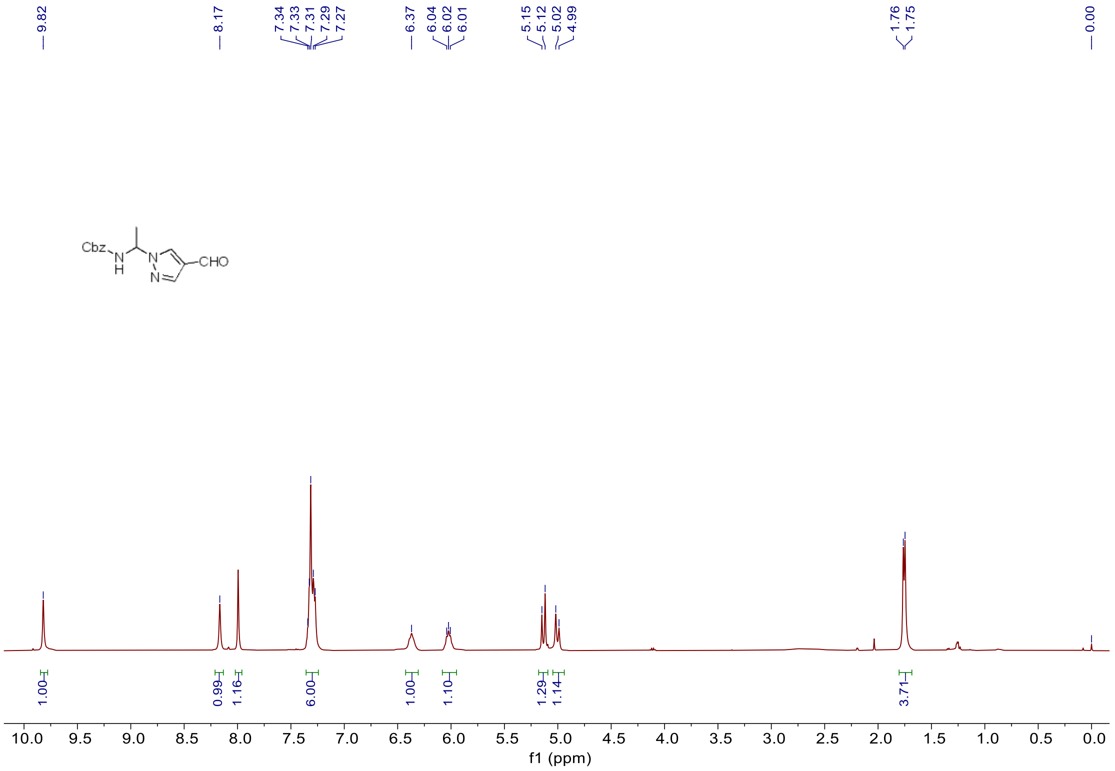
^{1}H NMR spectrum (400 MHz) of **11c** (see procedure)



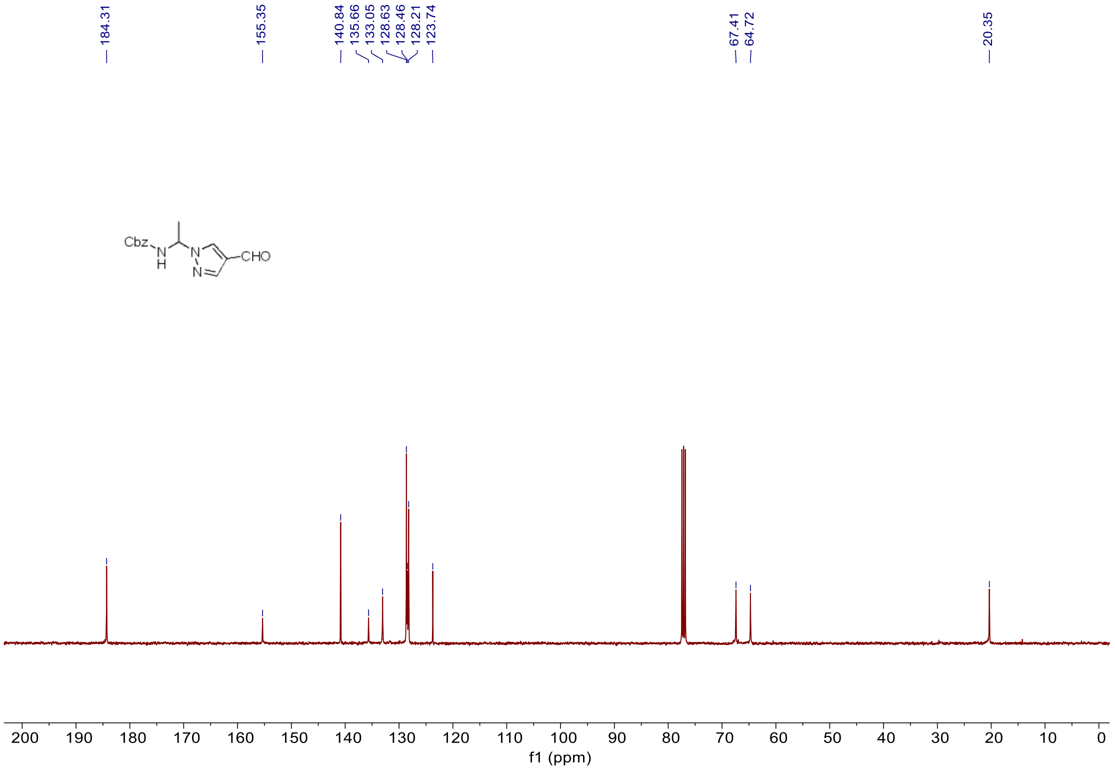
^{13}C NMR spectrum (101 MHz) of **11c** (see procedure)



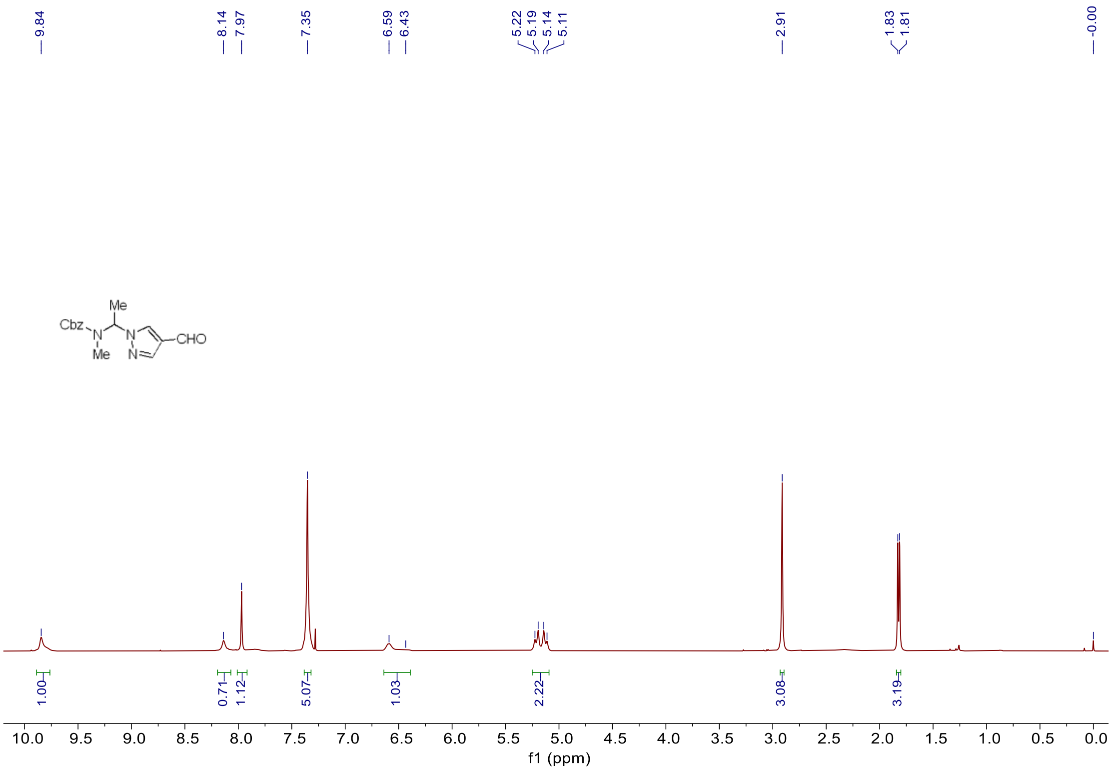
^{1}H NMR spectrum (400 MHz) of **12c** (see procedure)



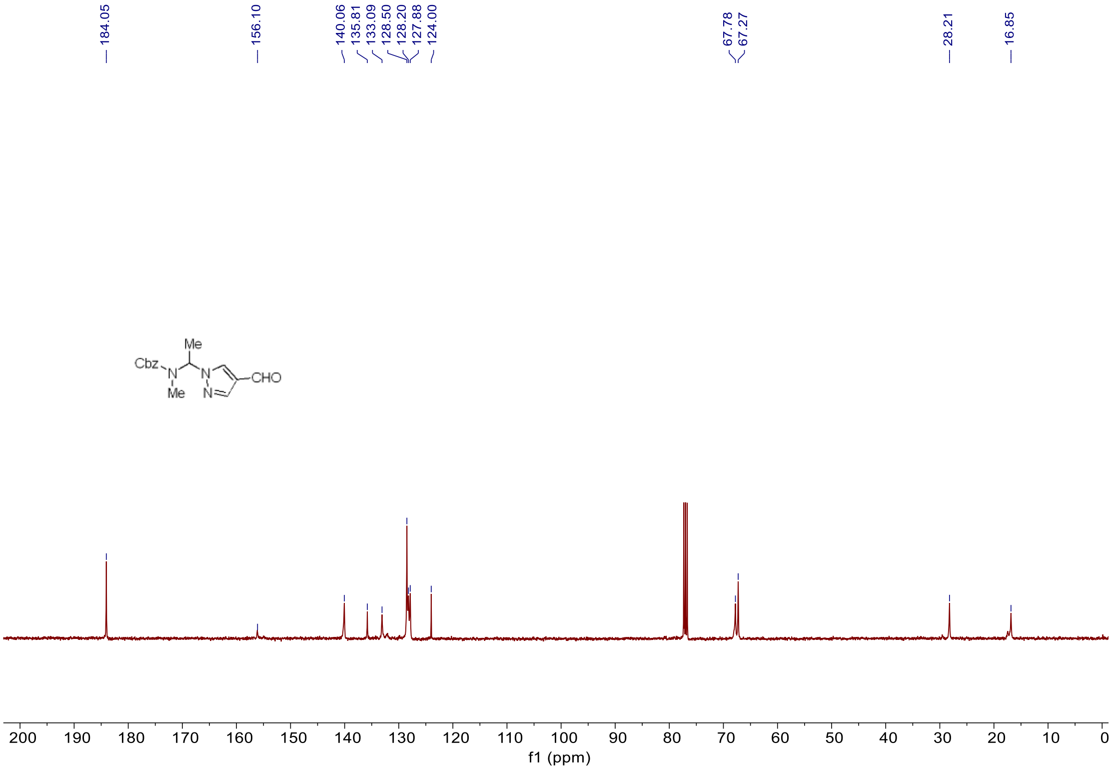
^{13}C NMR spectrum (101 MHz) of **12c** (see procedure)



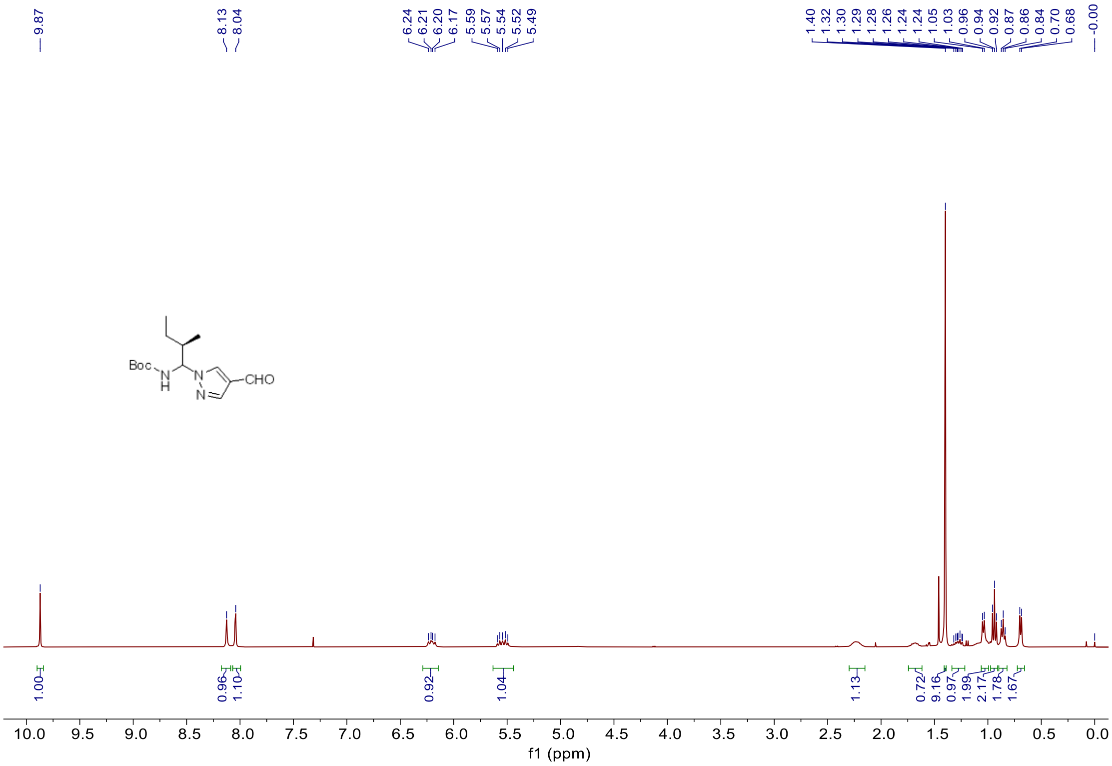
^{1}H NMR spectrum (400 MHz) of **13c** (see procedure)



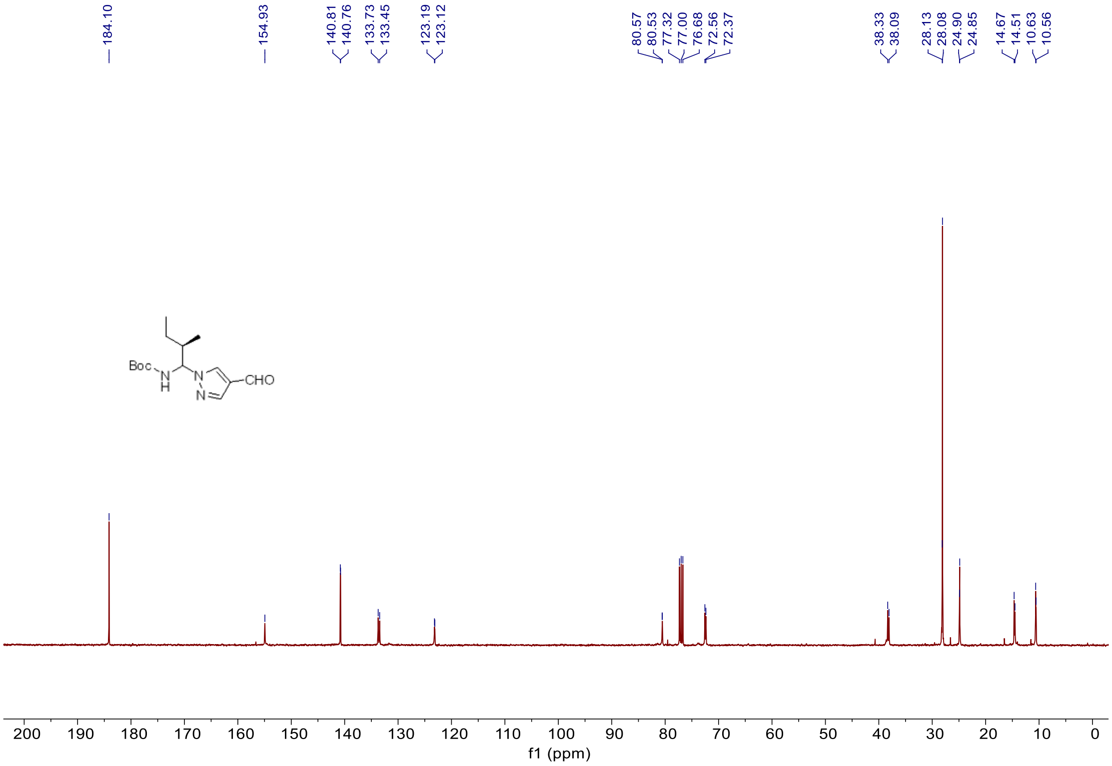
^{13}C NMR spectrum (101 MHz) of **13c** (see procedure)



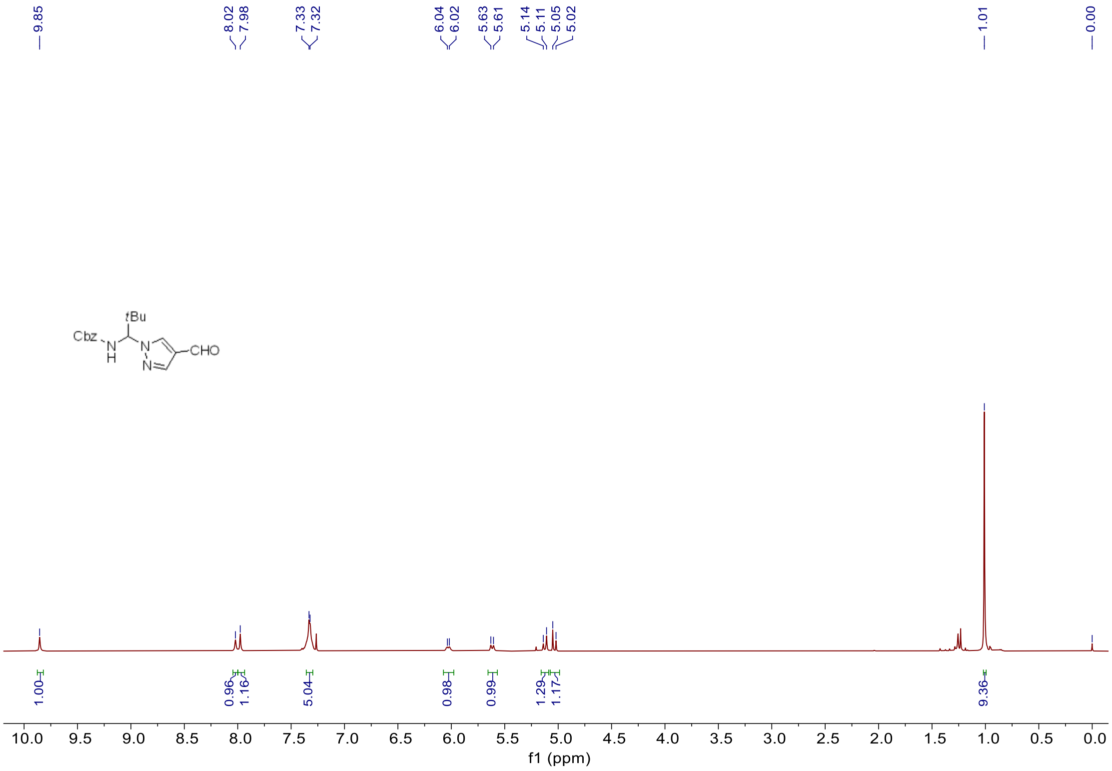
^{1}H NMR spectrum (400 MHz) of **14c** (see procedure)



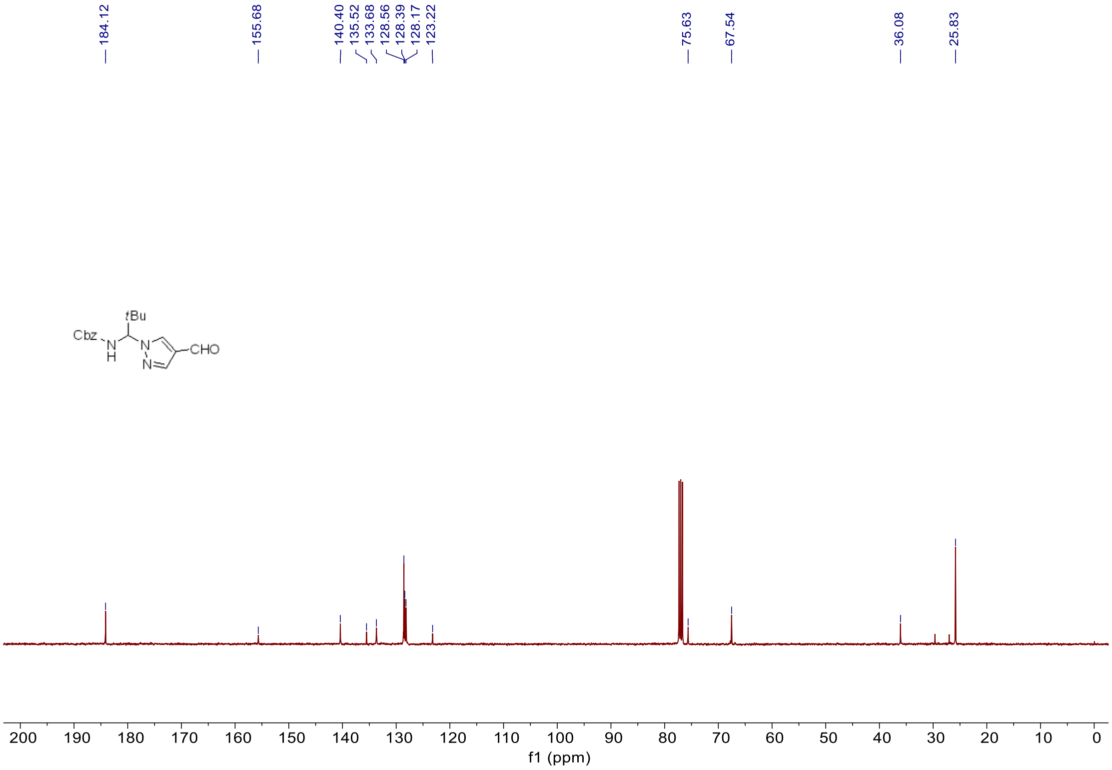
^{13}C NMR spectrum (101 MHz) of **14c** (see procedure)



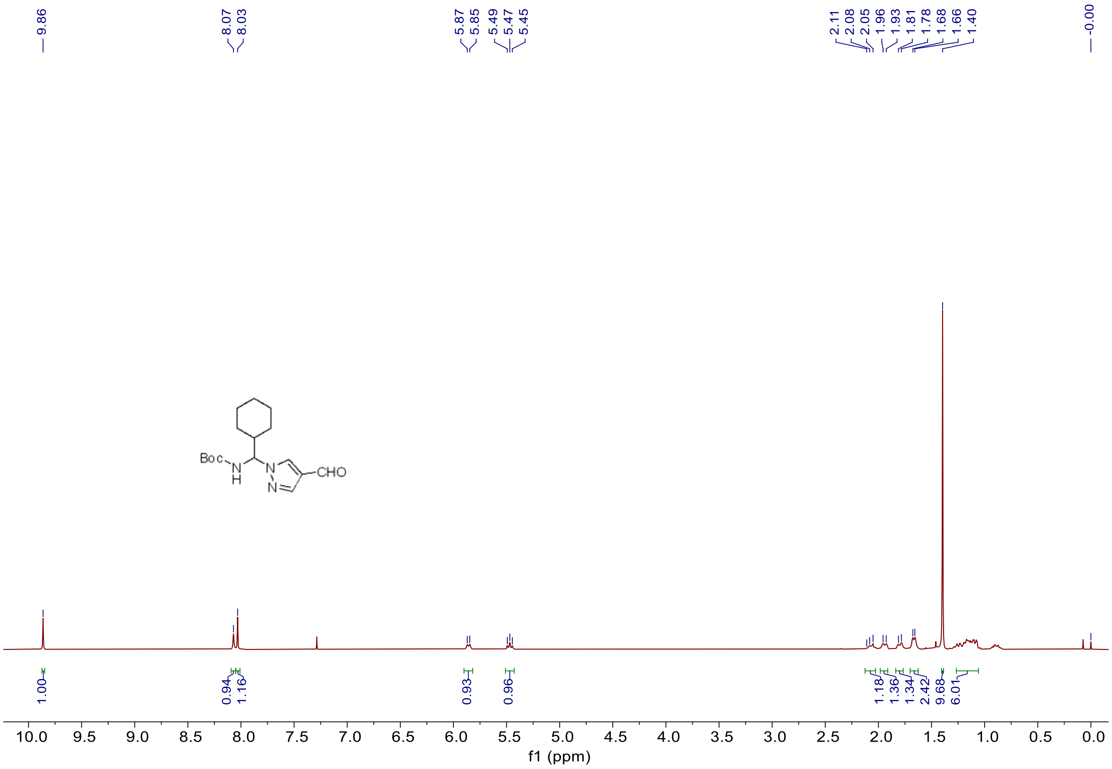
^{1}H NMR (400 MHz) spectrum of **15c** (see procedure)



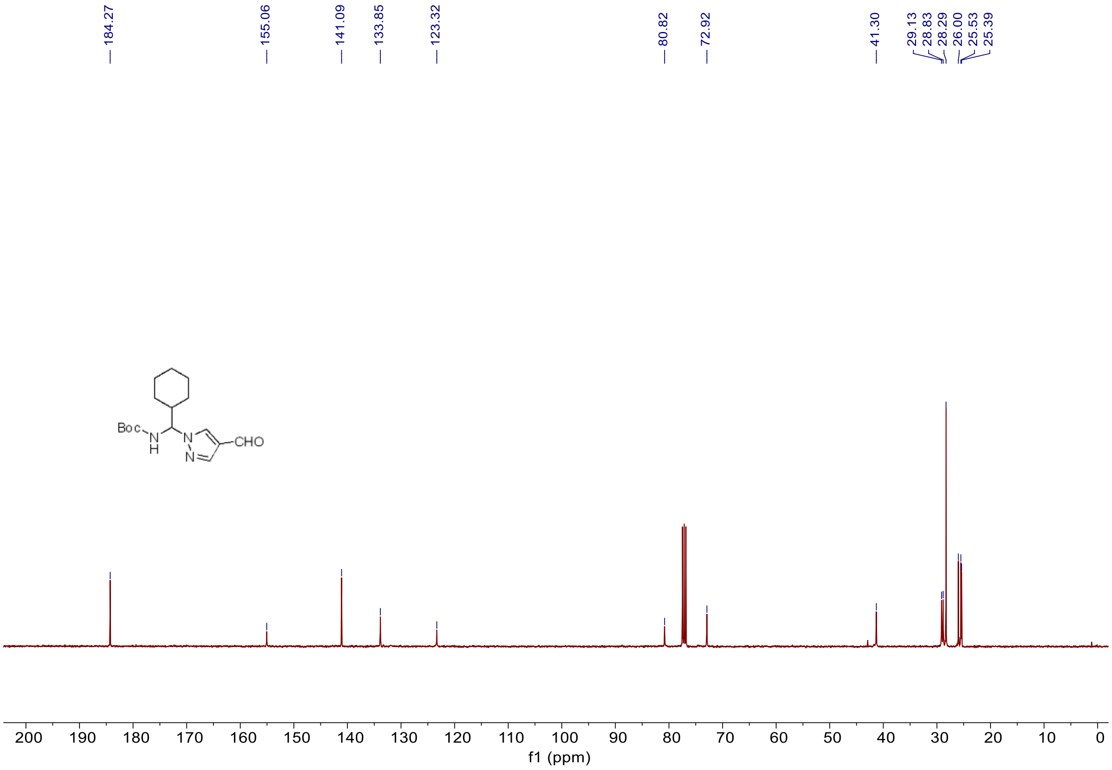
^{13}C NMR spectrum (101 MHz) of **15c** (see procedure)



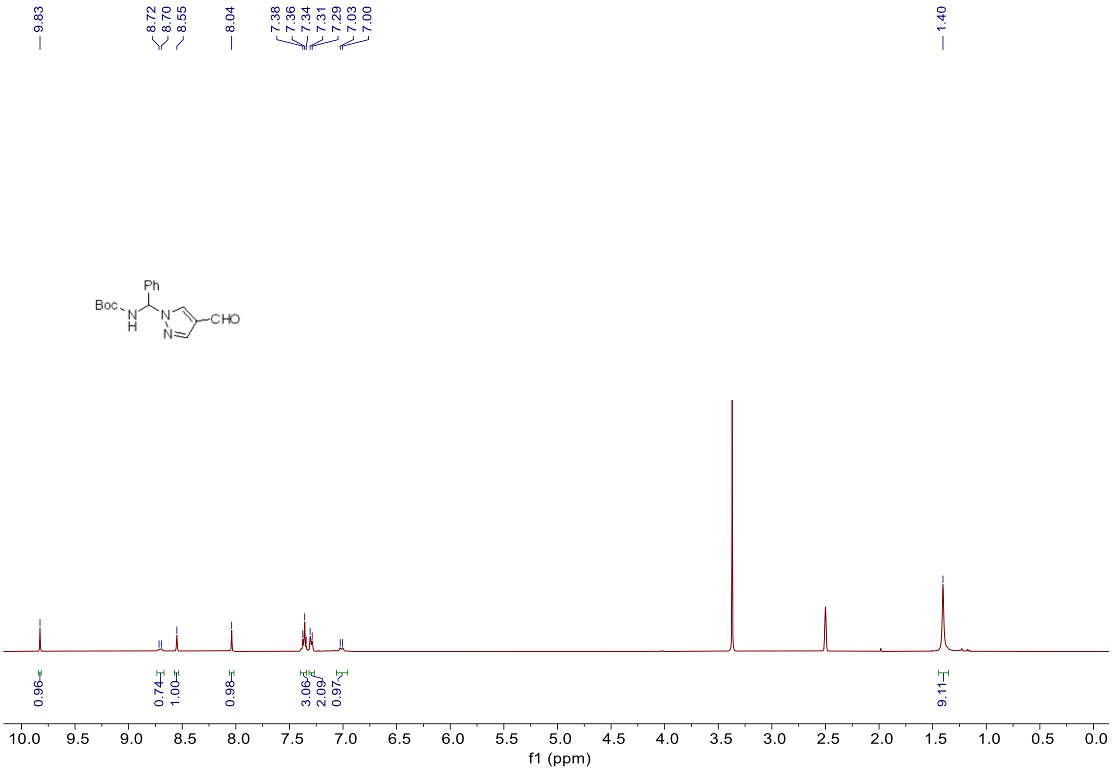
^{1}H NMR spectrum (400 MHz) of **16c** (see procedure)



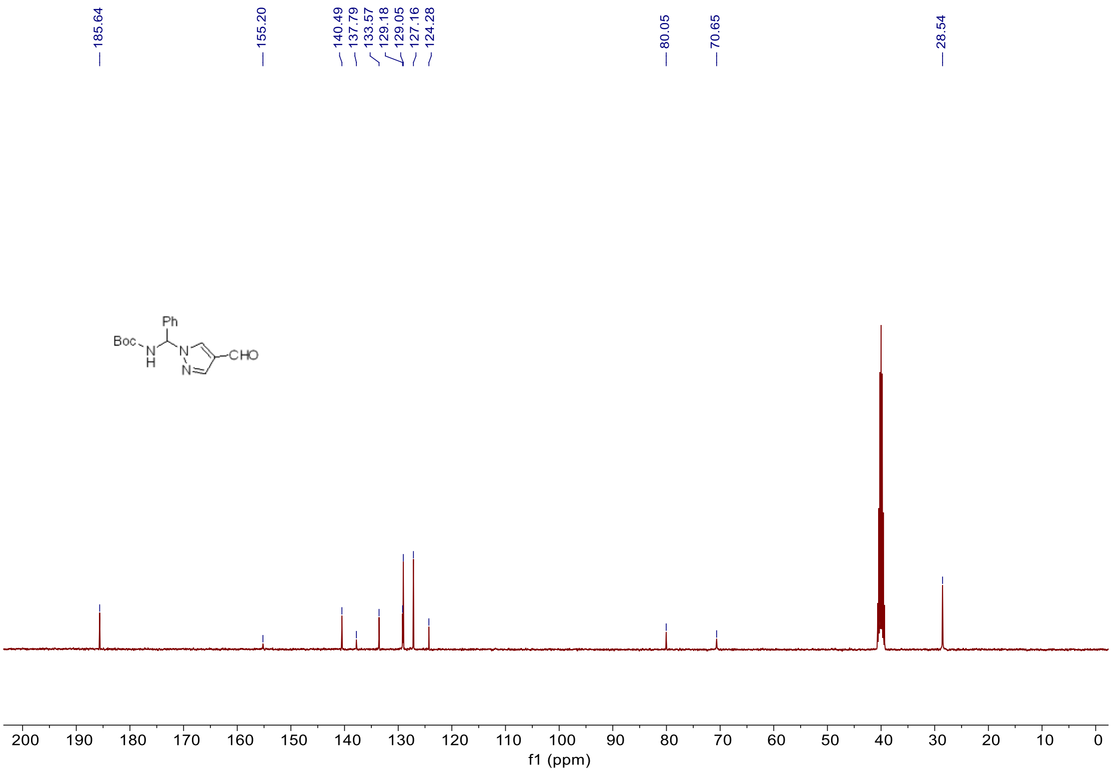
^{13}C NMR spectrum (101 MHz) of **16c** (see procedure)



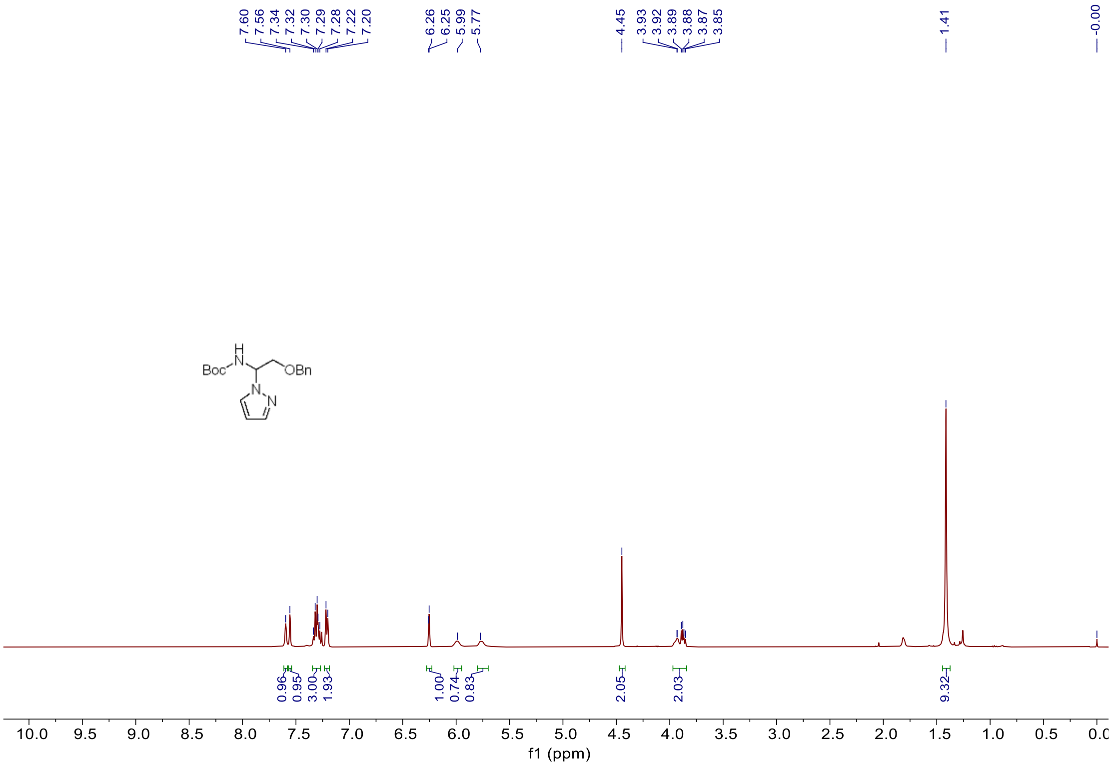
^{1}H NMR spectrum (400 MHz) of **17c** (see procedure)



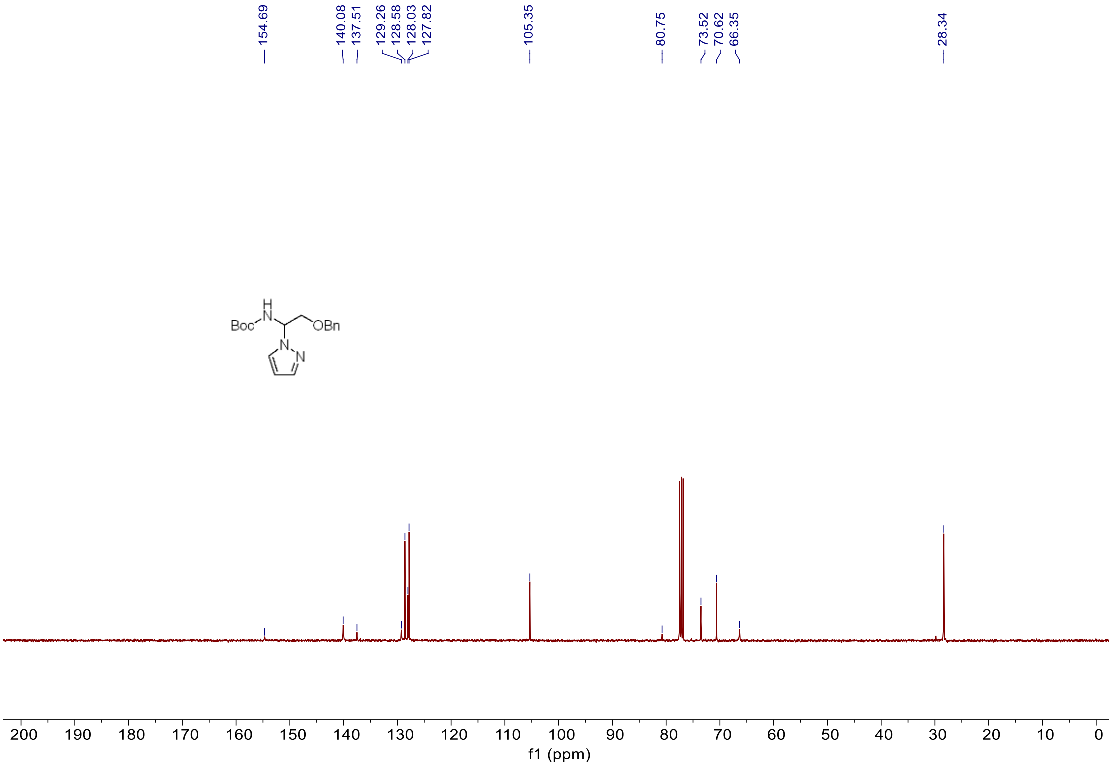
^{13}C NMR spectrum (101 MHz) of **17c** (see procedure)



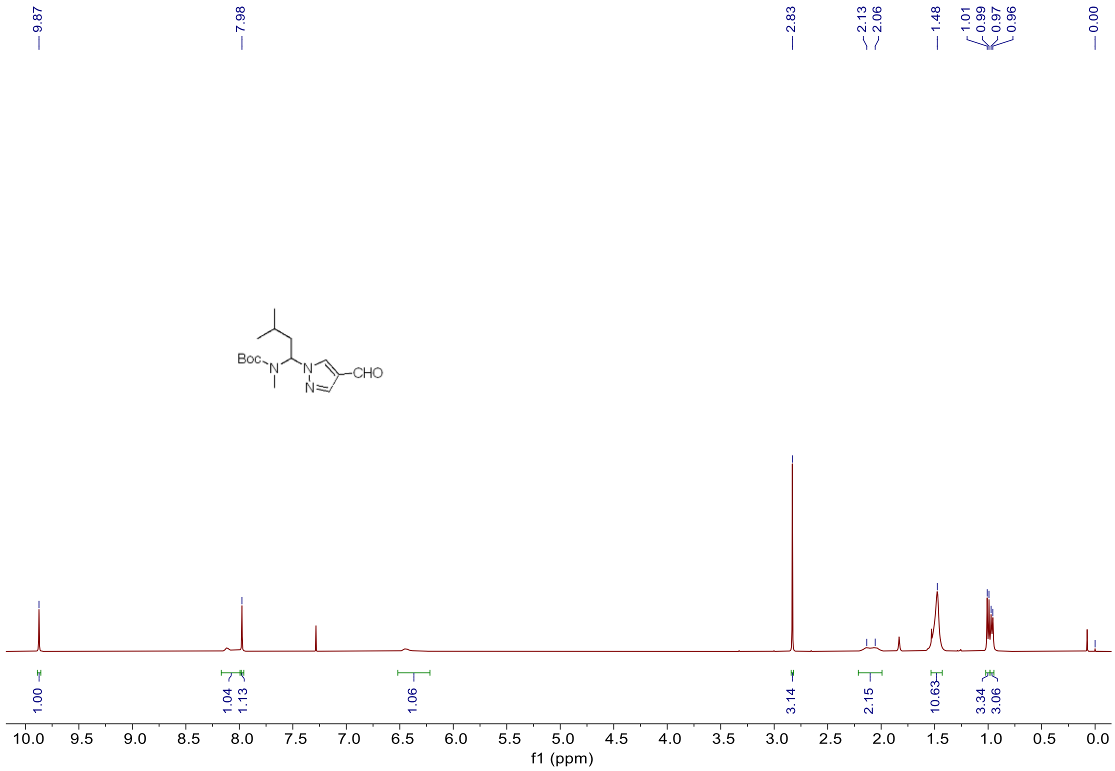
^{1}H NMR spectrum (400 MHz) of **18c** (see procedure)



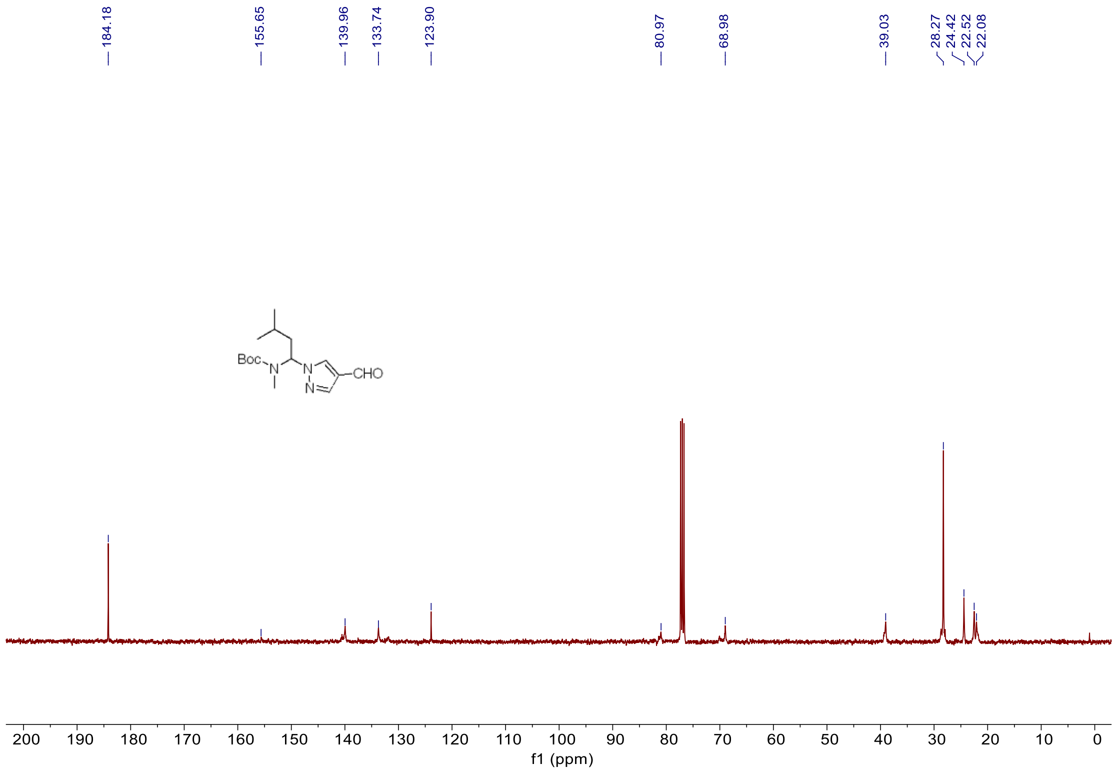
^{13}C NMR spectrum (101 MHz) of **18c** (see procedure)



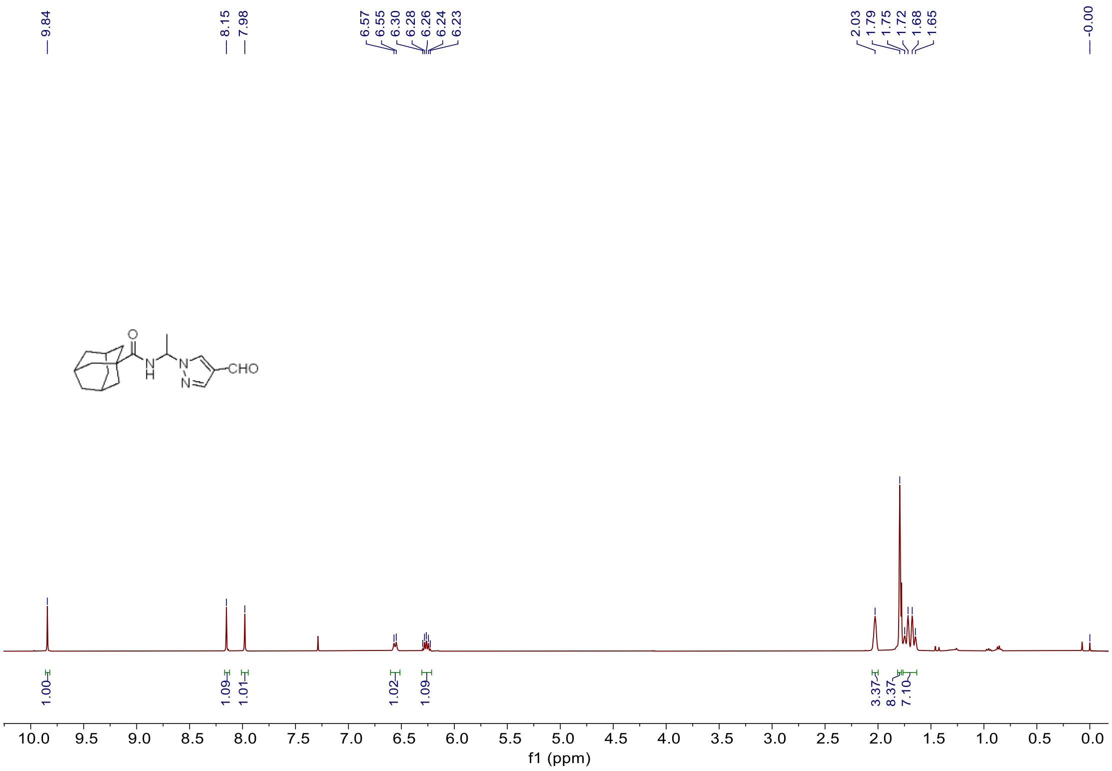
^{1}H NMR spectrum (400 MHz) of **19c** (see procedure)



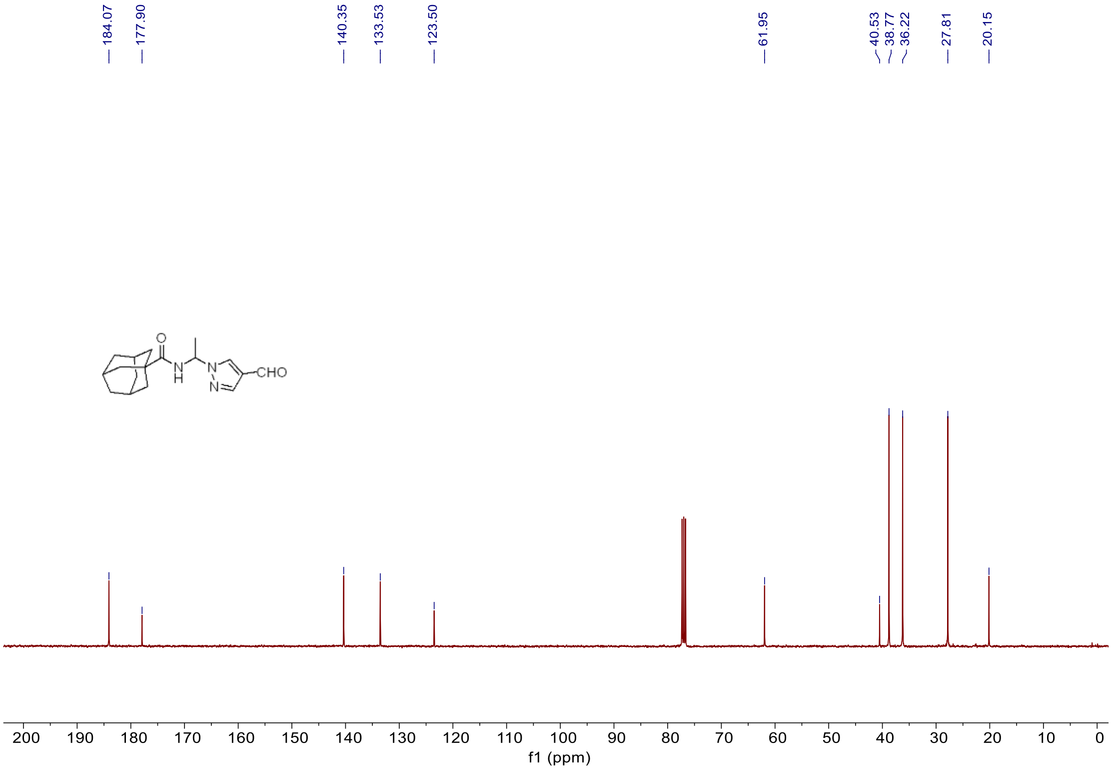
^{13}C NMR spectrum (101 MHz) of **19c** (see procedure)



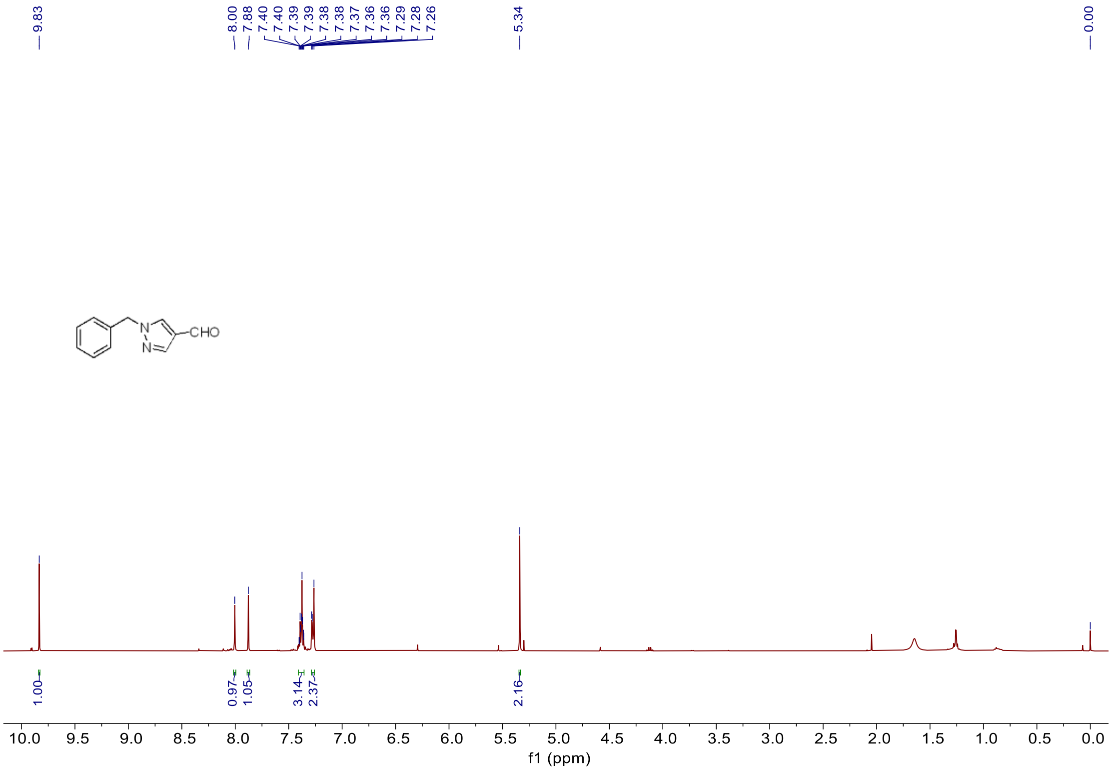
^{1}H NMR spectrum (400 MHz) of **20c** (see procedure)



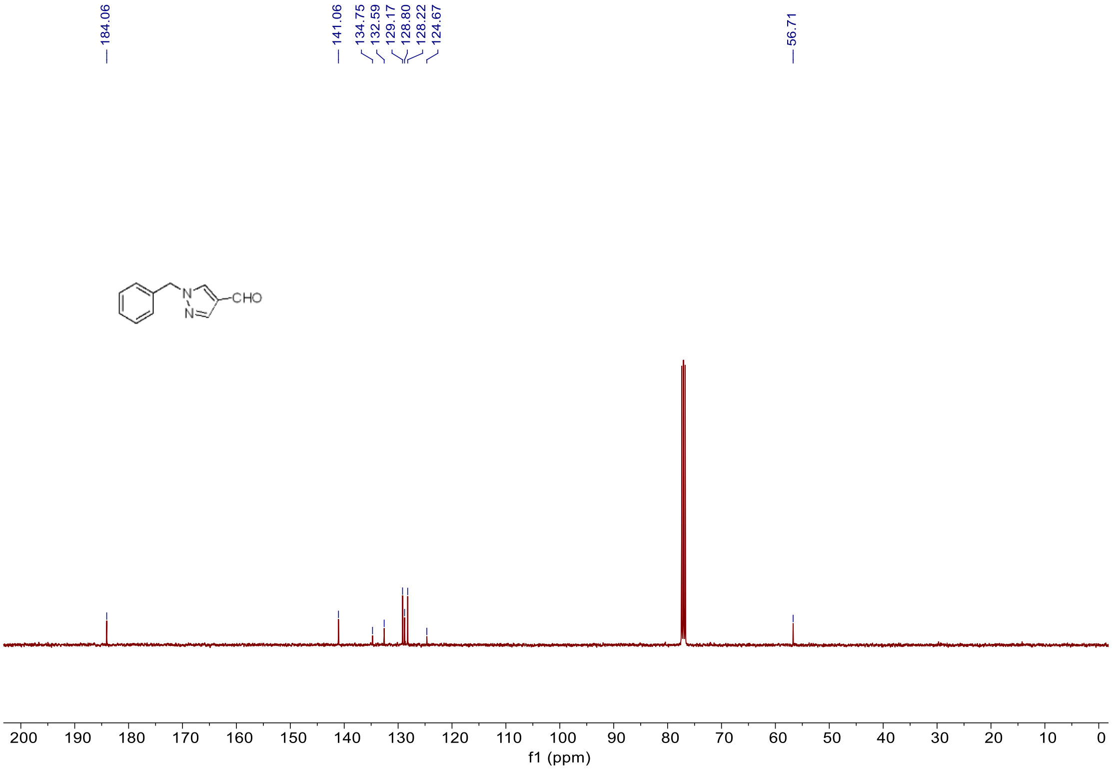
^{13}C NMR spectrum (101 MHz) of **20c** (see procedure)



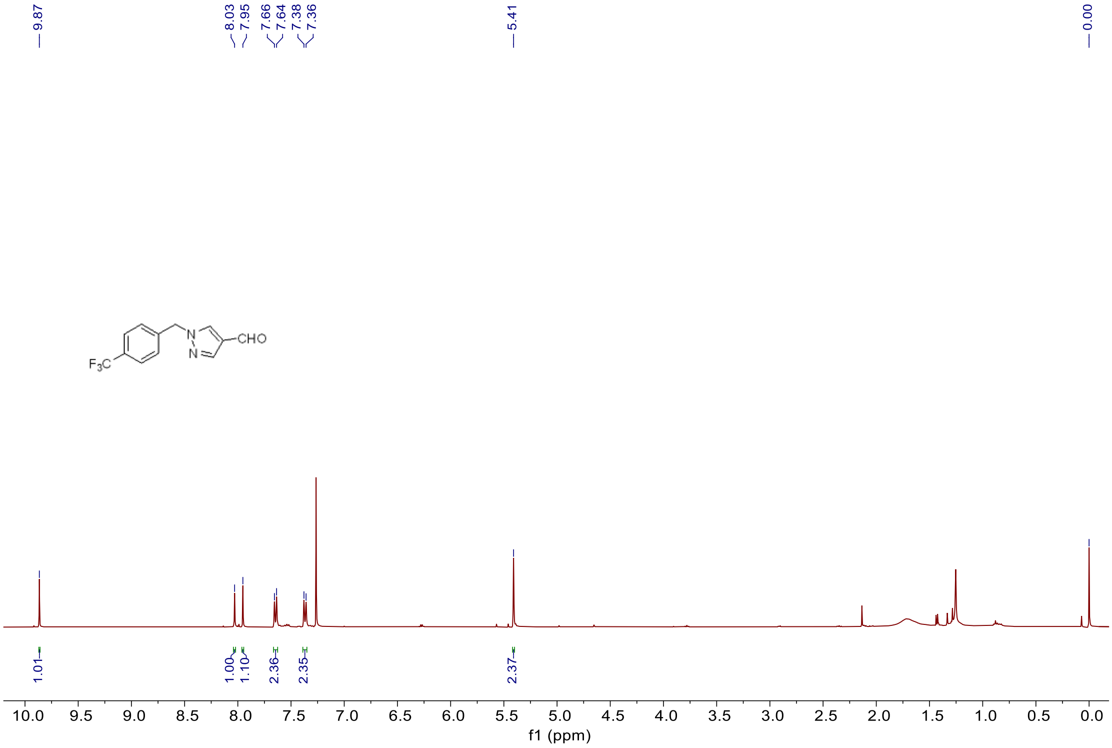
^{1}H NMR spectrum (400 MHz) of **21c** (see procedure)



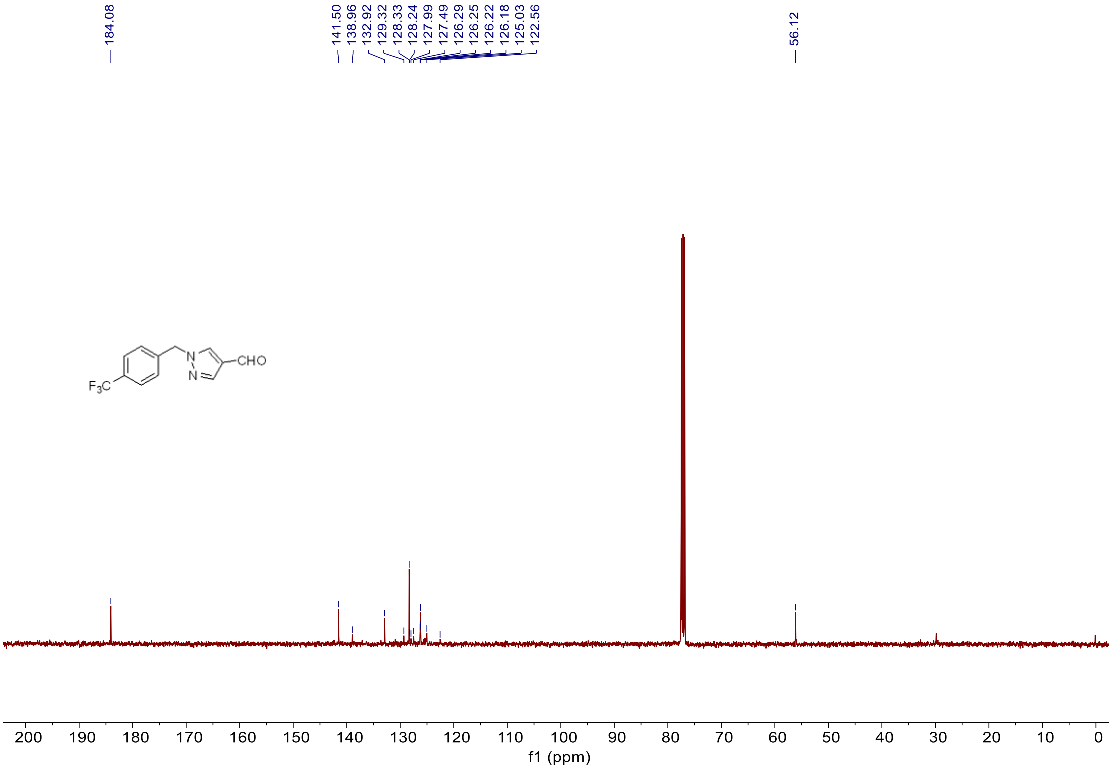
^{13}C NMR spectrum (101 MHz) of **21c** (see procedure)



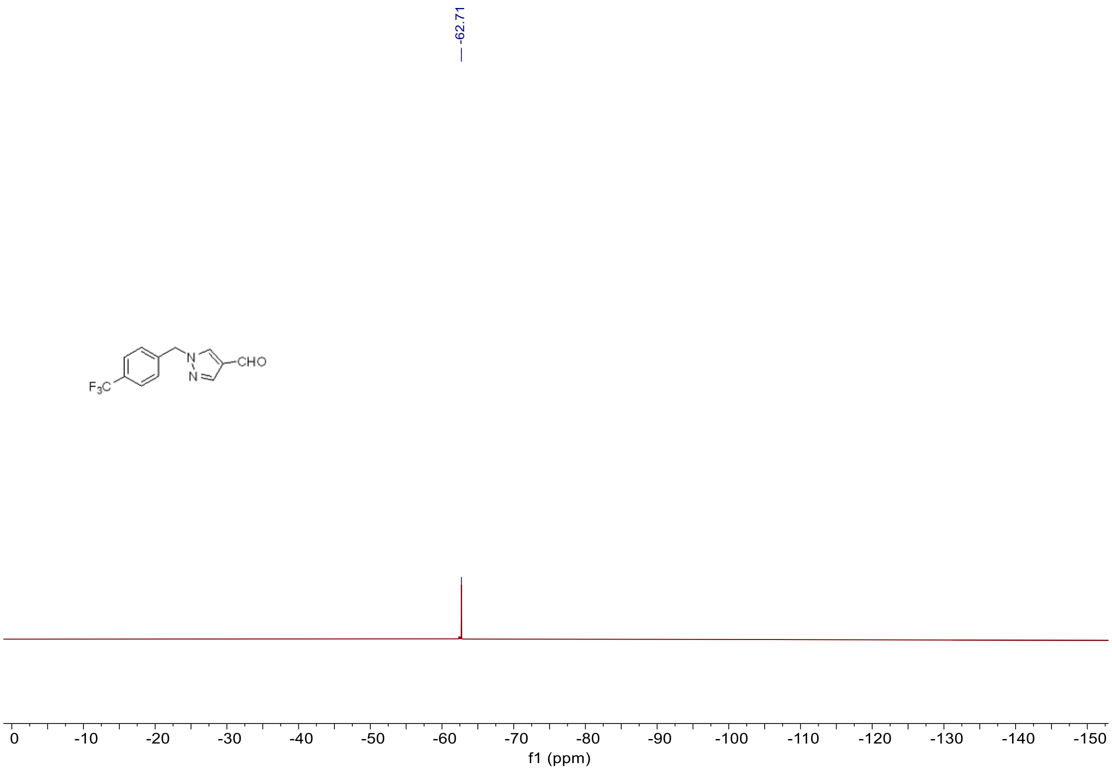
^{1}H NMR spectrum (400 MHz) of **22c** (see procedure)



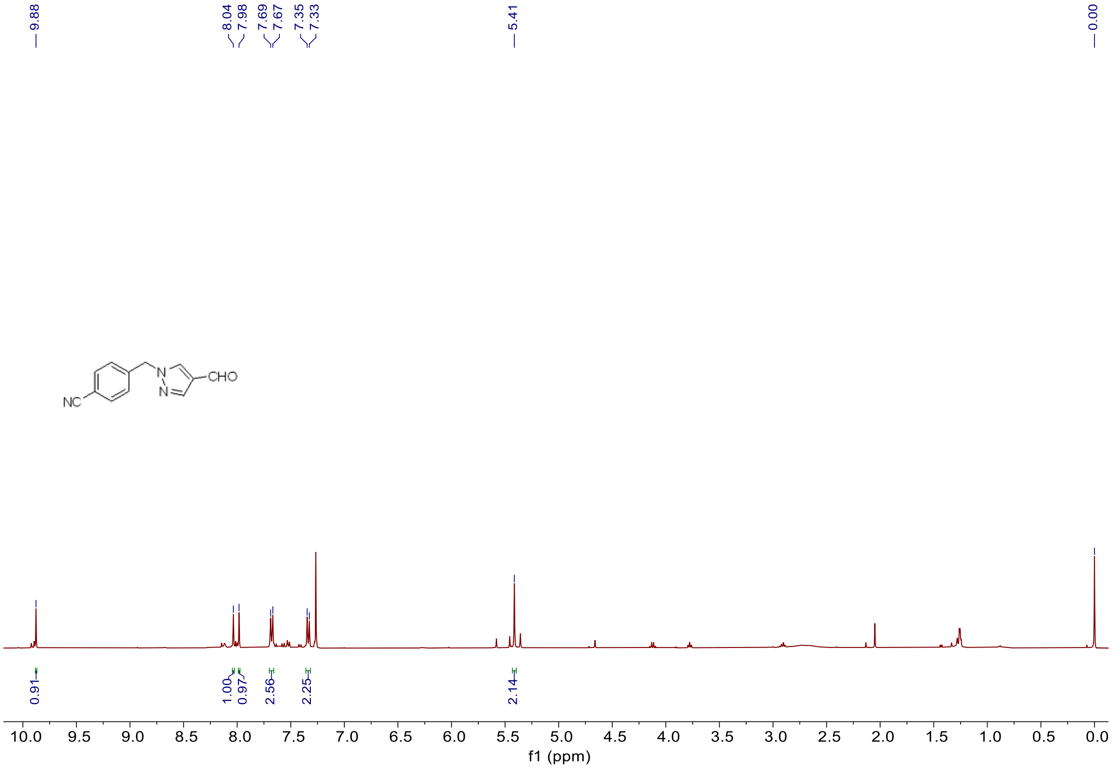
^{13}C NMR spectrum (101 MHz) of **22c** (see procedure)



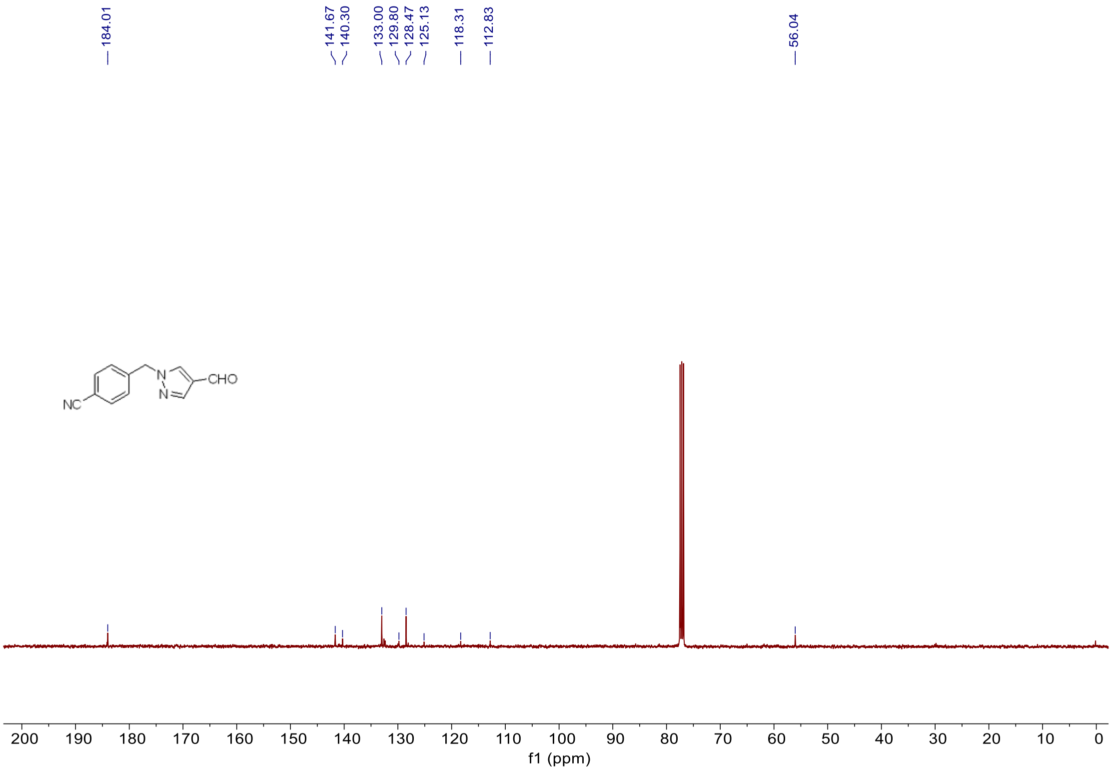
^{19}F NMR spectrum (376 MHz) of **22c** (see procedure)



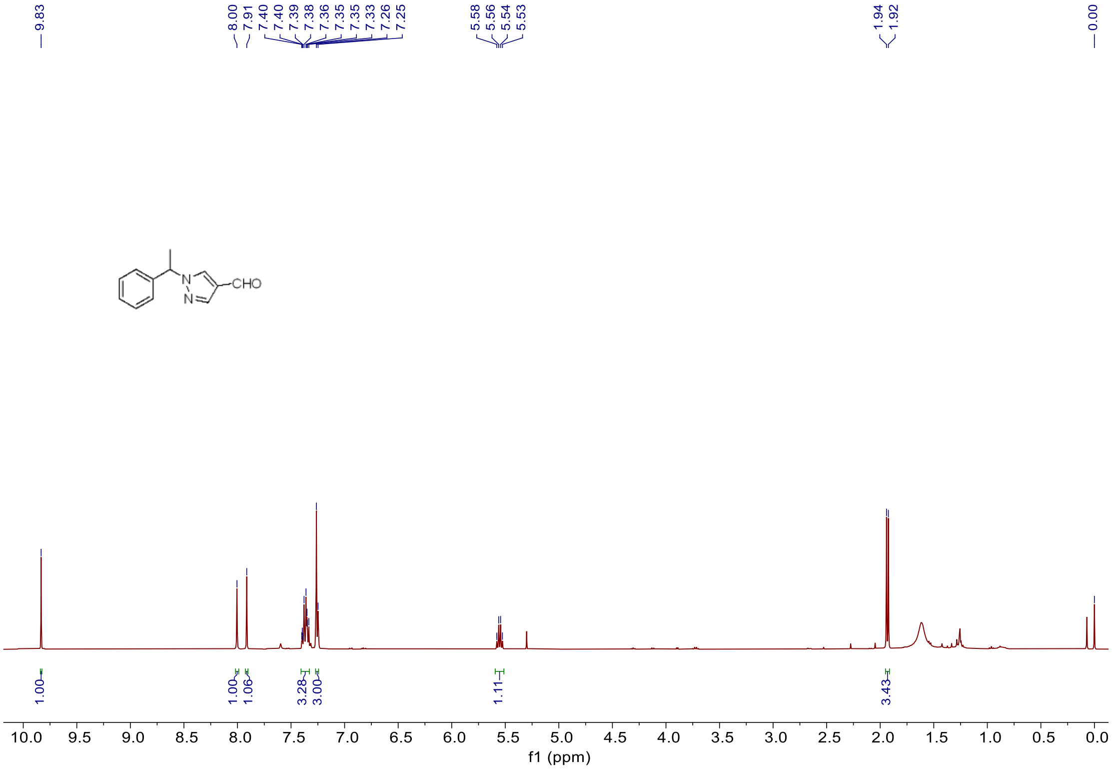
^{1}H NMR spectrum (400 MHz) of **23c** (see procedure)



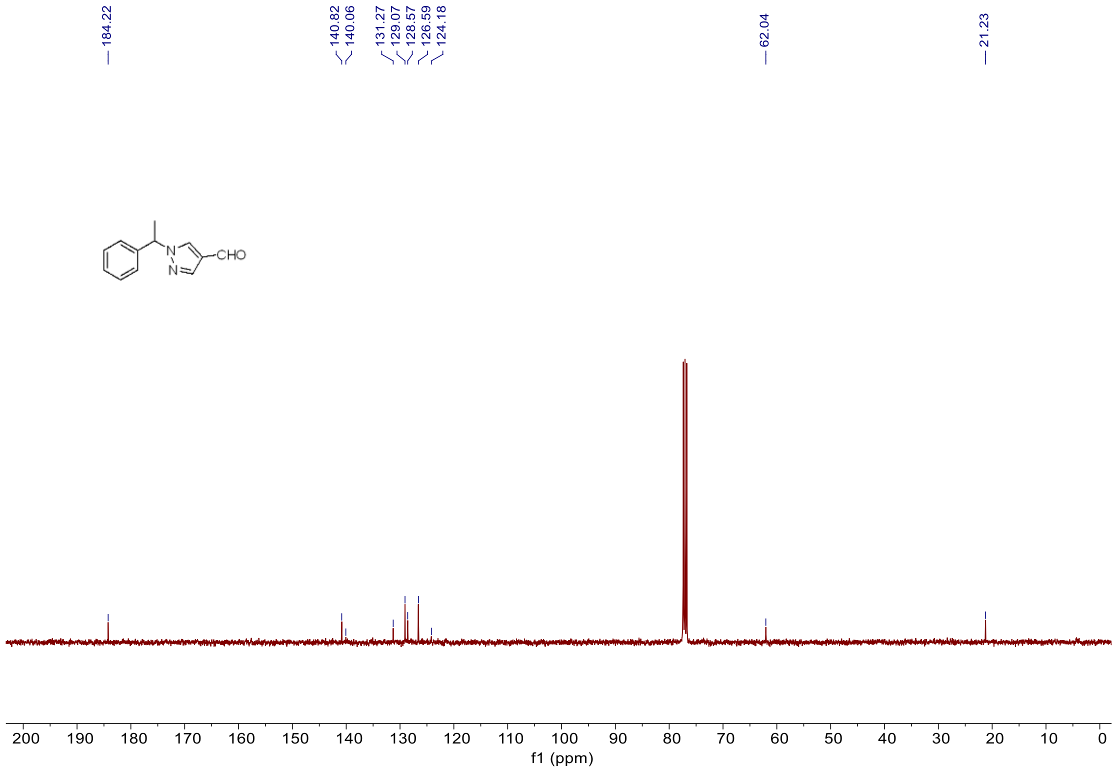
^{13}C NMR spectrum (101 MHz) of **23c** (see procedure)



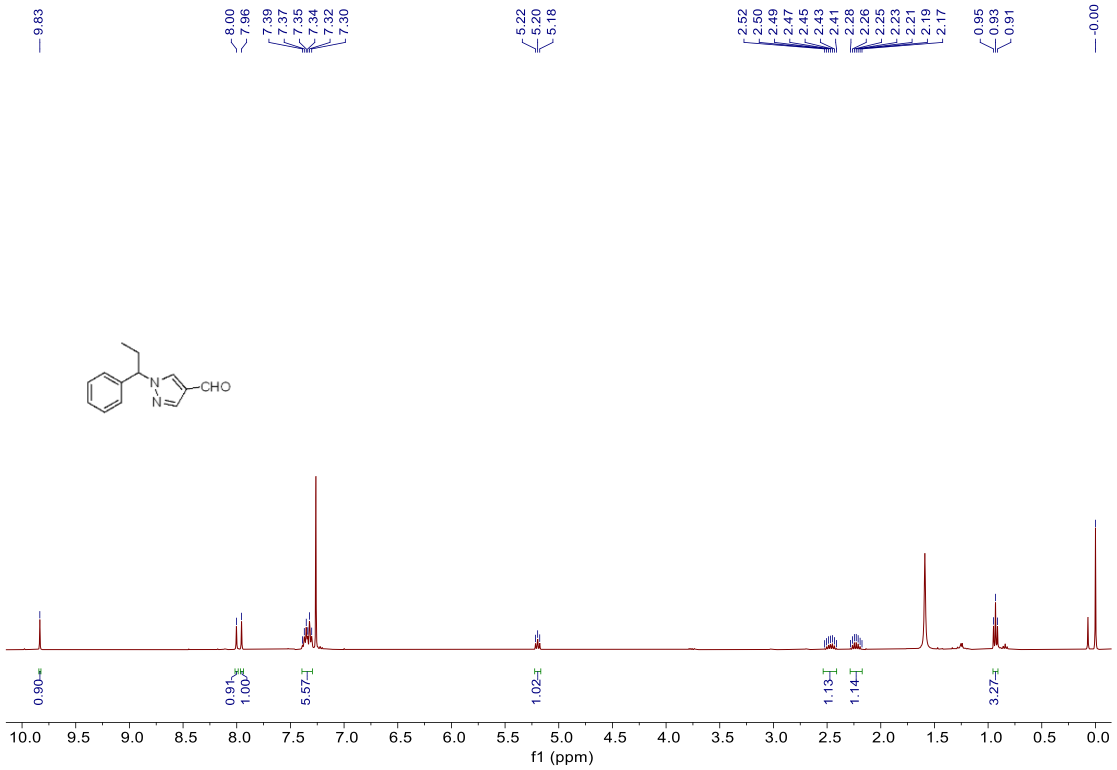
^{1}H NMR spectrum (400 MHz) of **24c** (see procedure)



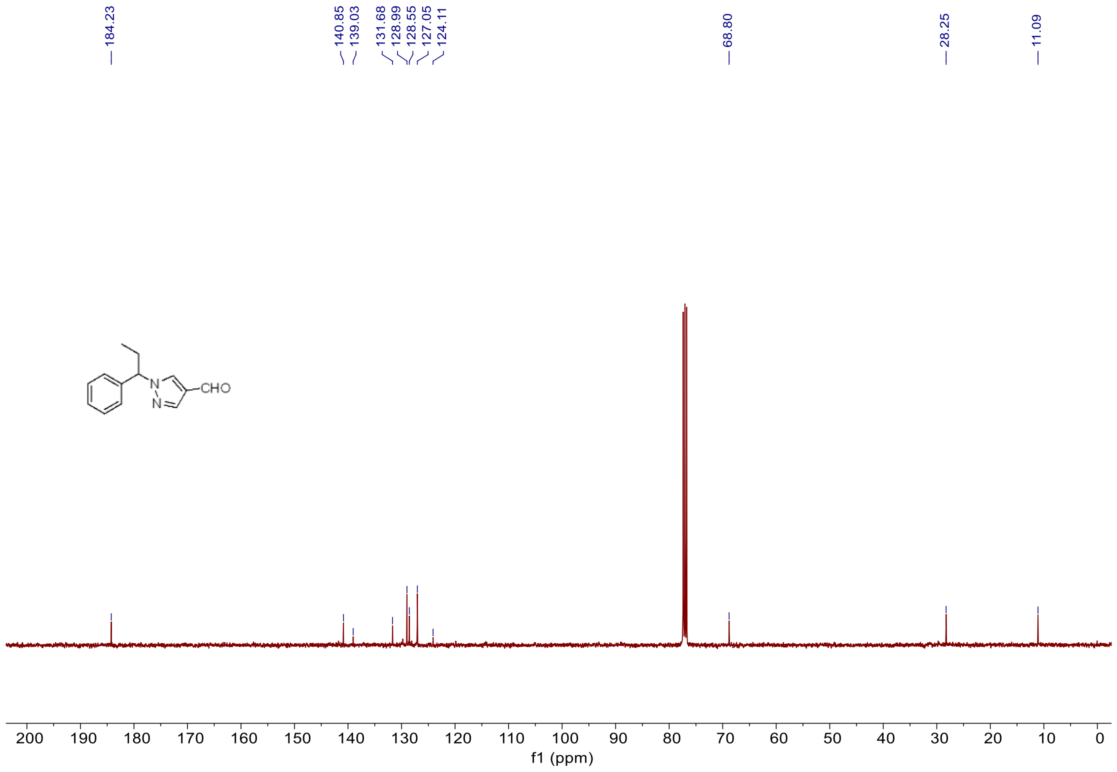
^{13}C NMR spectrum (101 MHz) of **24c** (see procedure)



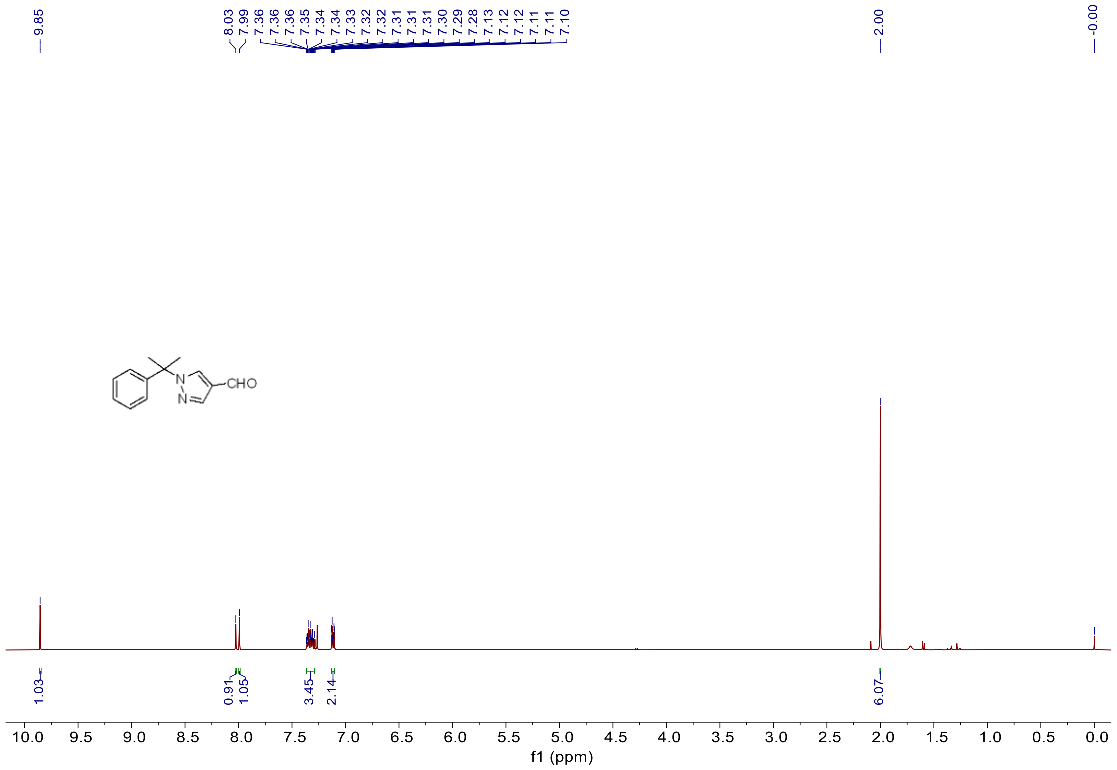
^{1}H NMR spectrum (400 MHz) of **25c** (see procedure)



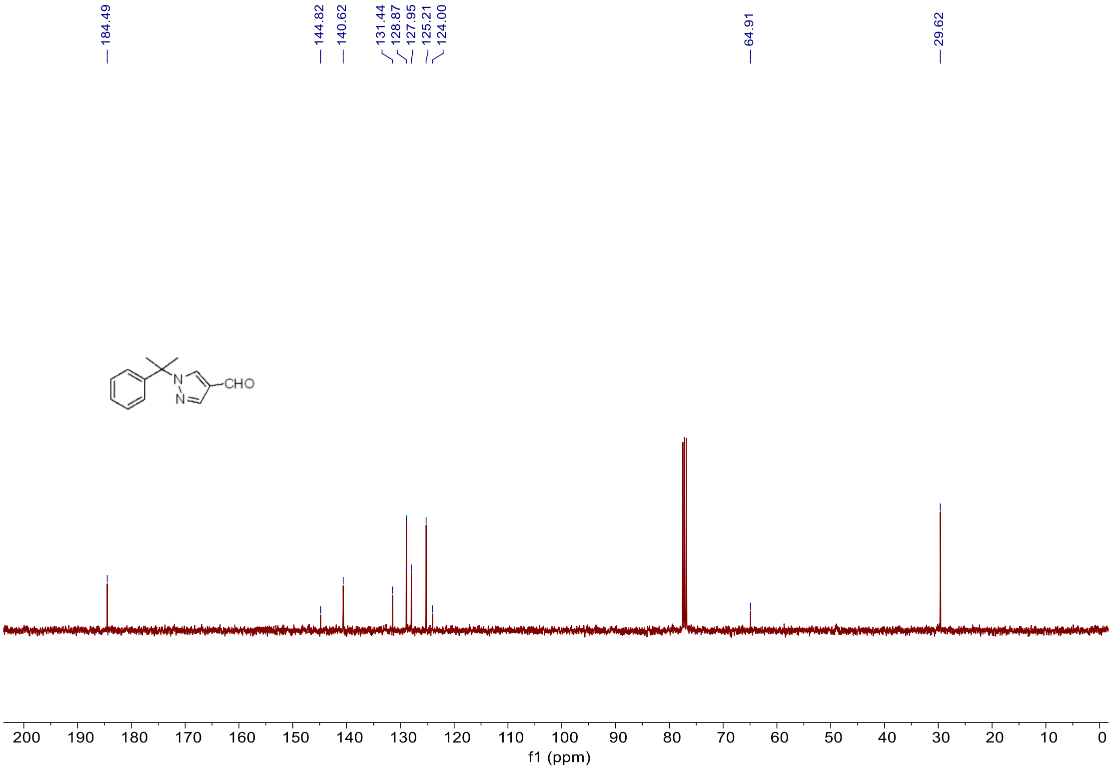
^{13}C NMR spectrum (101 MHz) of **25c** (see procedure)



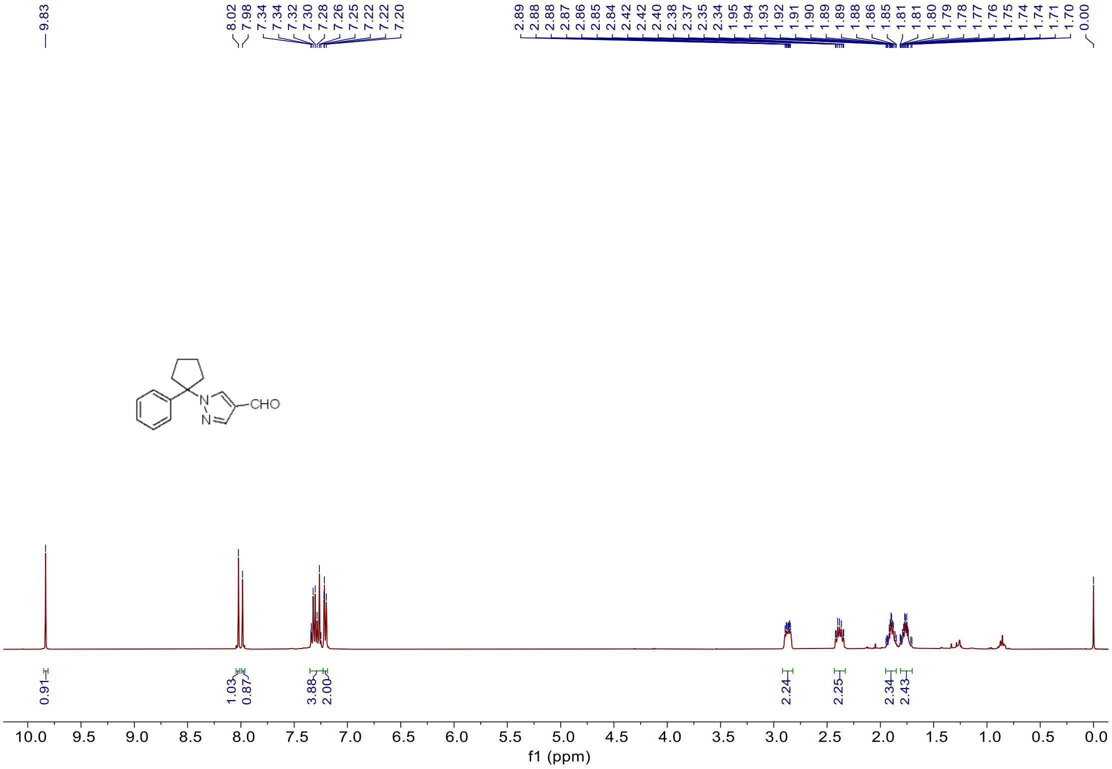
^{1}H NMR spectrum (400 MHz) of **26c** (see procedure)



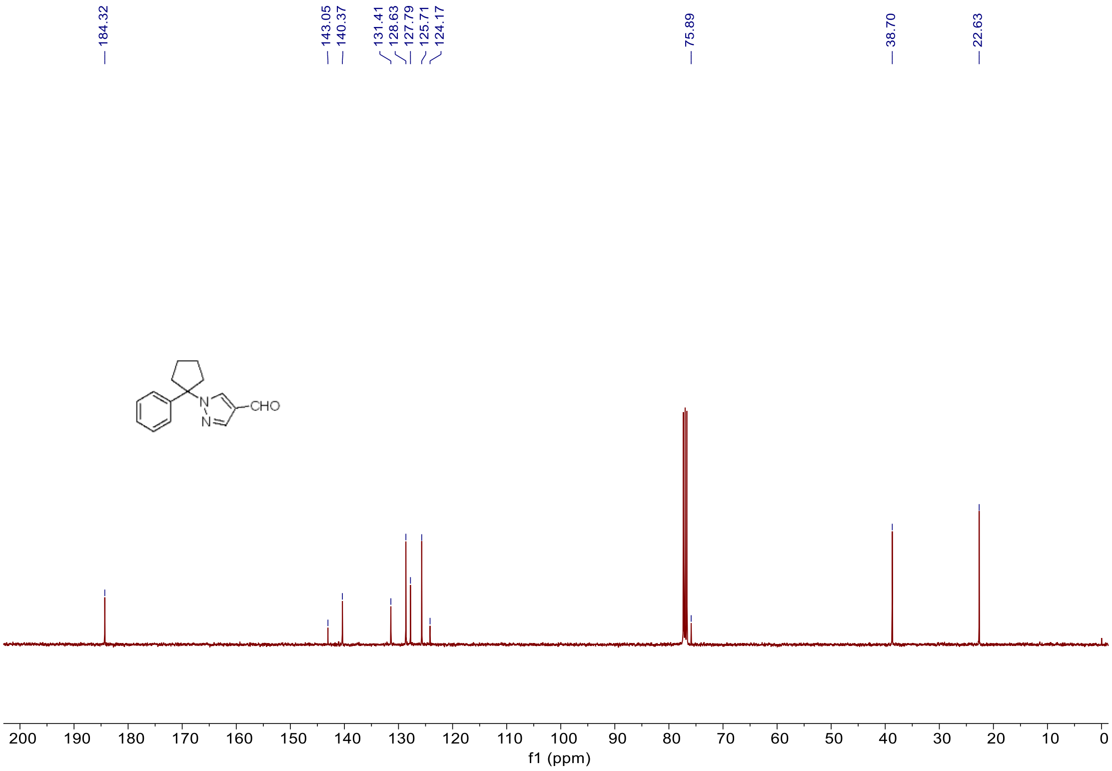
^{13}C NMR spectrum (101 MHz) of **26c** (see procedure)



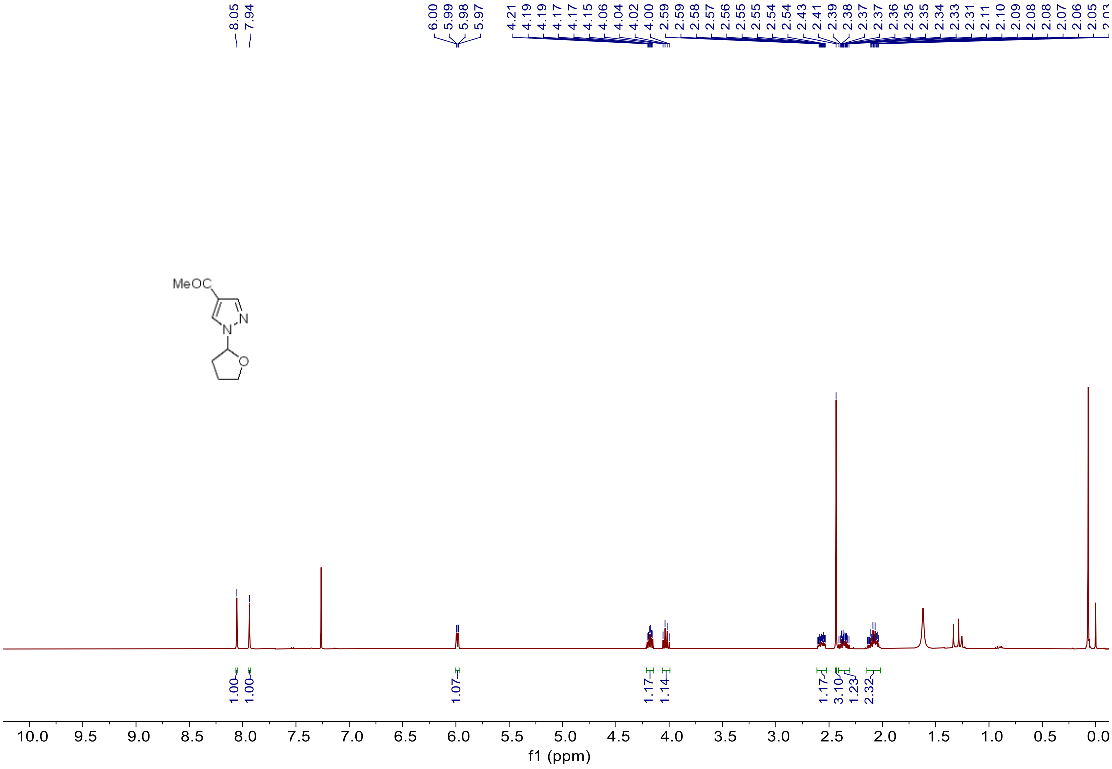
^{1}H NMR spectrum (400 MHz) of **27c** (see procedure)



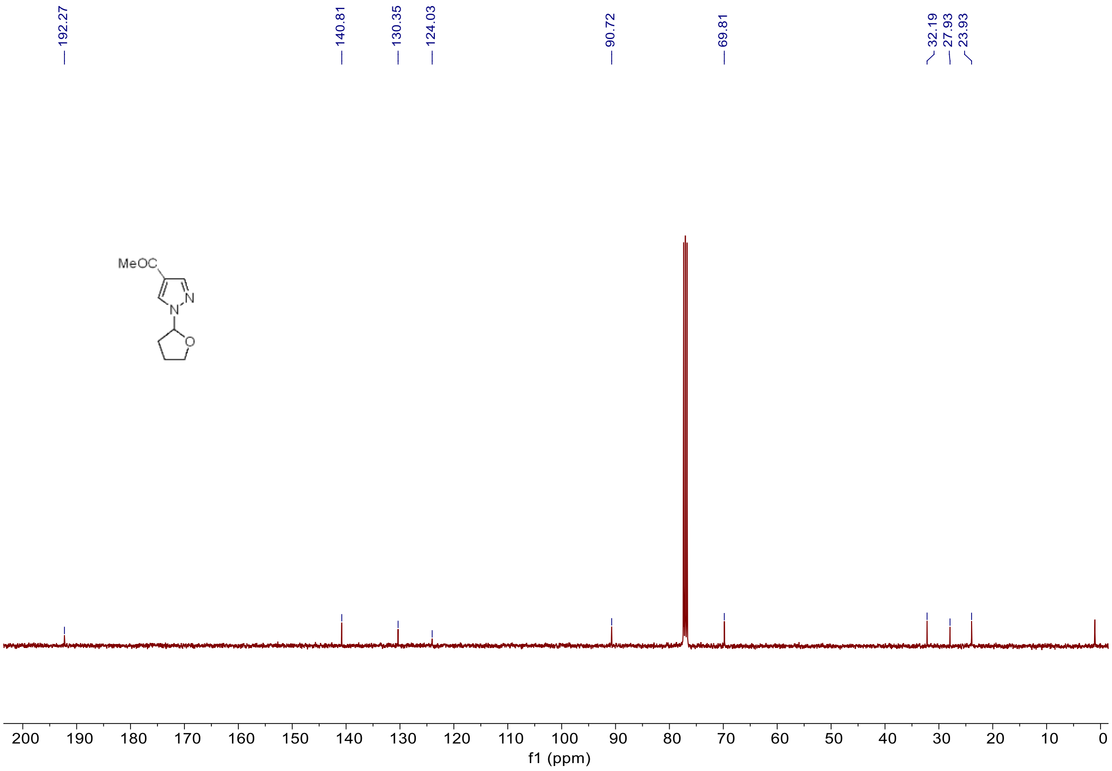
^{13}C NMR spectrum (101 MHz) of **27c** (see procedure)



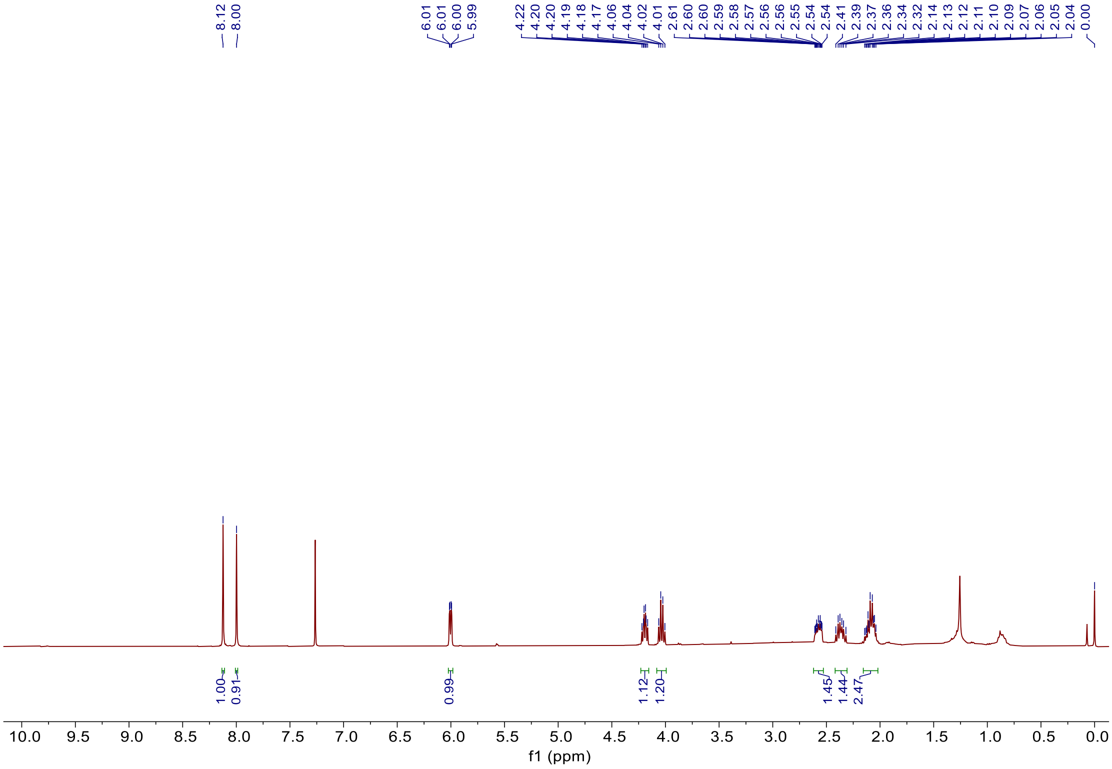
^{1}H NMR spectrum (400 MHz) of **28c** (see procedure)



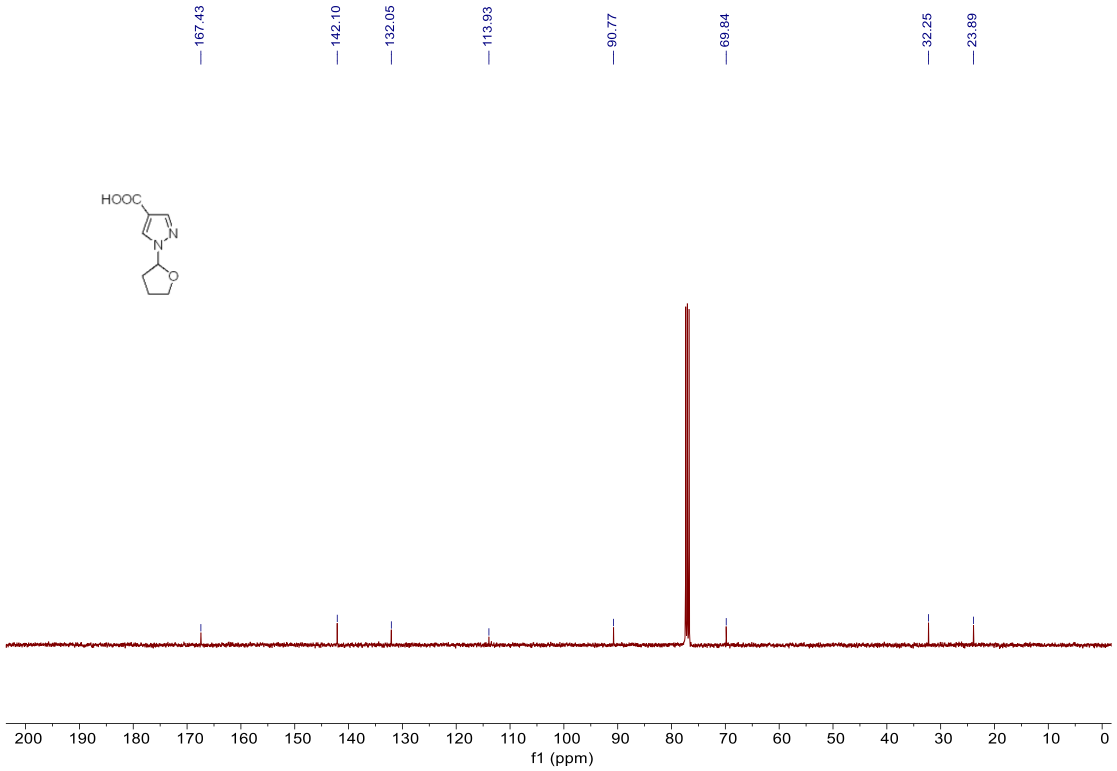
^{13}C NMR spectrum (101 MHz) of **28c** (see procedure)



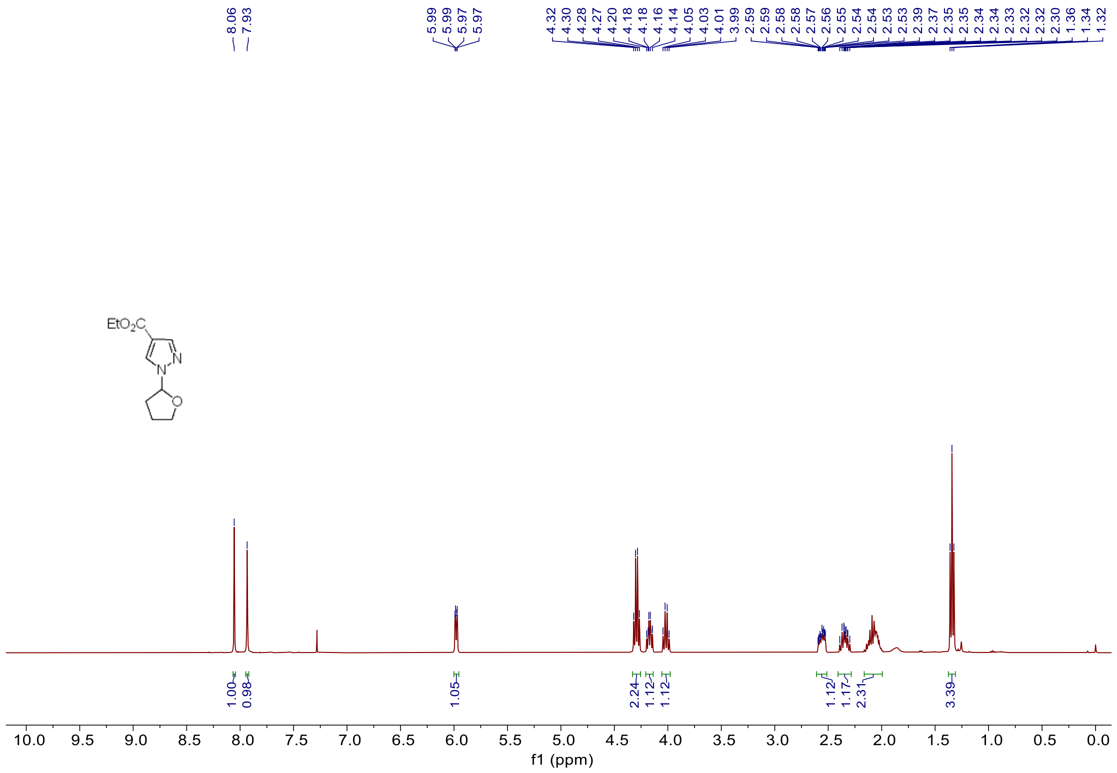
^{1}H NMR spectrum (400 MHz) of **29c** (see procedure)



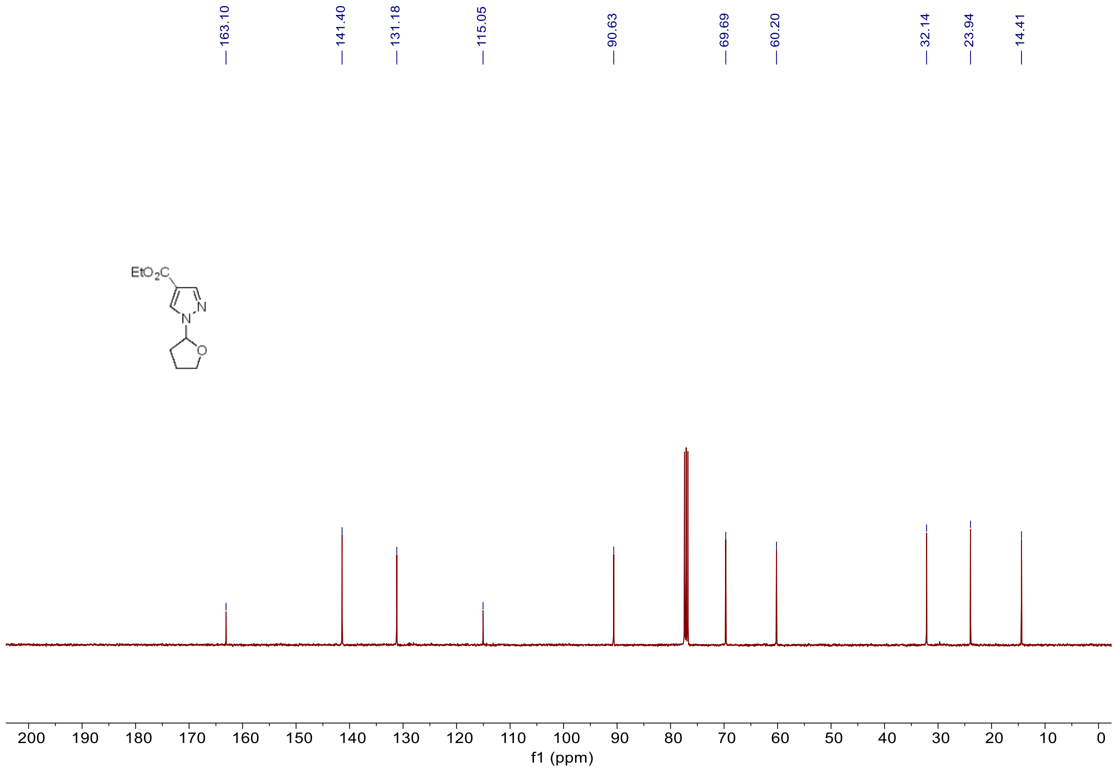
^{13}C NMR spectrum (101 MHz) of **29c** (see procedure)



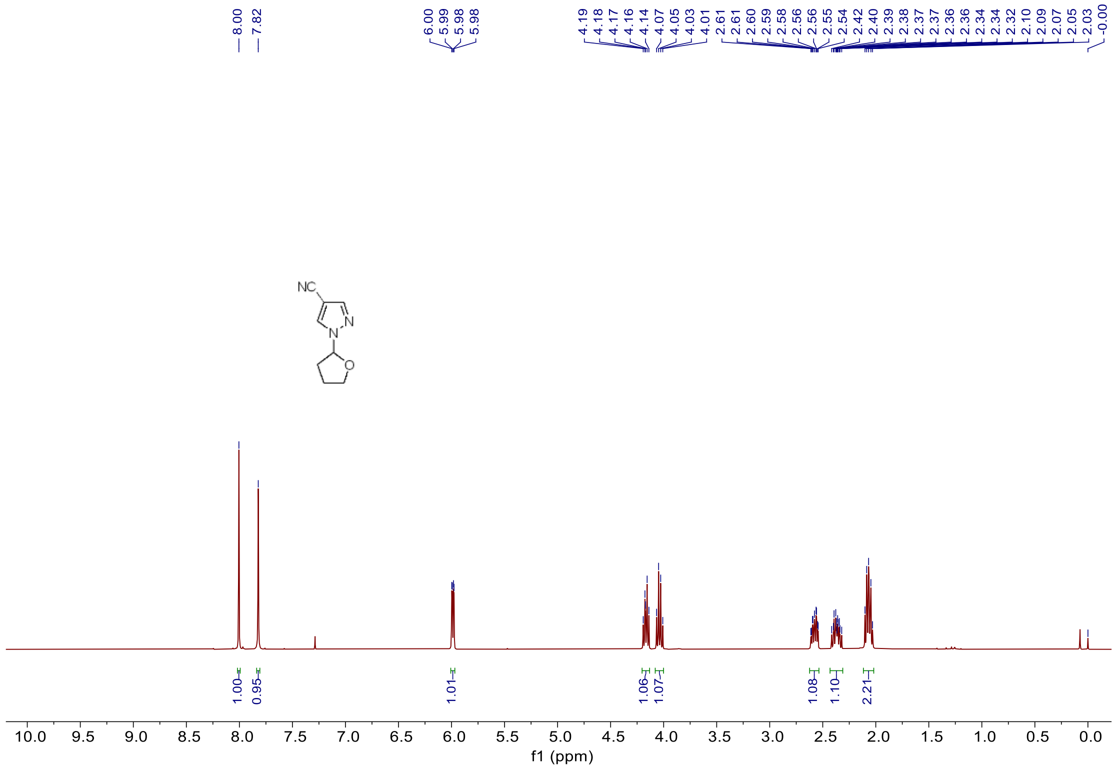
^{1}H NMR spectrum (400 MHz) of **30c** (see procedure)



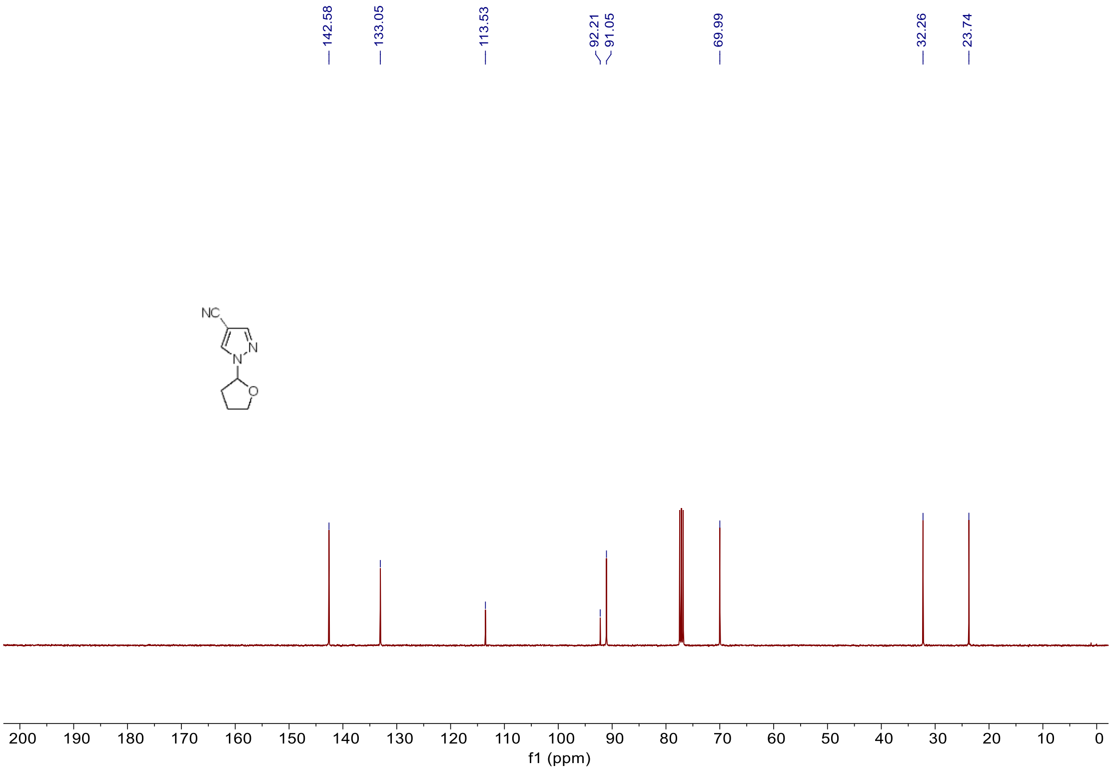
^{13}C NMR spectrum (101 MHz) of **30c** (see procedure)



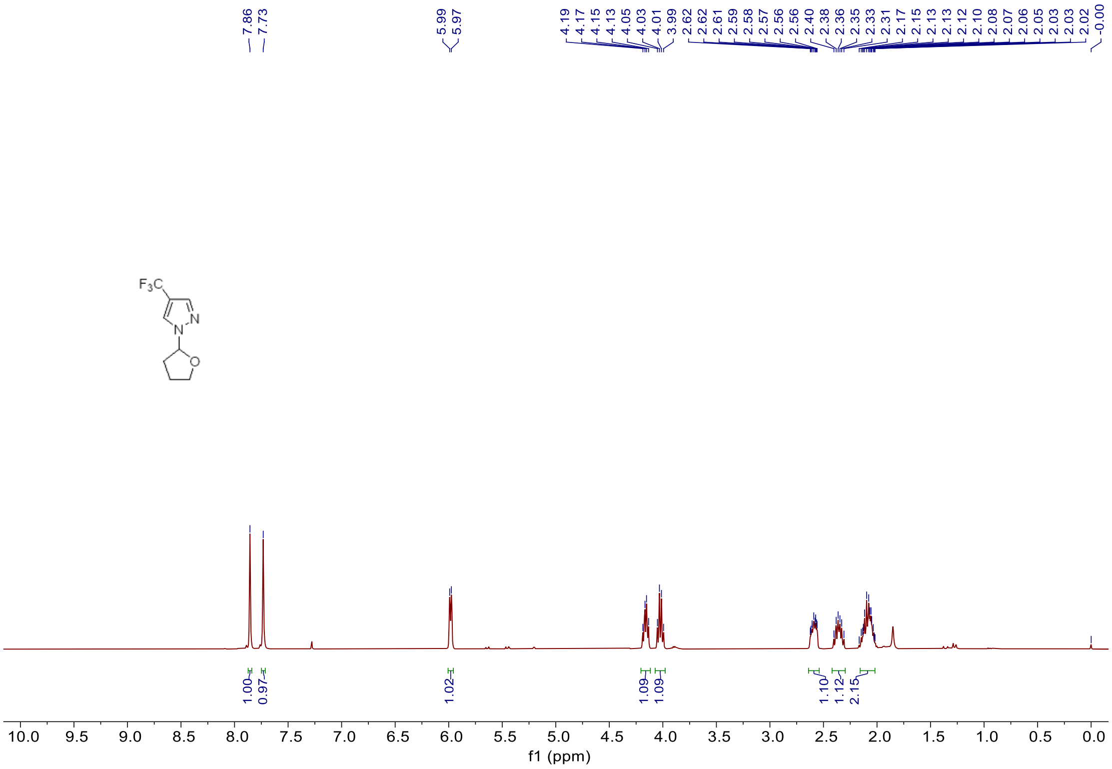
^{1}H NMR spectrum (400 MHz) of **31c** (see procedure)



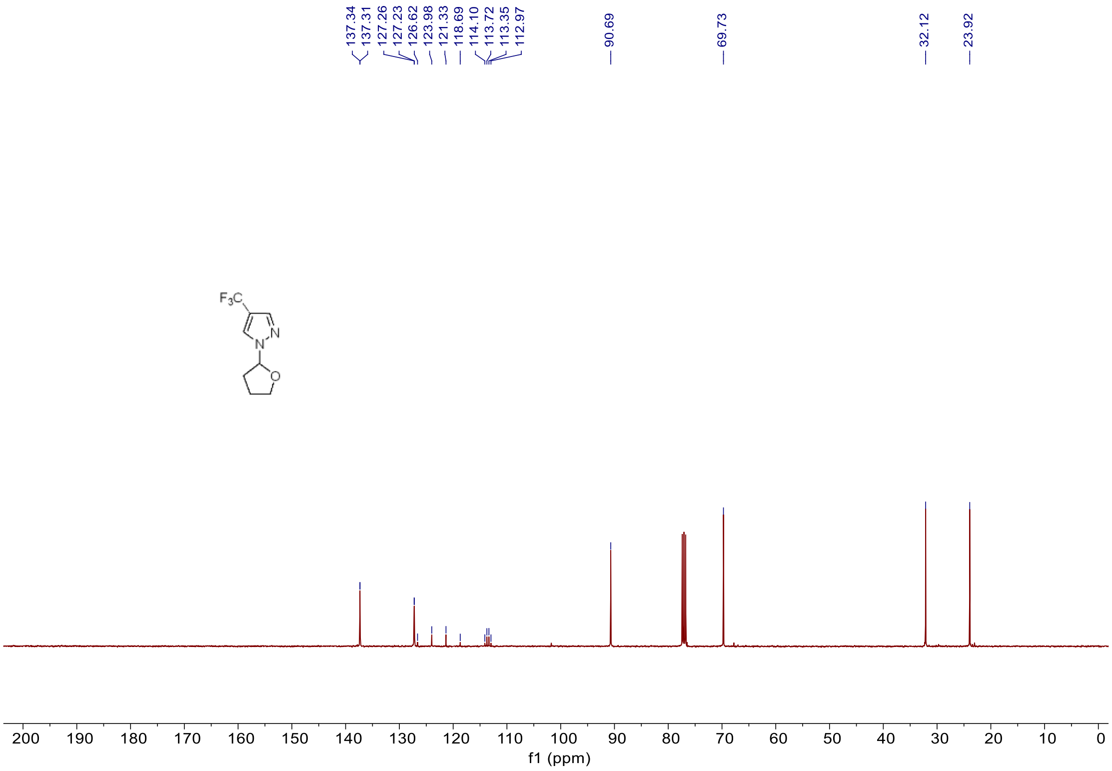
^{13}C NMR spectrum (101 MHz) of **31c** (see procedure)



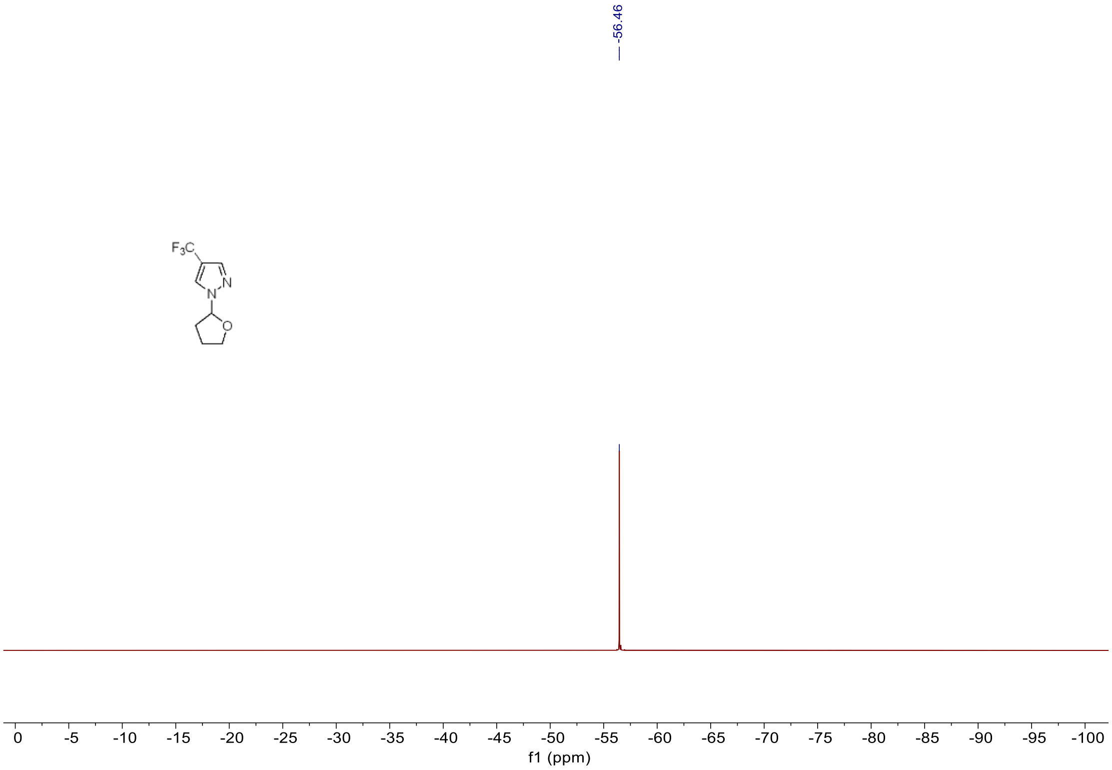
^{1}H NMR spectrum (400 MHz) of **32c** (see procedure)



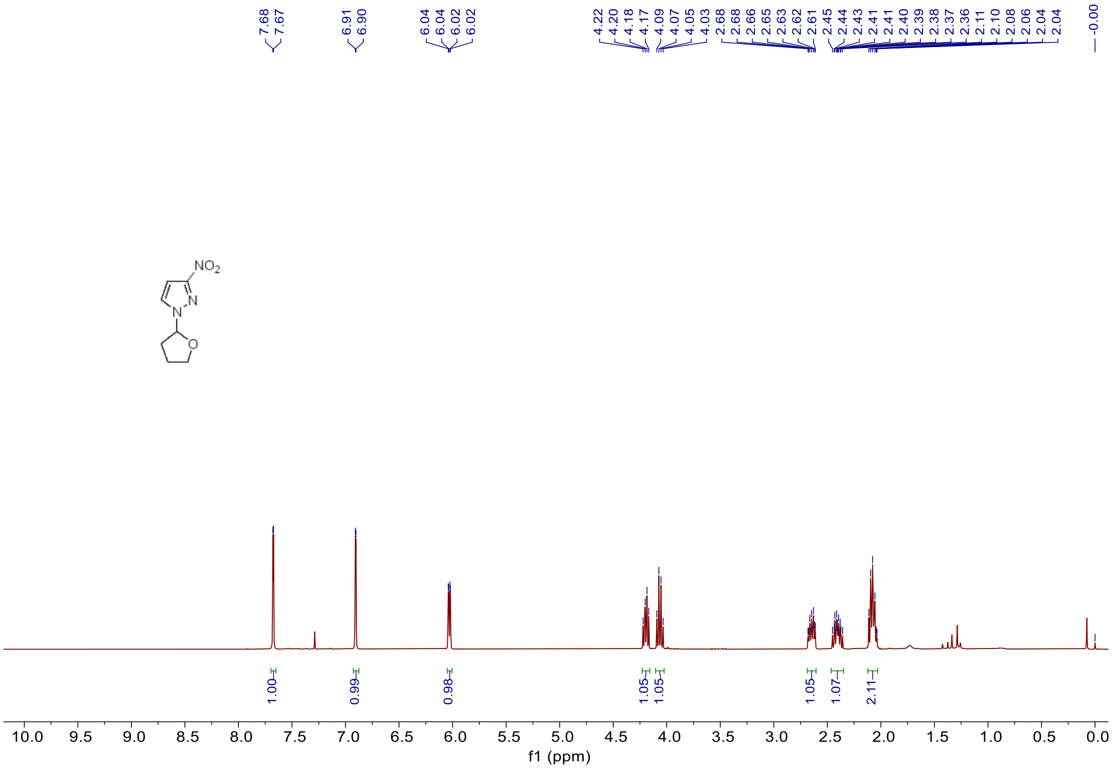
^{13}C NMR spectrum (101 MHz) of **32c** (see procedure)



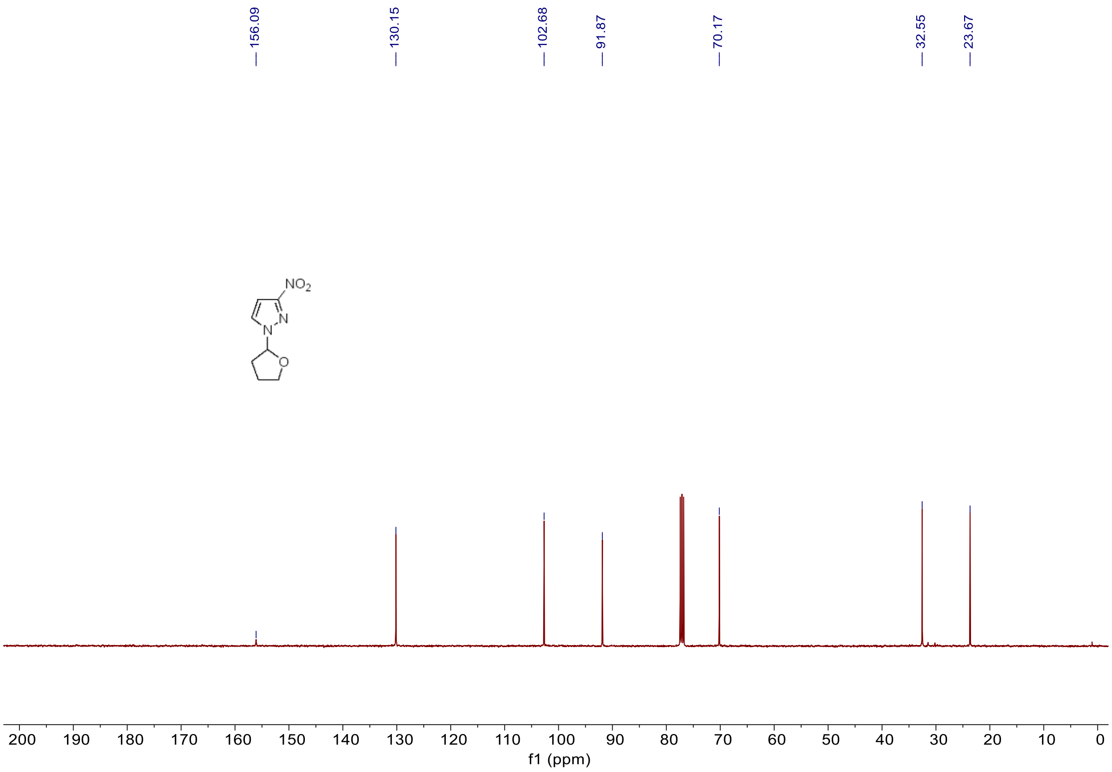
^{19}F NMR spectrum (376 MHz) of **32c** (see procedure)



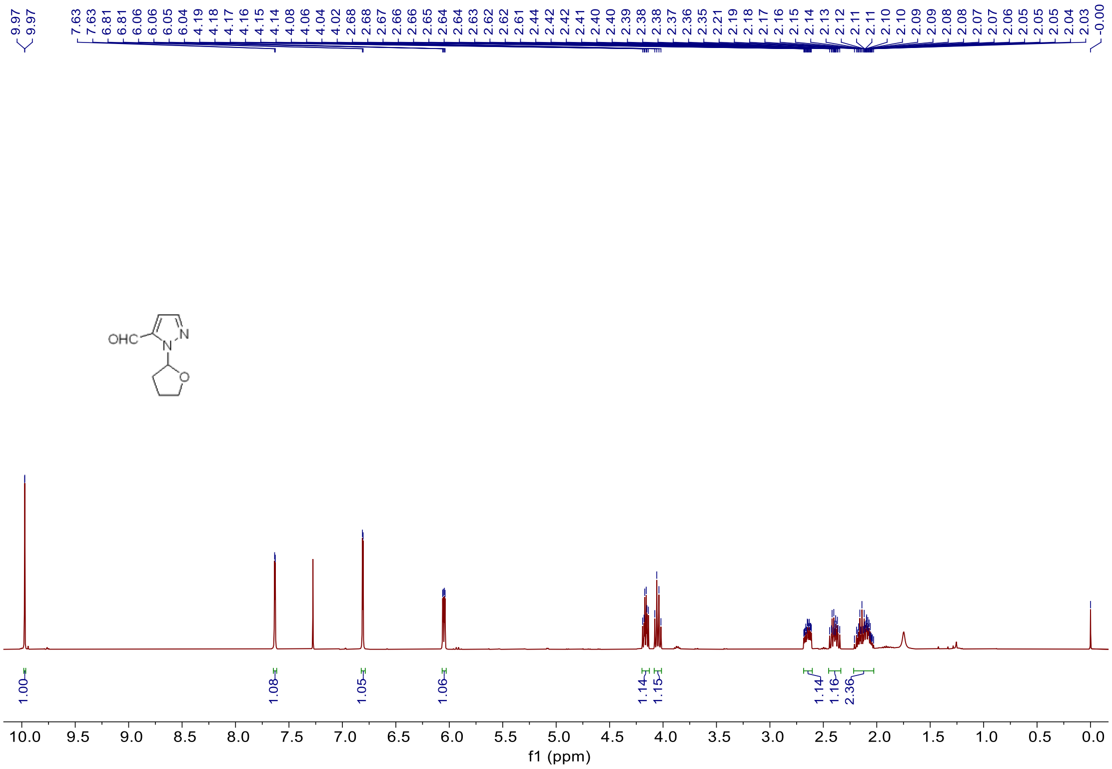
^{1}H NMR spectrum (400 MHz) of **33c** (see procedure)



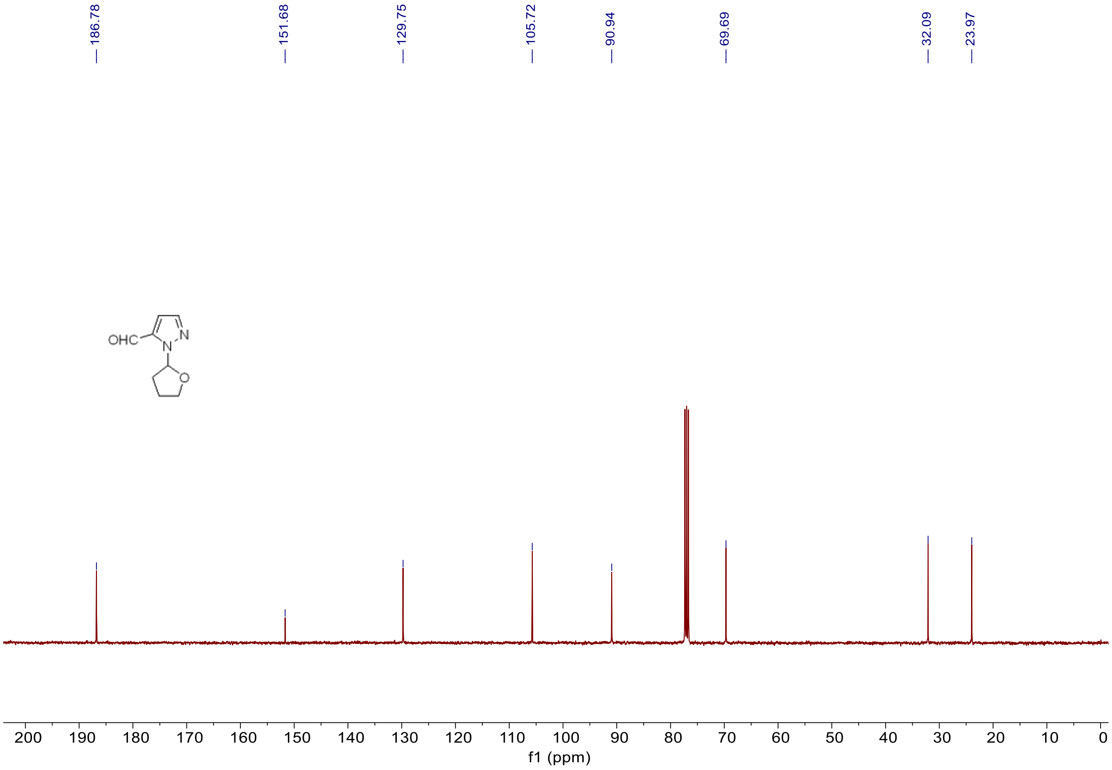
^{13}C NMR spectrum (101 MHz) of **33c** (see procedure)



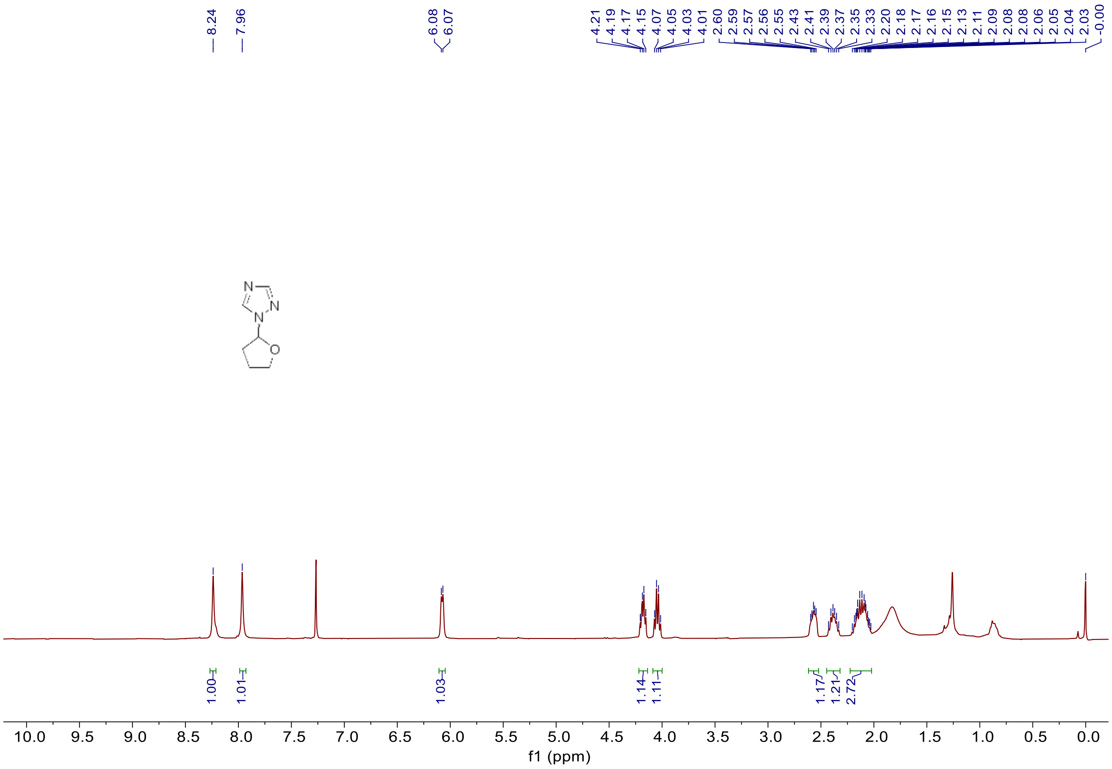
^{1}H NMR spectrum (400 MHz) of **34c** (see procedure)



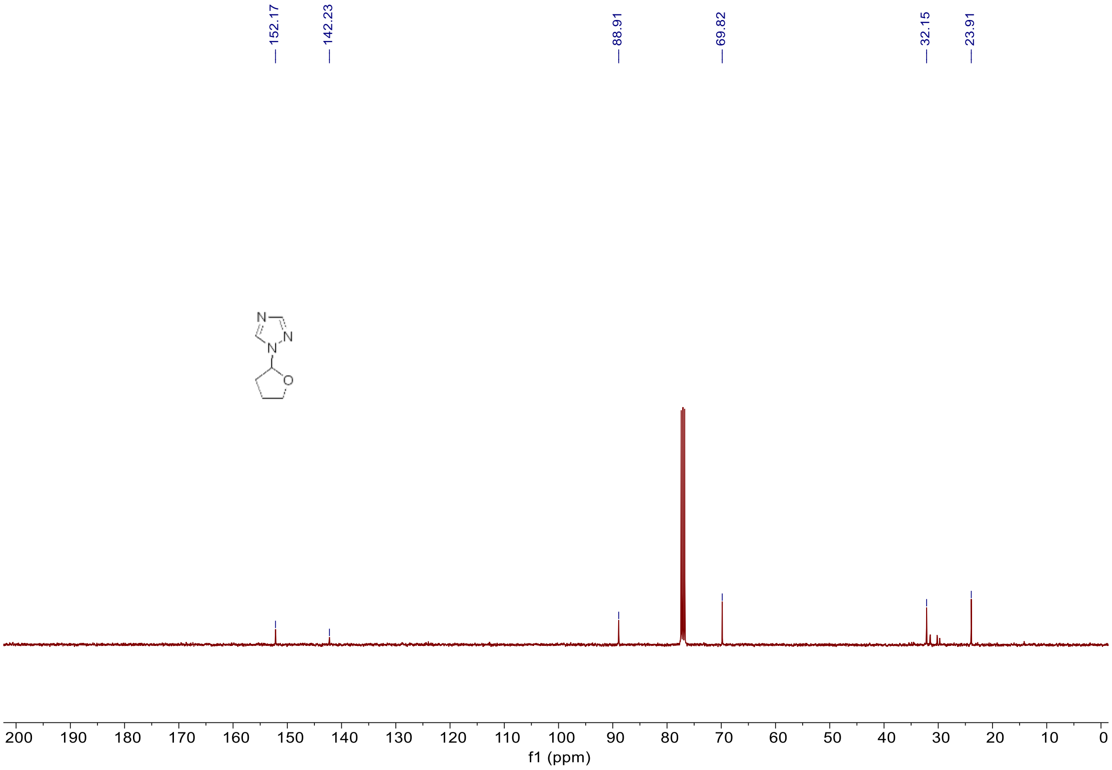
^{13}C NMR spectrum (101 MHz) of **34c** (see procedure)



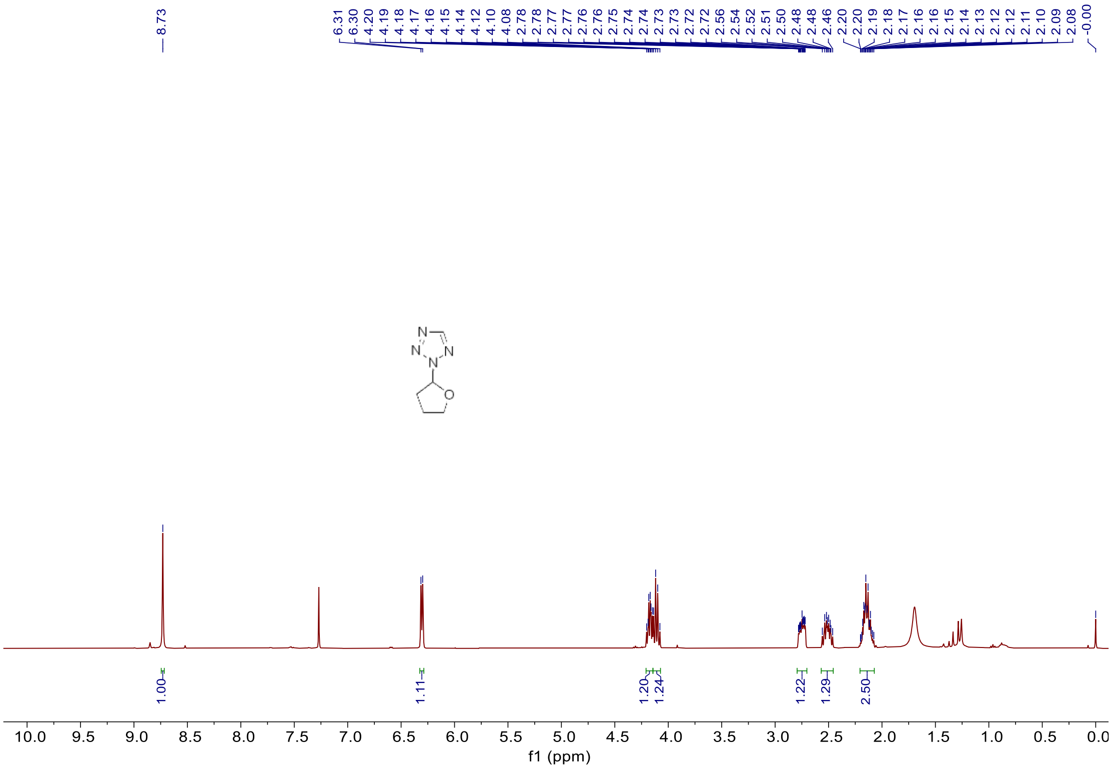
^{1}H NMR spectrum (400 MHz) of **35c** (see procedure)



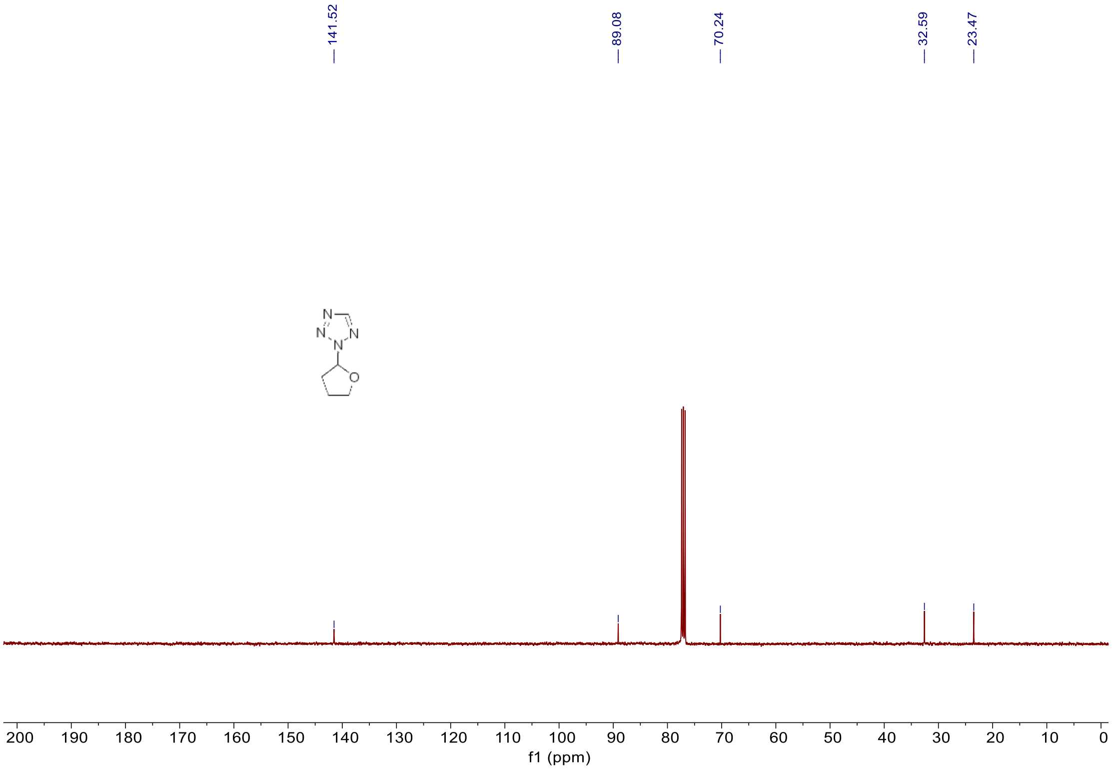
^{13}C NMR spectrum (101 MHz) of **35c** (see procedure)



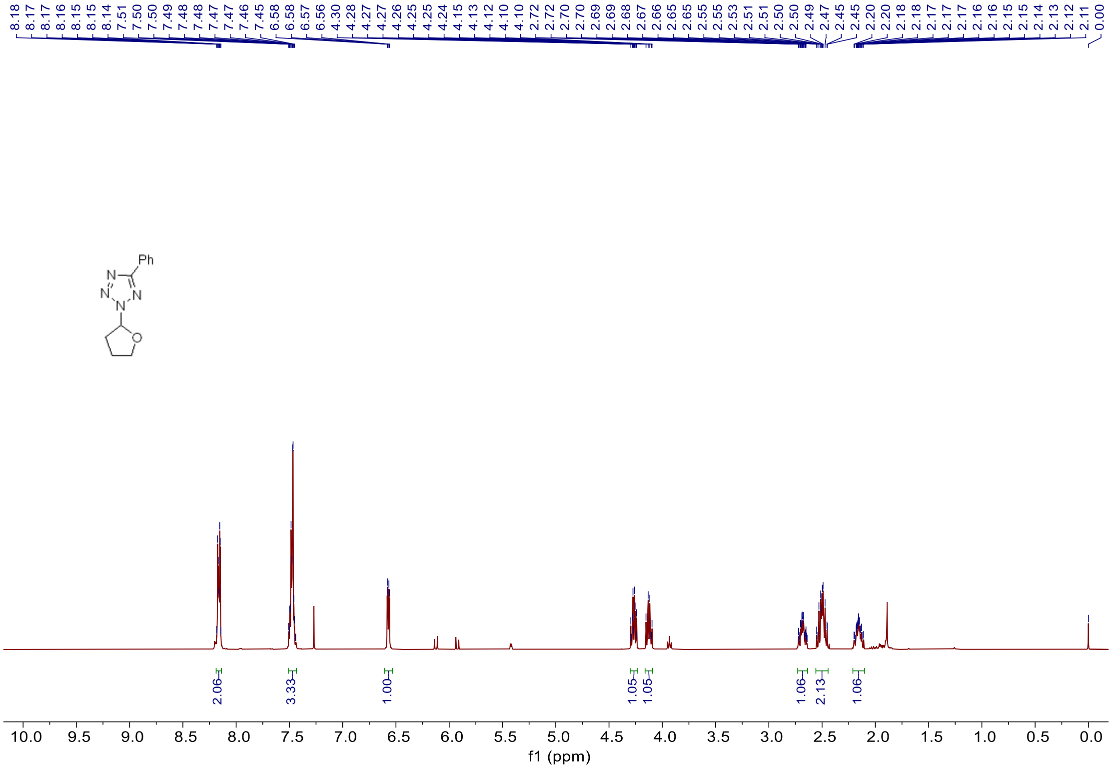
^{1}H NMR spectrum (400 MHz) of **36c** (see procedure)



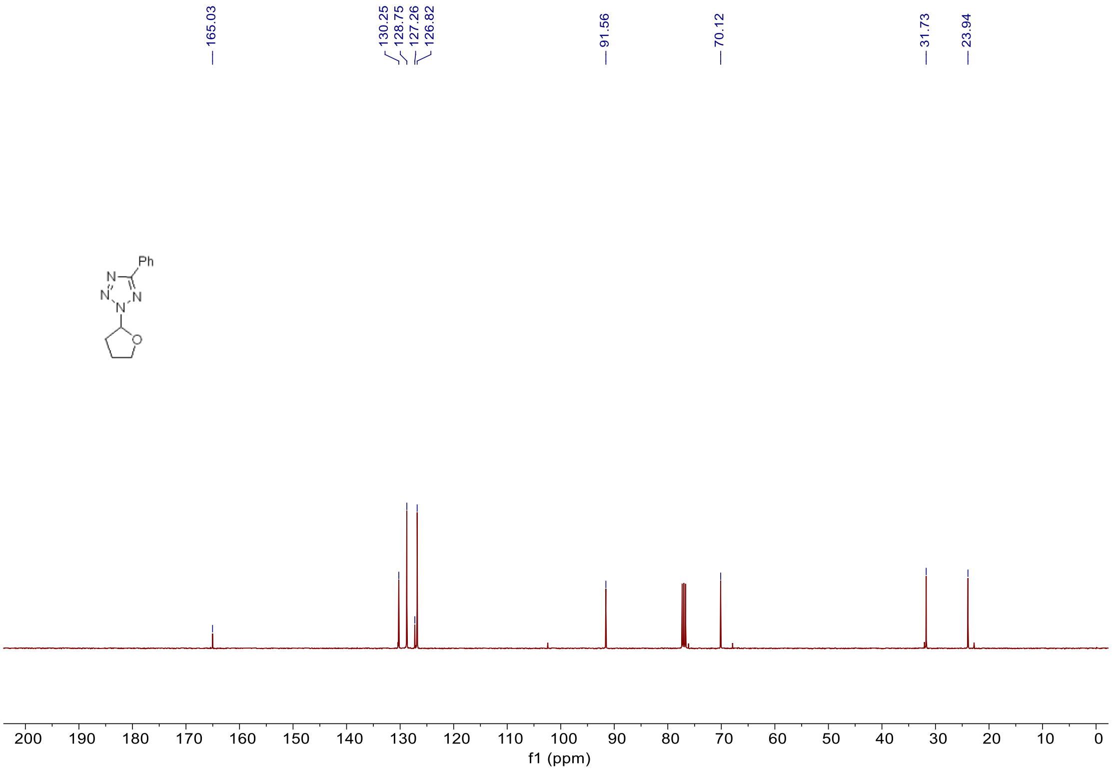
^{13}C NMR spectrum (101 MHz) of **36c** (see procedure)



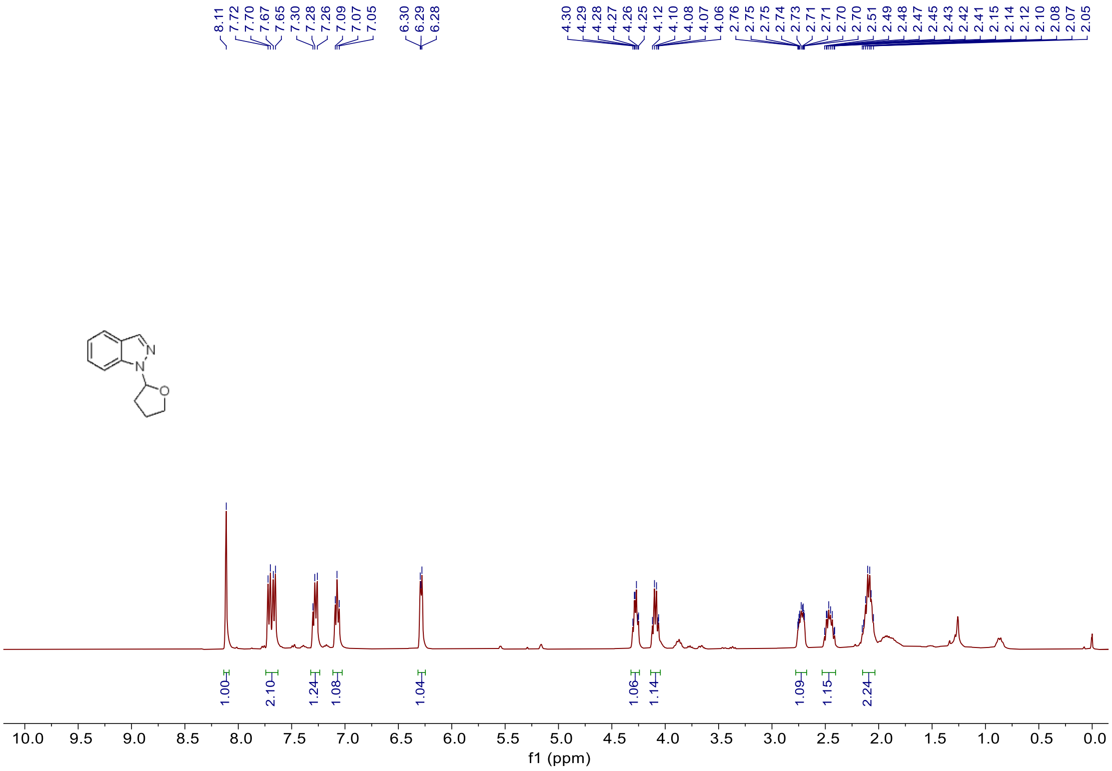
^{1}H NMR spectrum (400 MHz) of **37c** (see procedure)



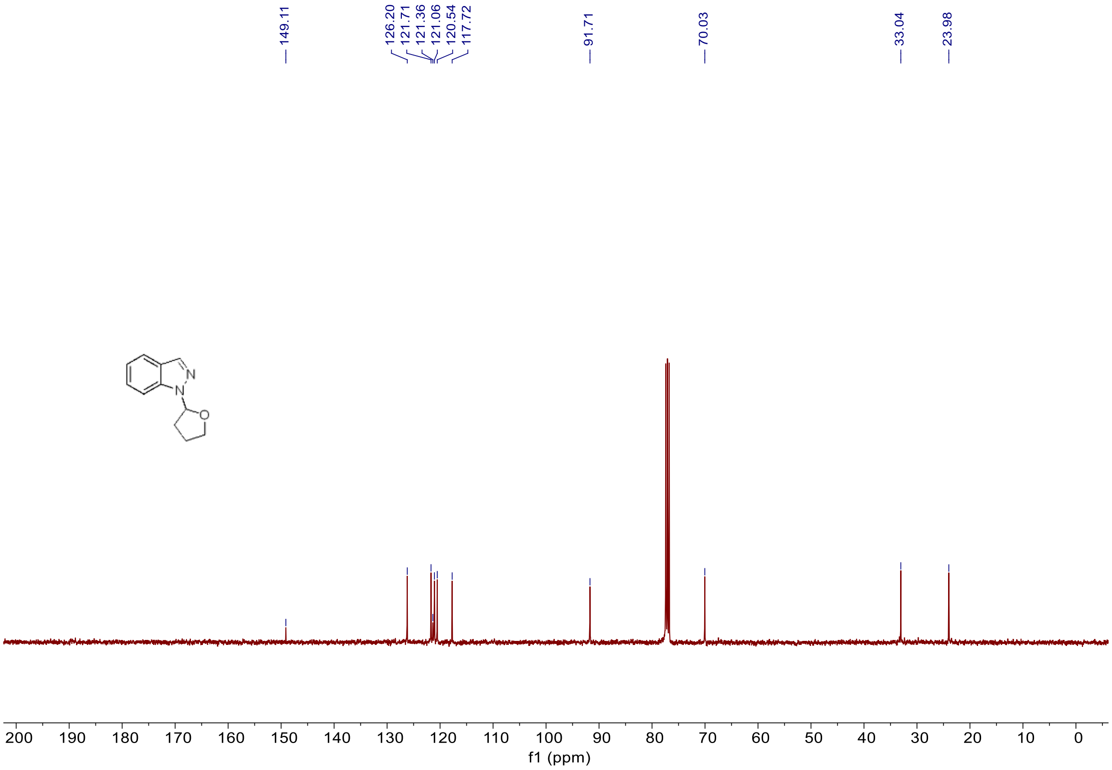
^{13}C NMR spectrum (101 MHz) of **37c** (see procedure)



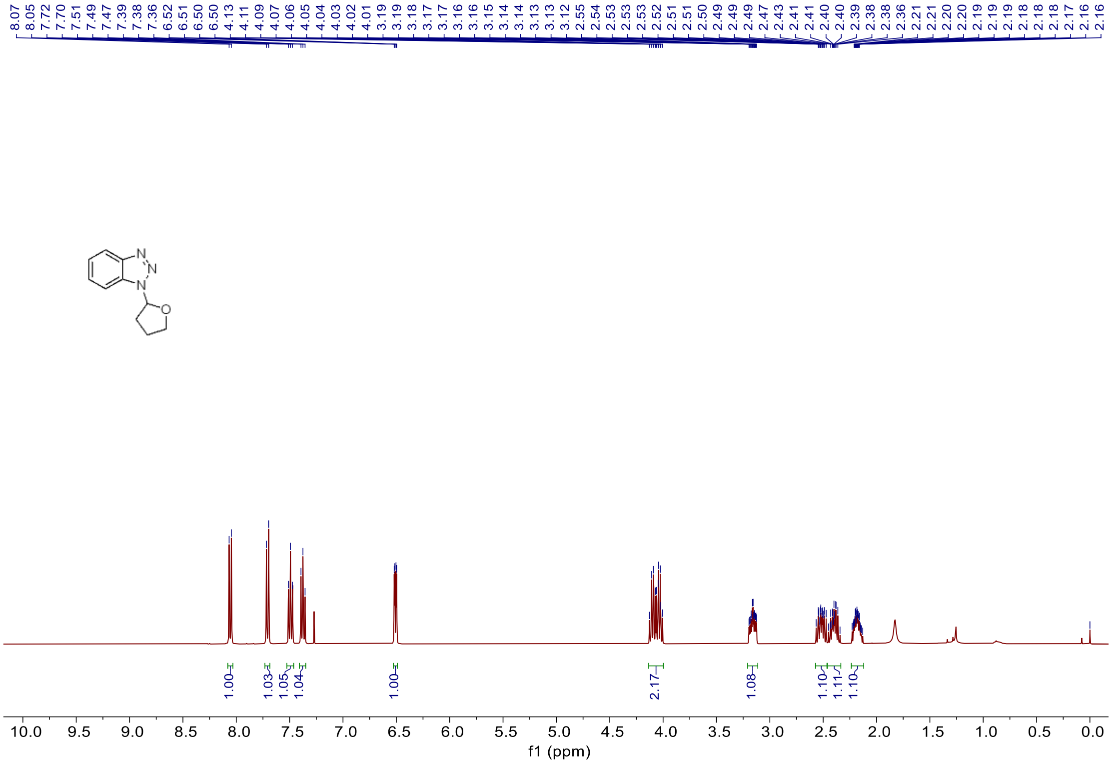
^{1}H NMR spectrum (400 MHz) of **38c** (see procedure)



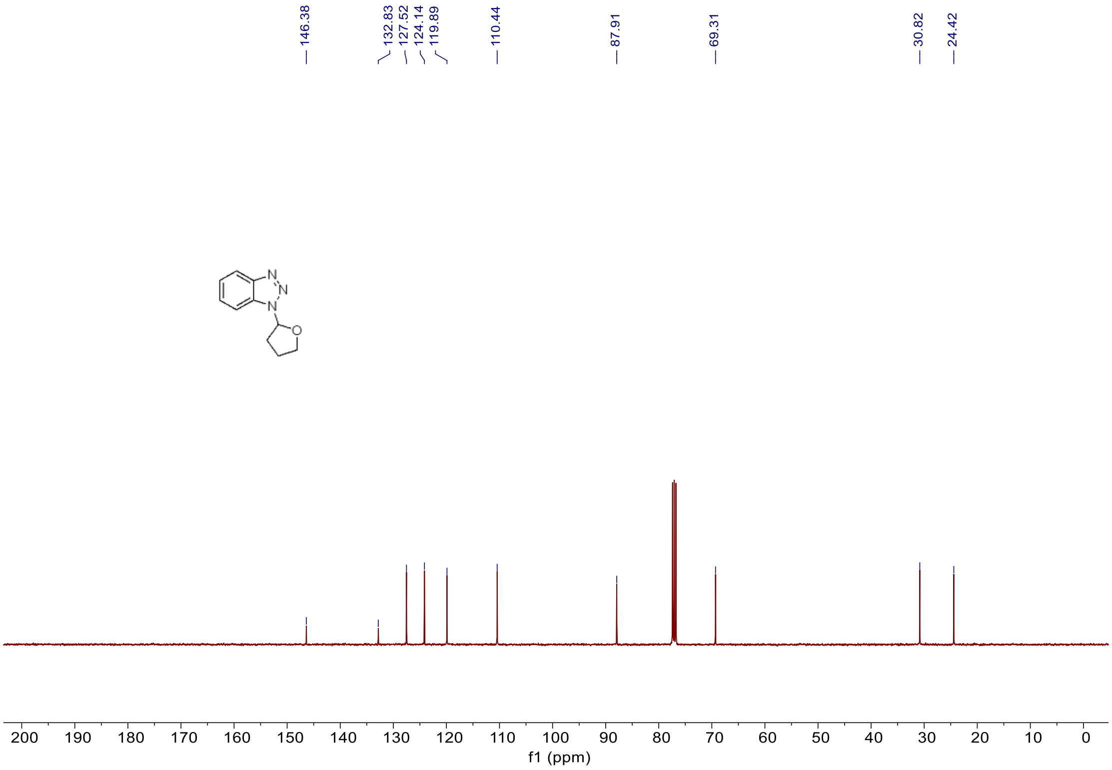
^{13}C NMR spectrum (101 MHz) of **38c** (see procedure)



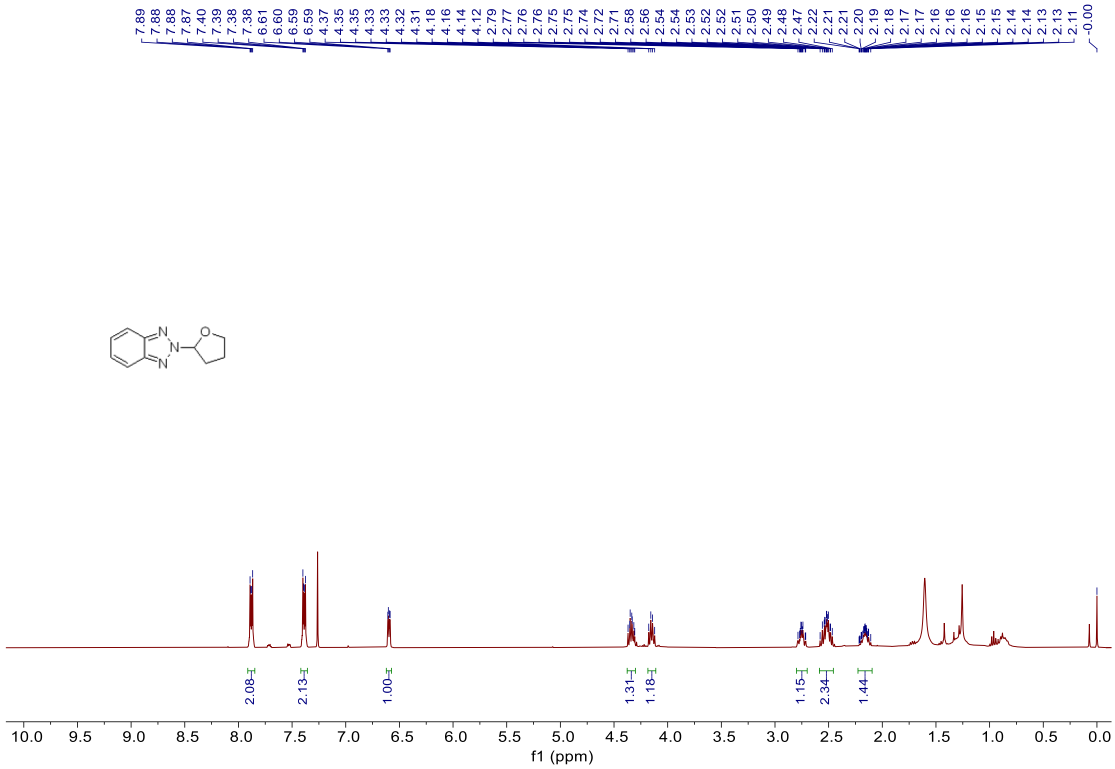
^{1}H NMR spectrum (400 MHz) of **39c-1** (see procedure)



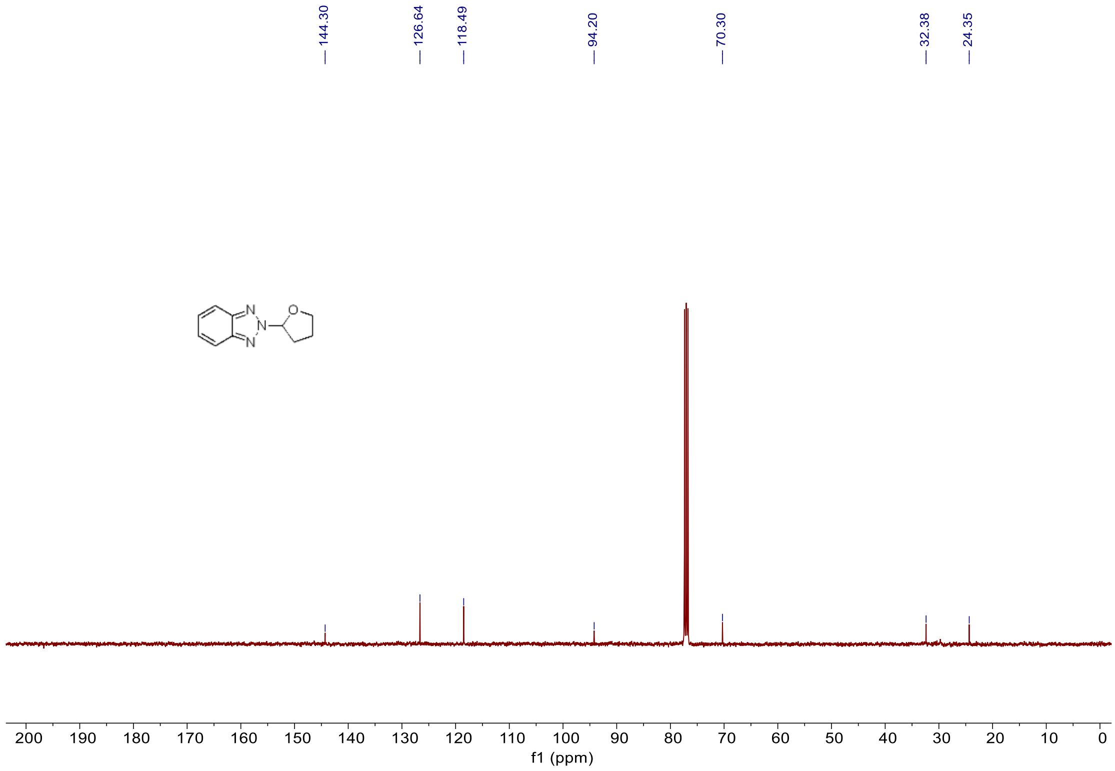
^{13}C NMR spectrum (101 MHz) of **39c-1** (see procedure)



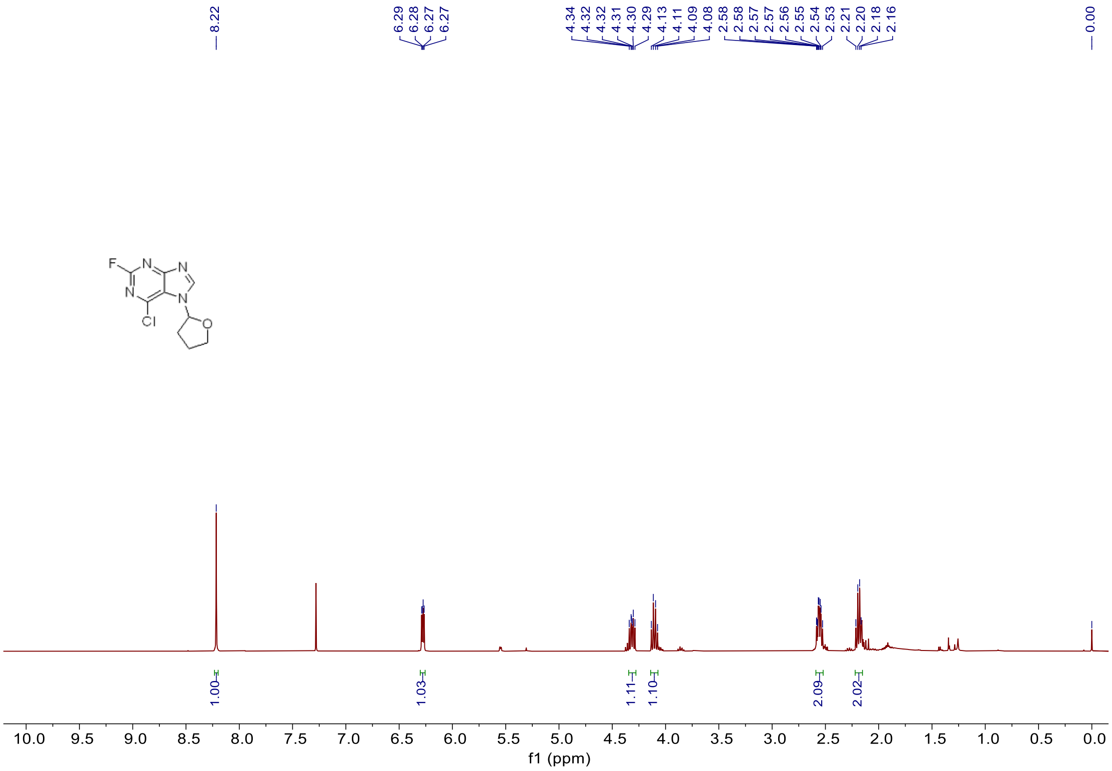
^{1}H NMR spectrum (400 MHz) of **39c-2** (see procedure)



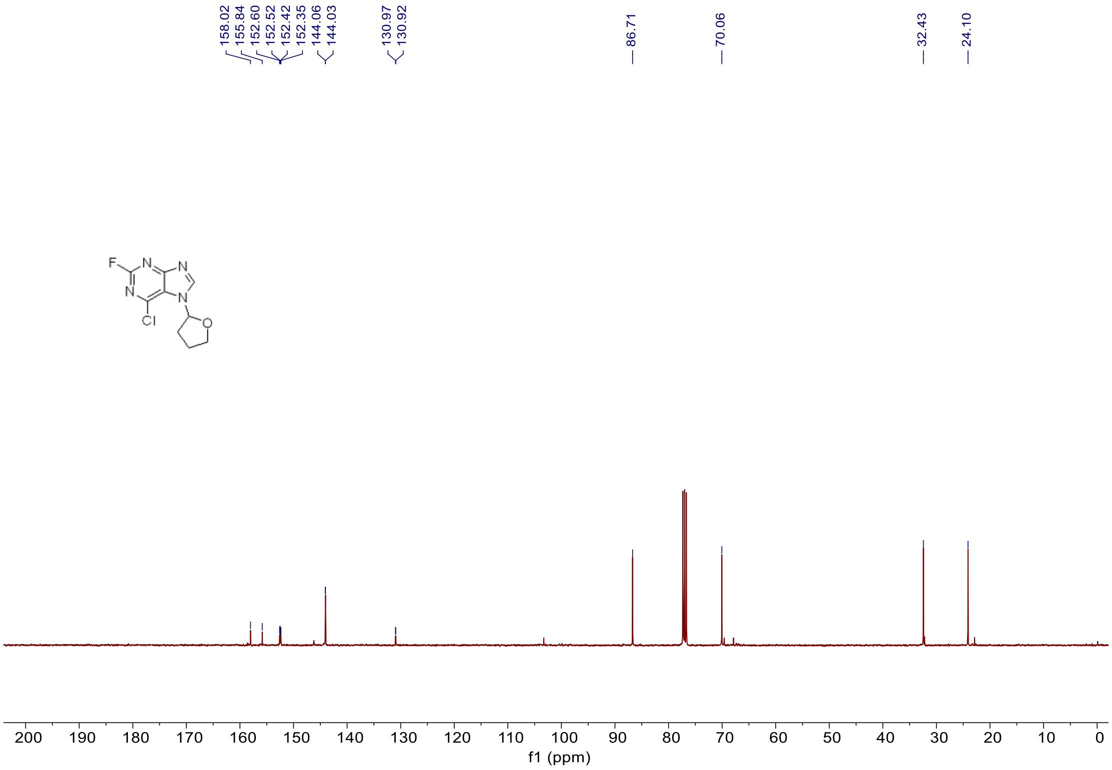
^{13}C NMR spectrum (101 MHz) of **38c-2** (see procedure)



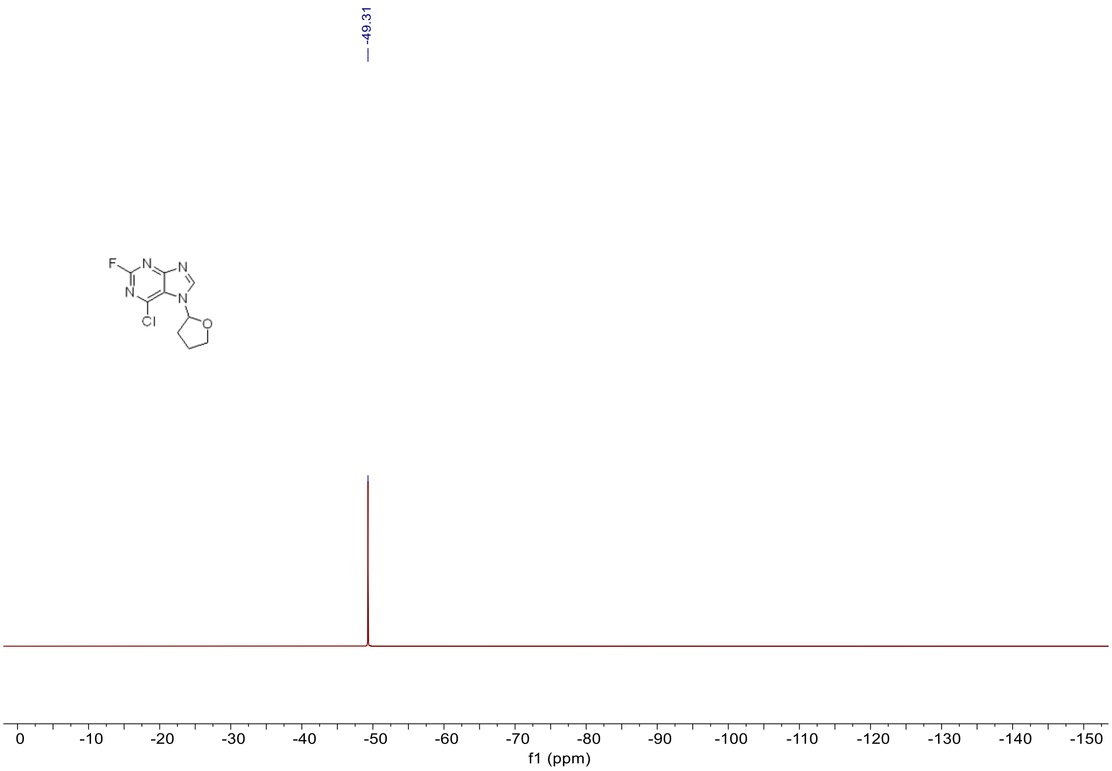
^{1}H NMR spectrum (400 MHz) of **40c** (see procedure)



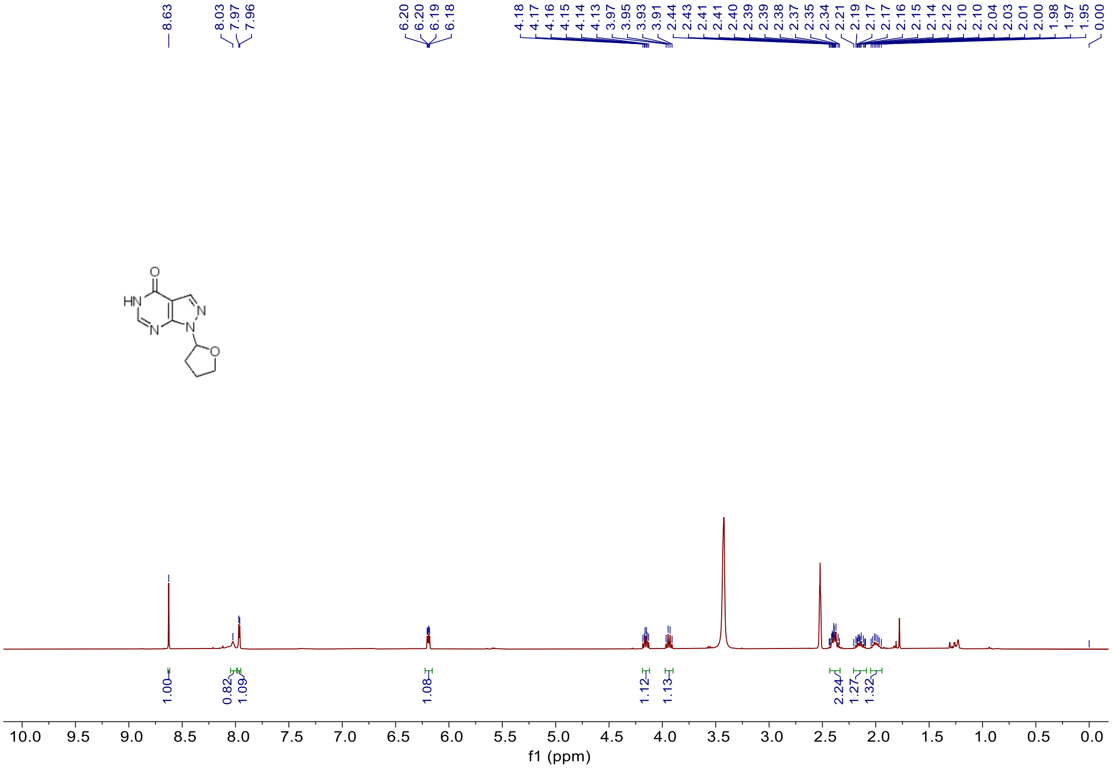
^{13}C NMR spectrum (101 MHz) of **40c** (see procedure)



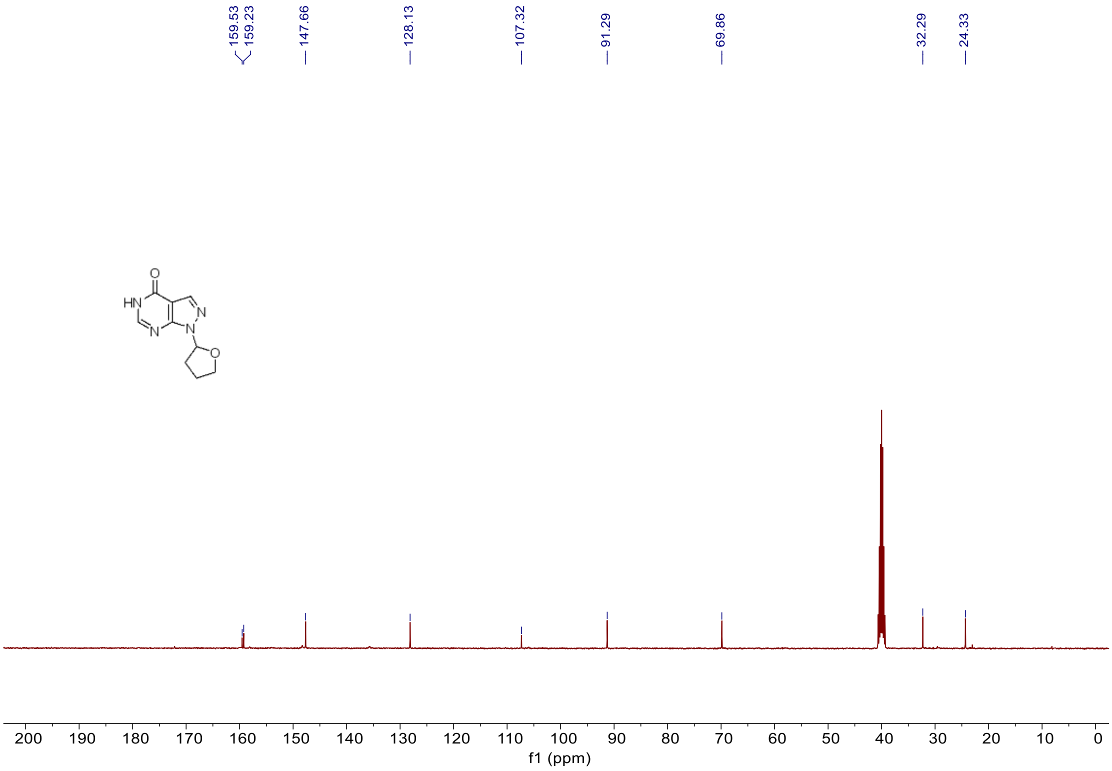
^{19}F NMR spectrum (376 MHz) of **40c** (see procedure)



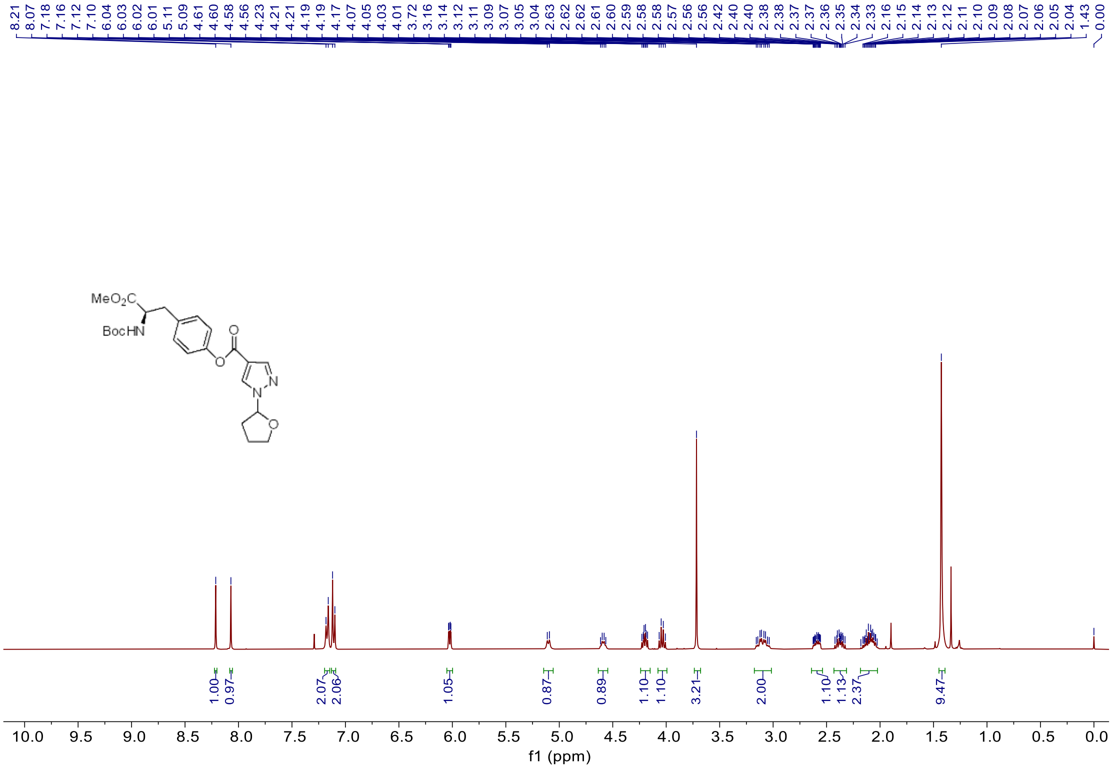
^{1}H NMR spectrum (400 MHz) of **41c** (see procedure)



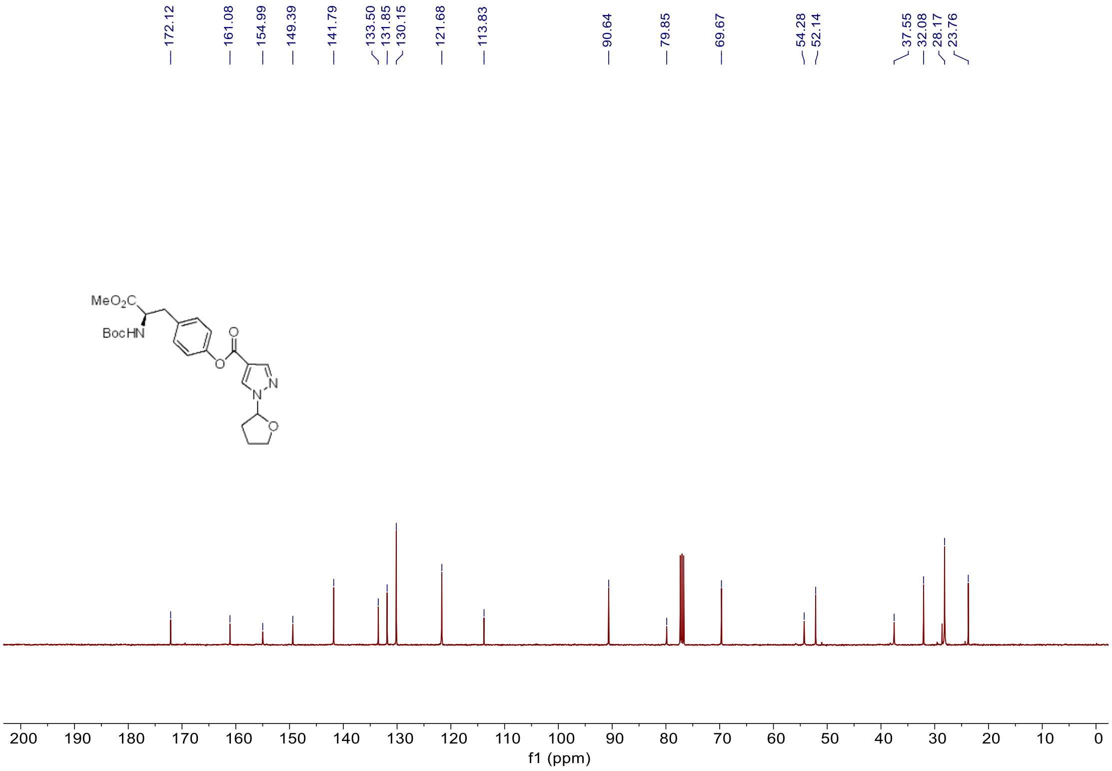
^{13}C NMR spectrum (101 MHz) of **41c** (see procedure)



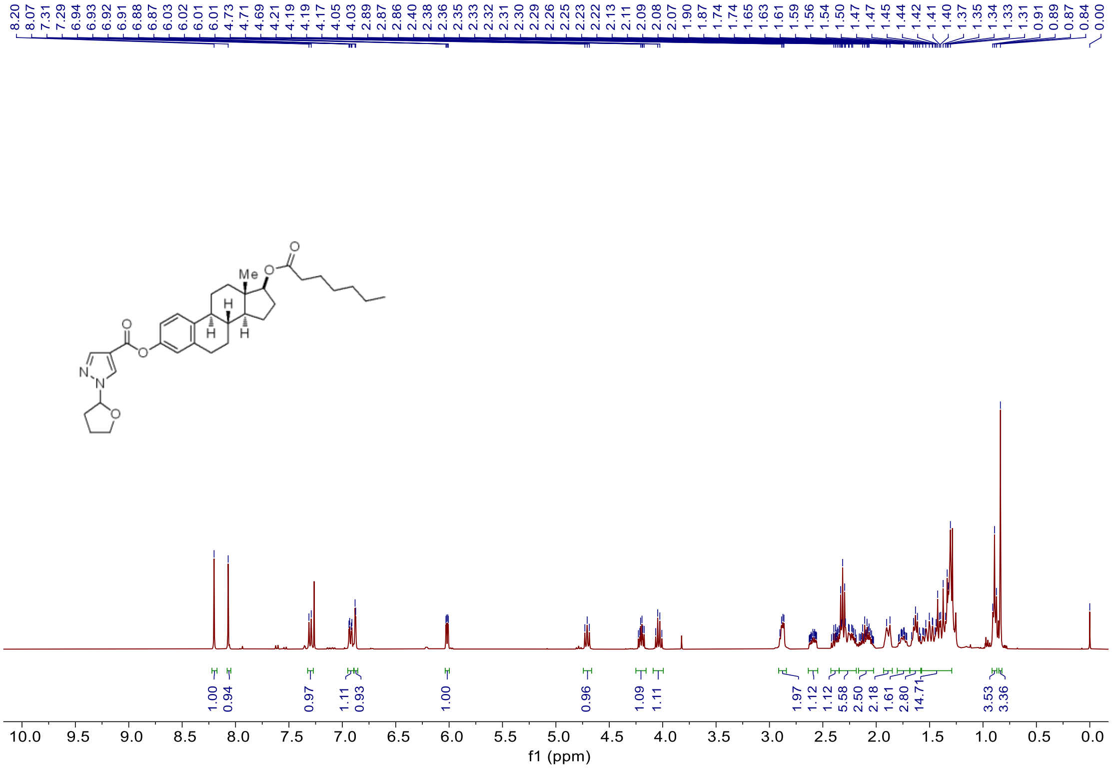
^{1}H NMR spectrum (400 MHz) of **42c** (see procedure)



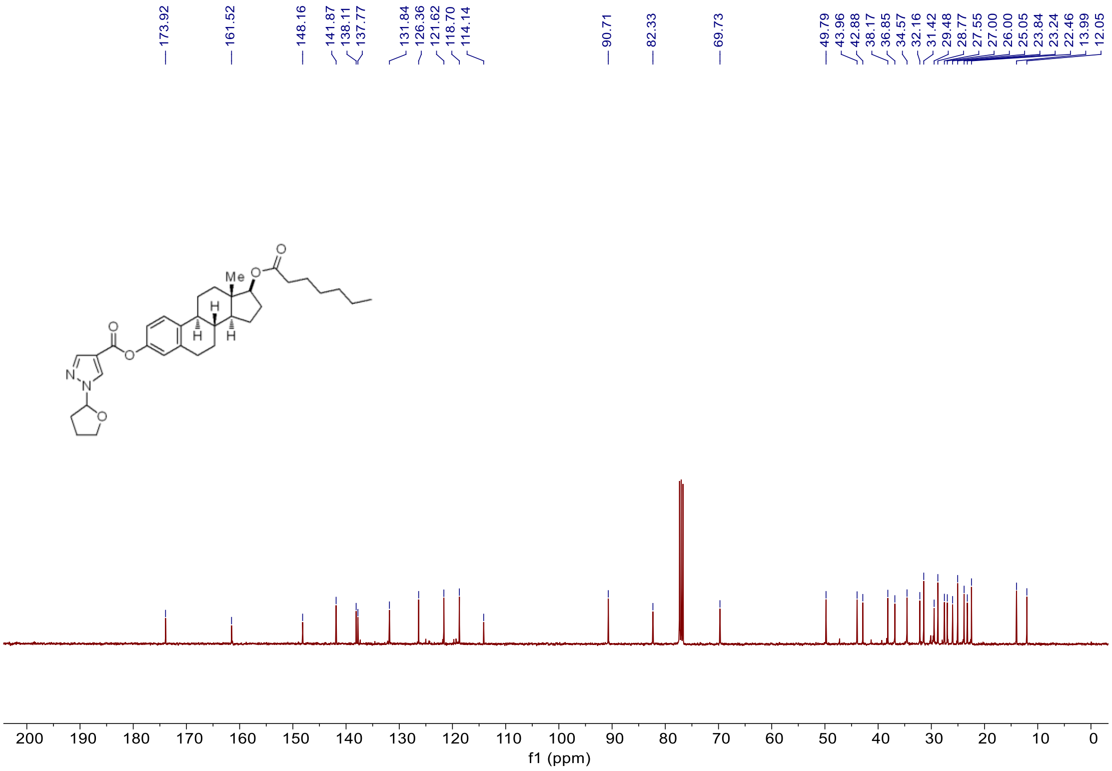
^{13}C NMR spectrum (101 MHz) of **42c** (see procedure)



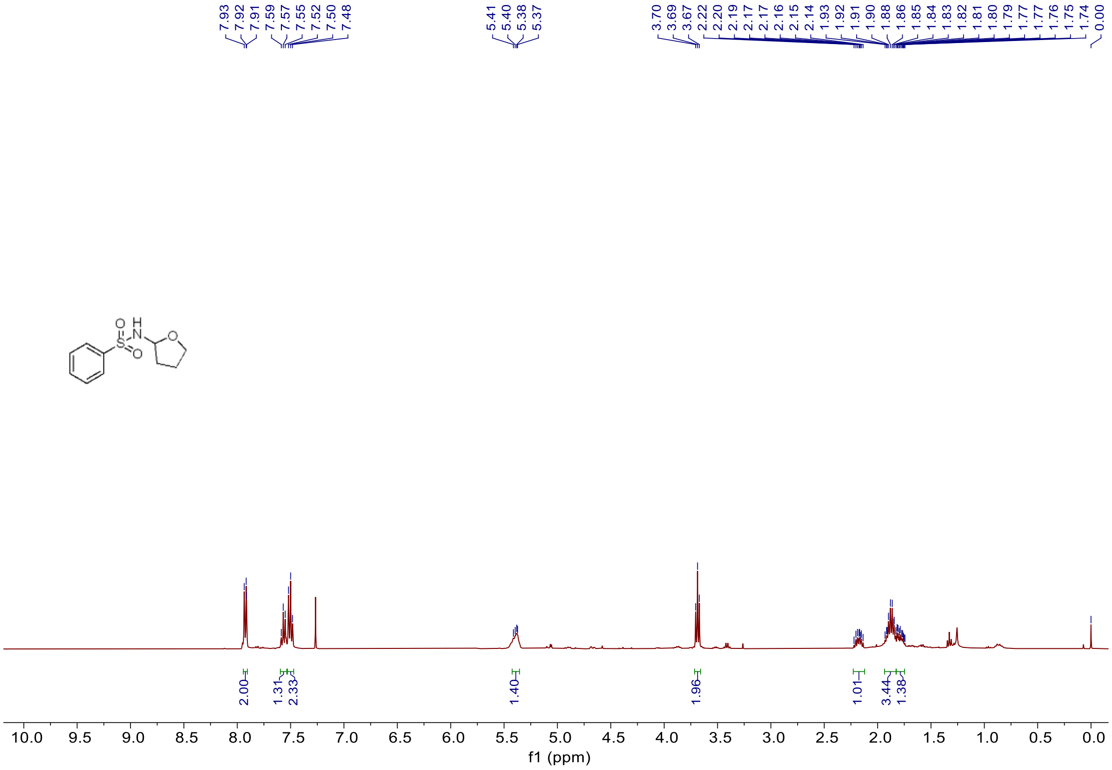
^{1}H NMR spectrum (400 MHz) of **43c** (see procedure)



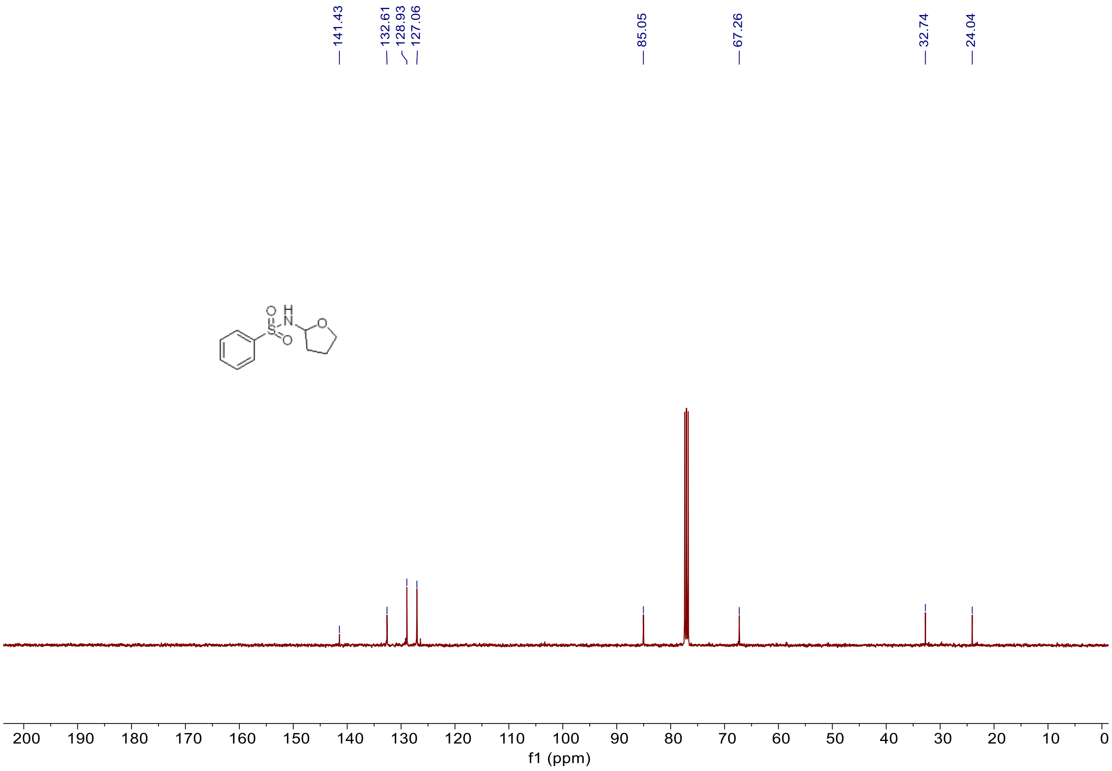
^{13}C NMR spectrum (101 MHz) of **43c** (see procedure)



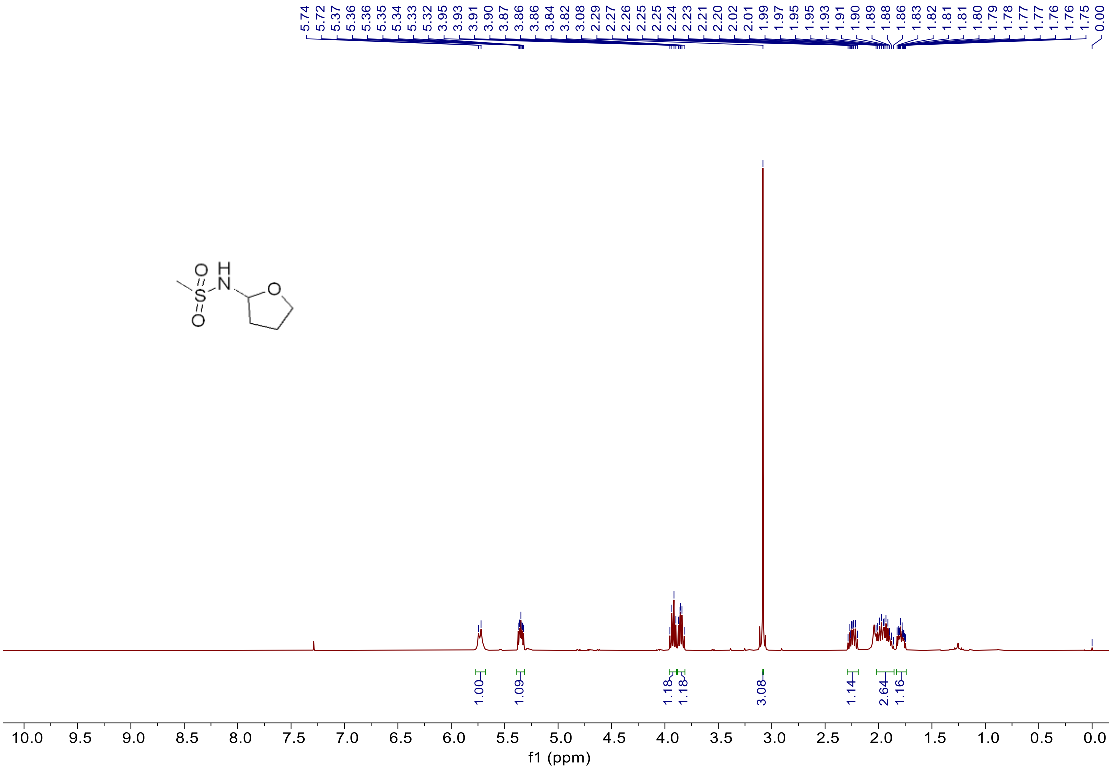
^{1}H NMR spectrum (400 MHz) of **44c** (see procedure)



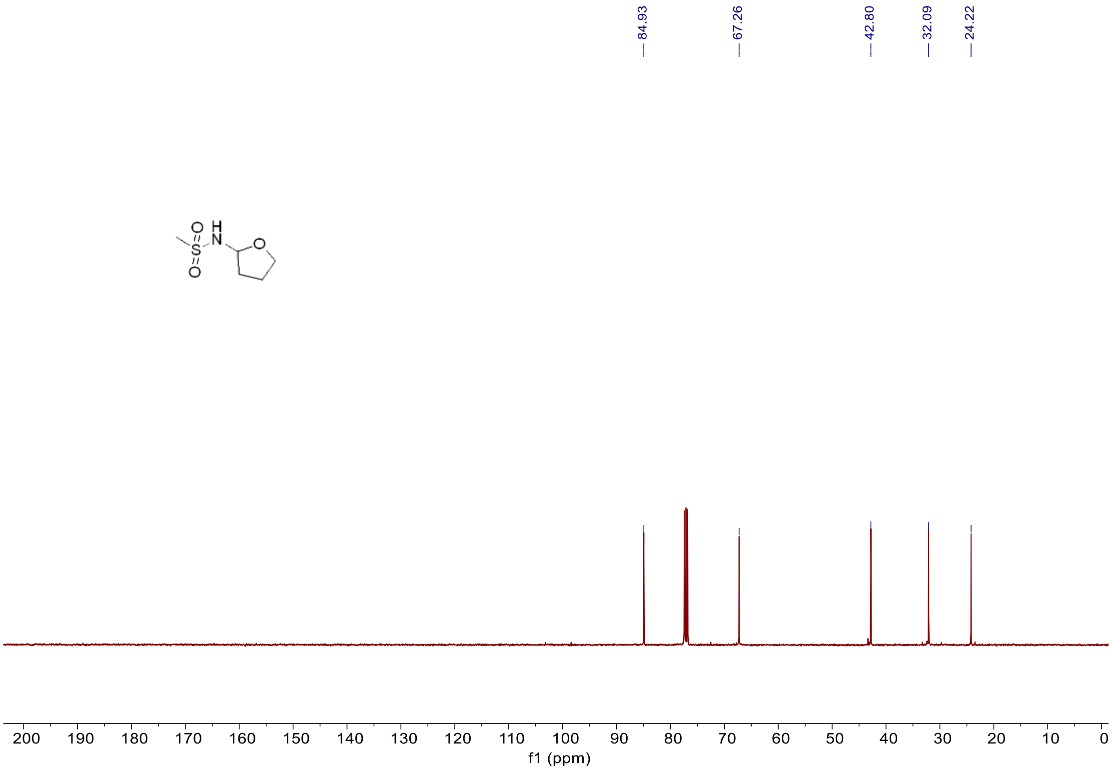
^{13}C NMR spectrum (101 MHz) of **44c** (see procedure)



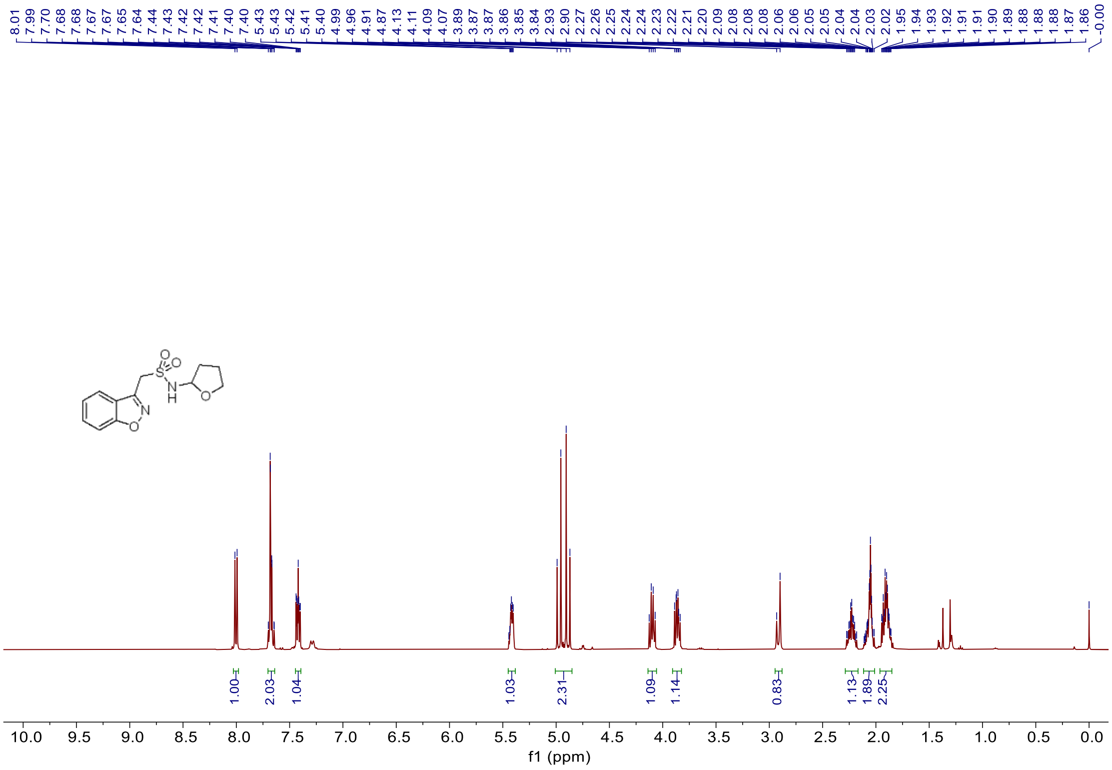
^{1}H NMR spectrum (400 MHz) of **45c** (see procedure)



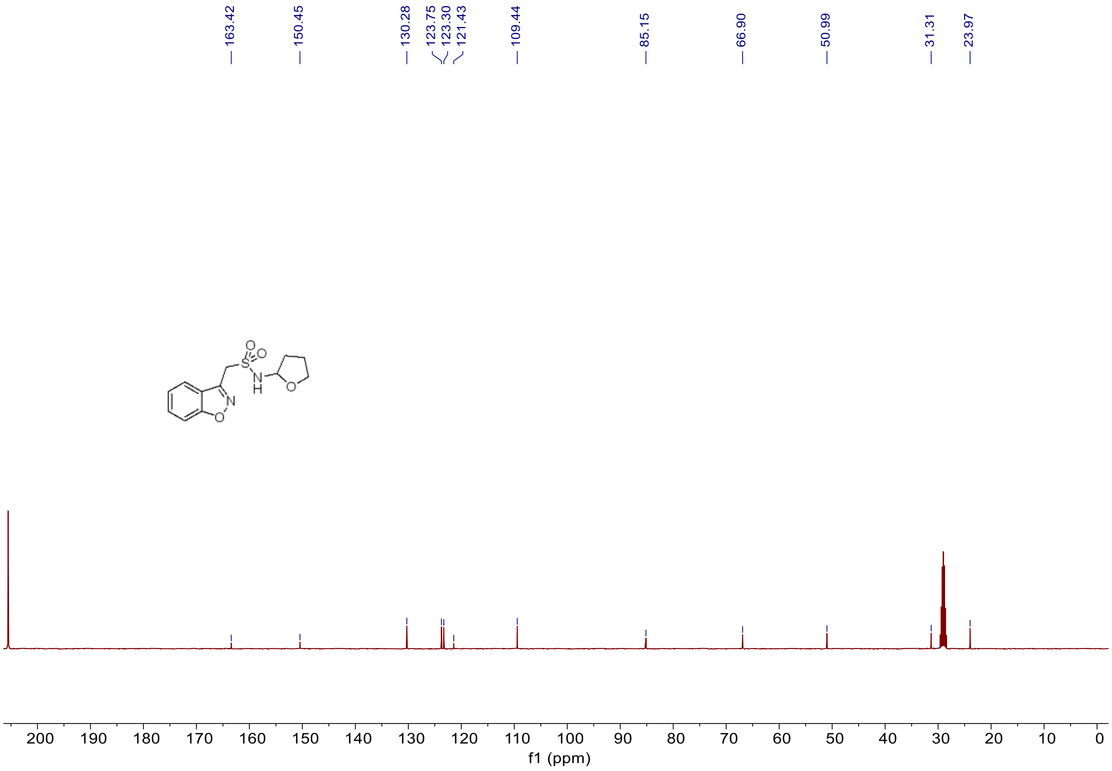
^{13}C NMR spectrum (101 MHz) of **45c** (see procedure)



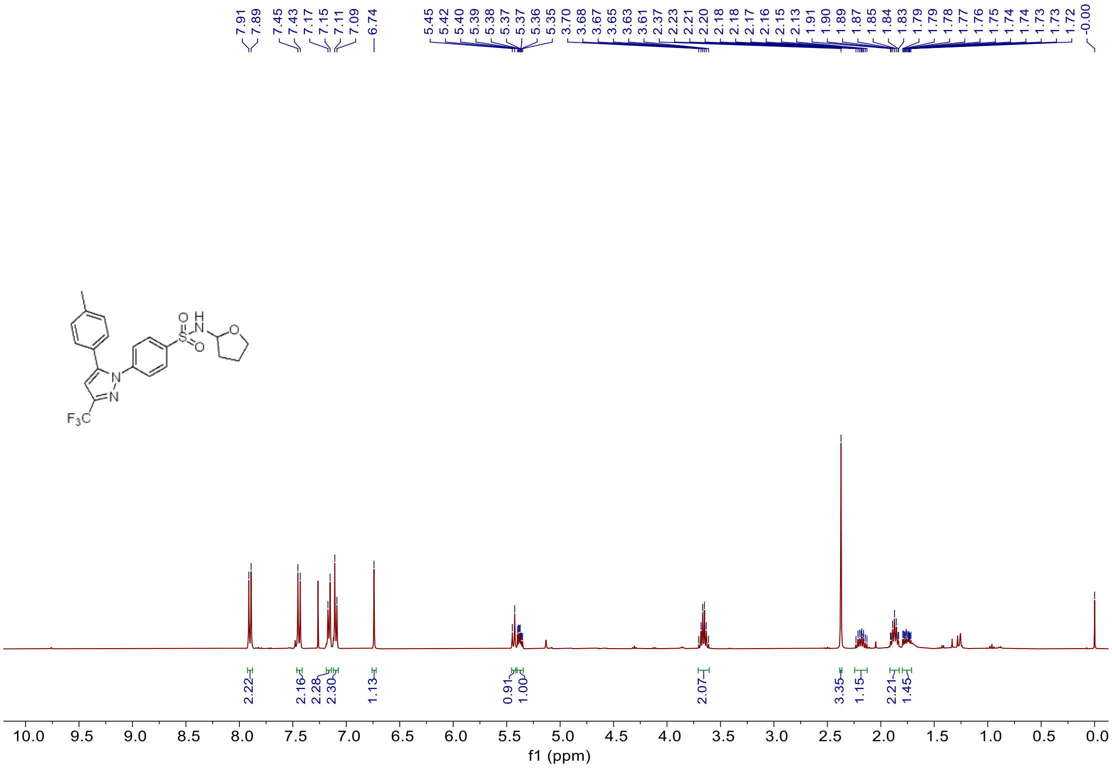
^{1}H NMR spectrum (400 MHz) of **46c** (see procedure)



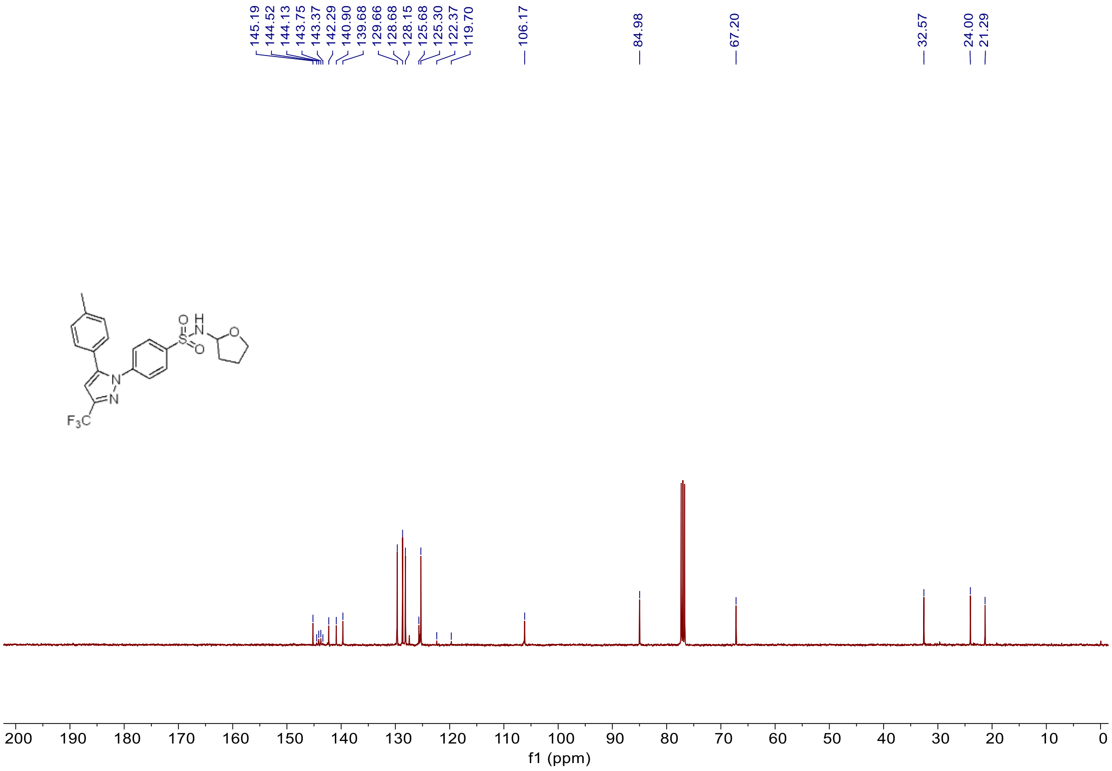
^{13}C NMR spectrum (101 MHz) of **46c** (see procedure)



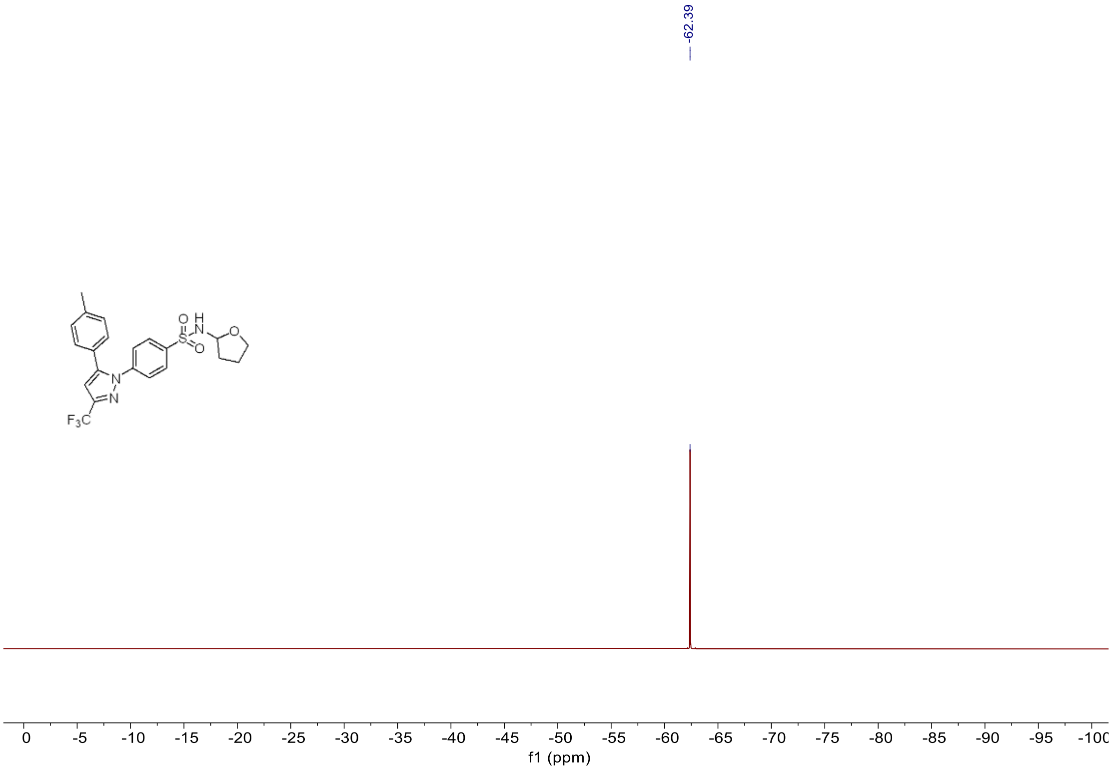
^{1}H NMR spectrum (400 MHz) of **47c** (see procedure)



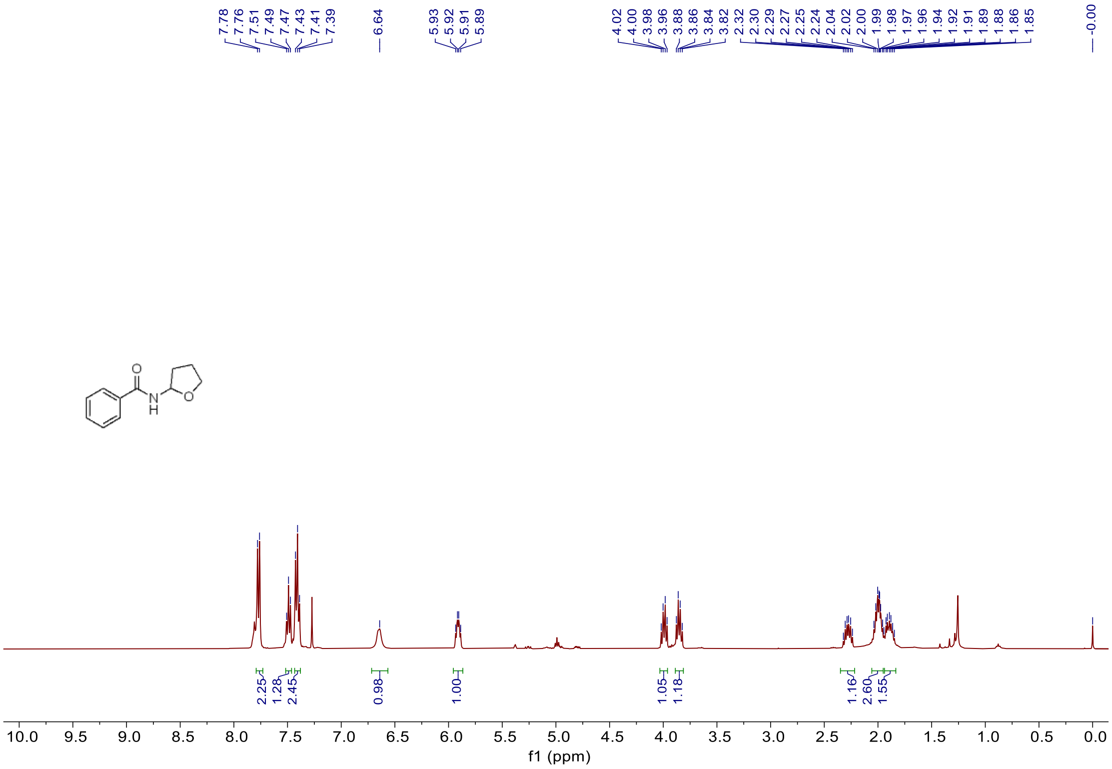
^{13}C NMR spectrum (101 MHz) of **47c** (see procedure)



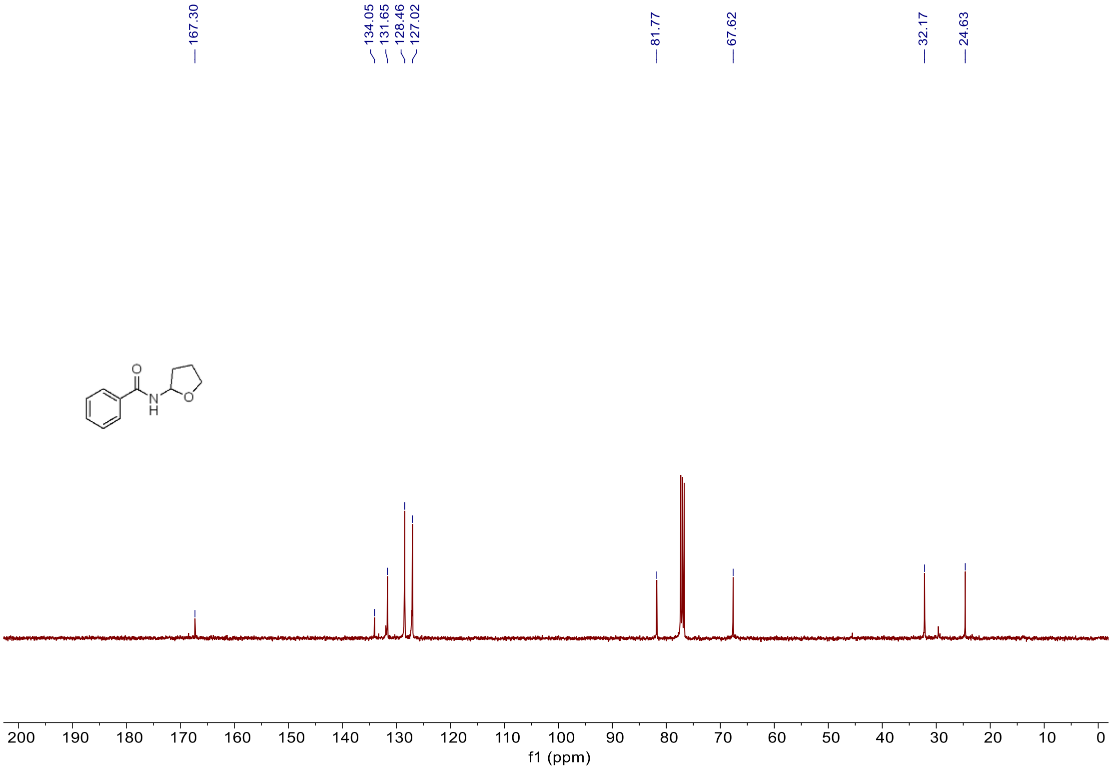
^{19}F NMR spectrum (376 MHz) of **47c** (see procedure)



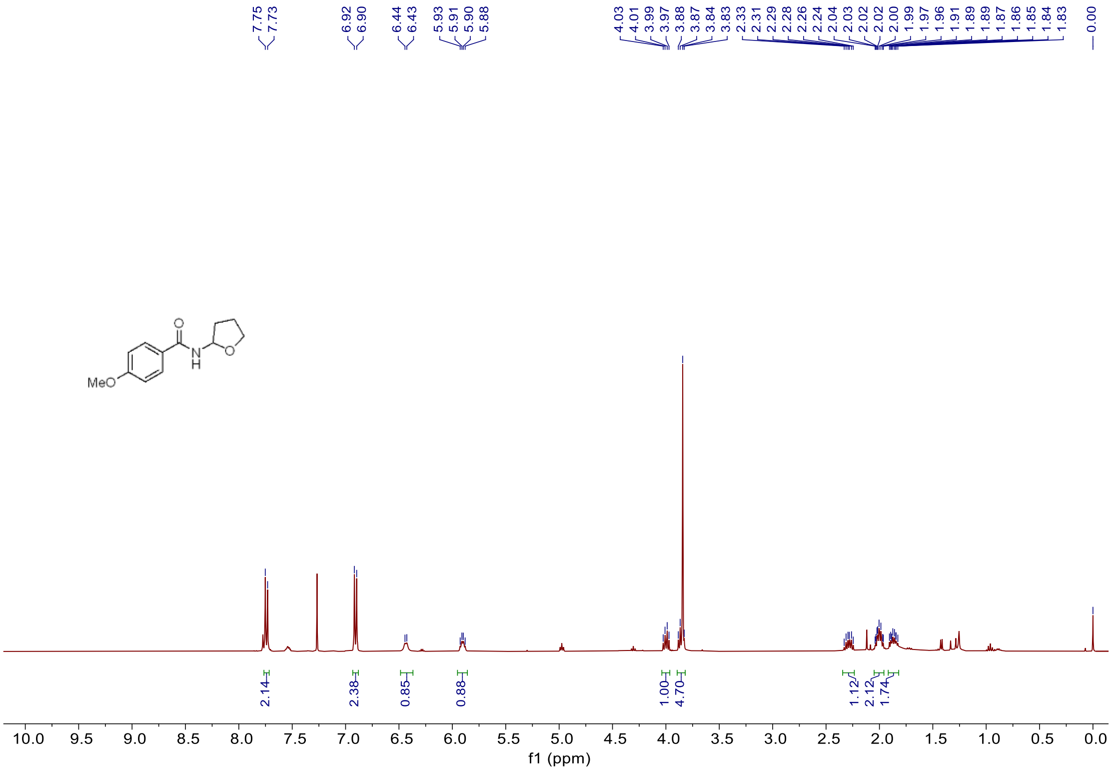
^{1}H NMR spectrum (400 MHz) of **48c** (see procedure)



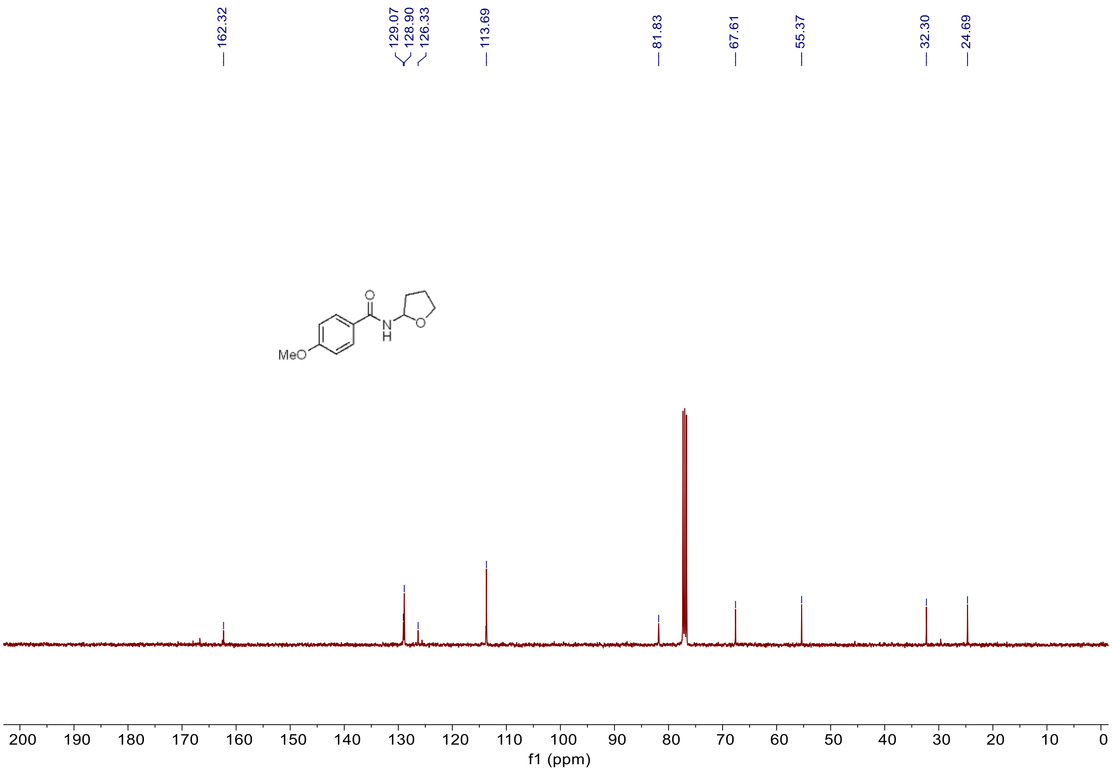
^{13}C NMR spectrum (101 MHz) of **48c** (see procedure)



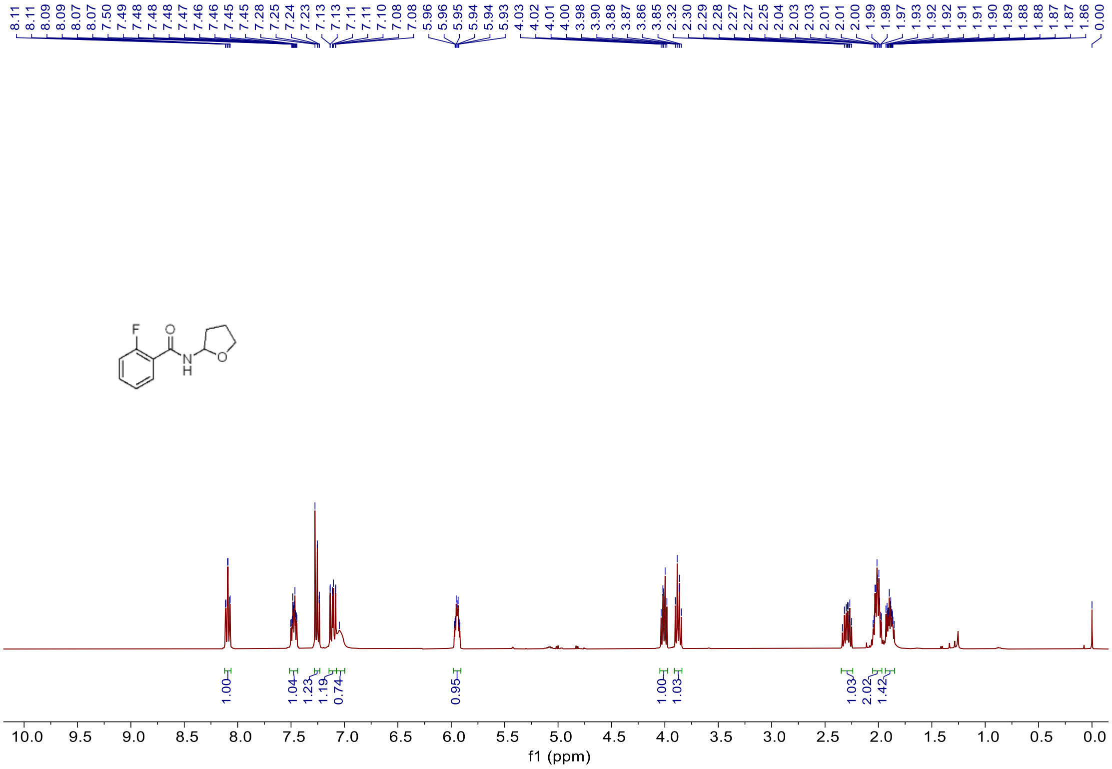
^{1}H NMR spectrum (400 MHz) of **49c** (see procedure)



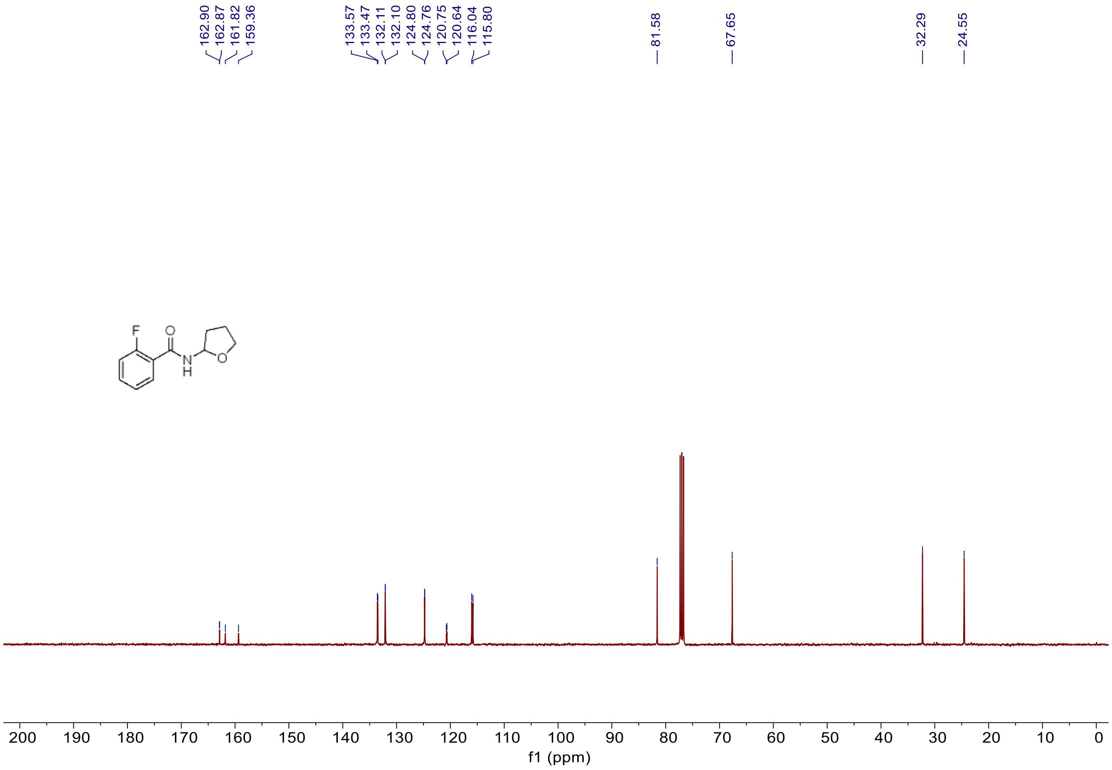
^{13}C NMR spectrum (101 MHz) of **49c** (see procedure)



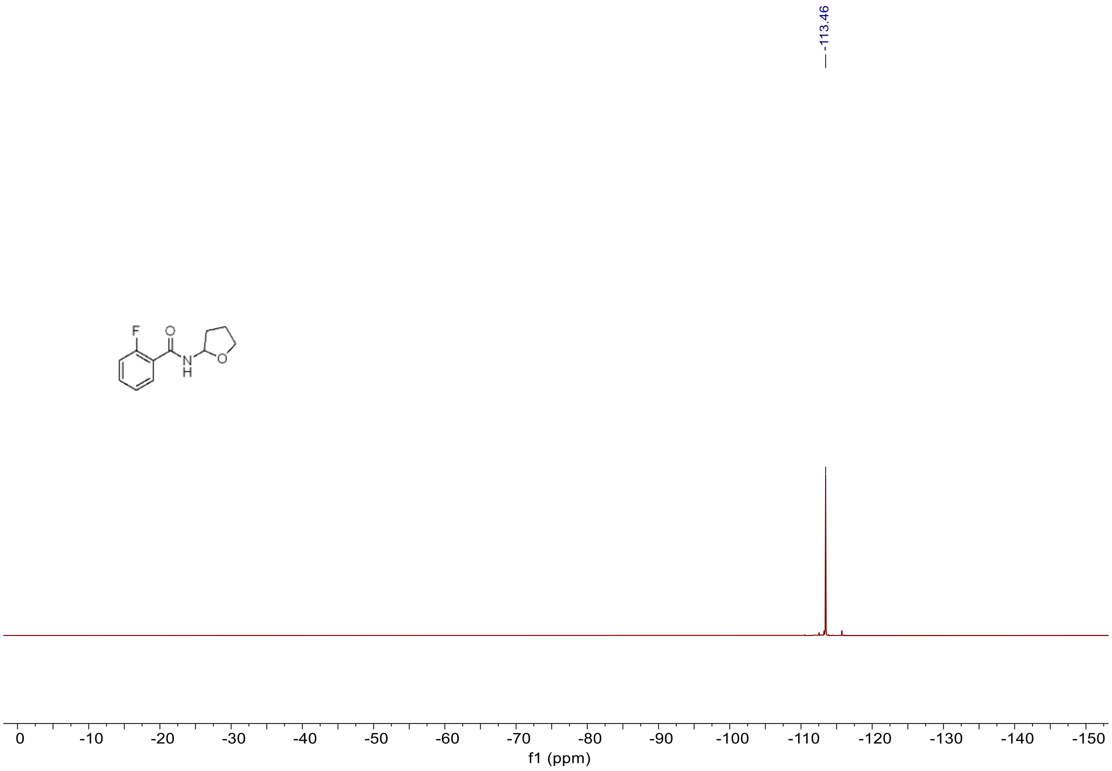
^{1}H NMR spectrum (400 MHz) of **50c** (see procedure)



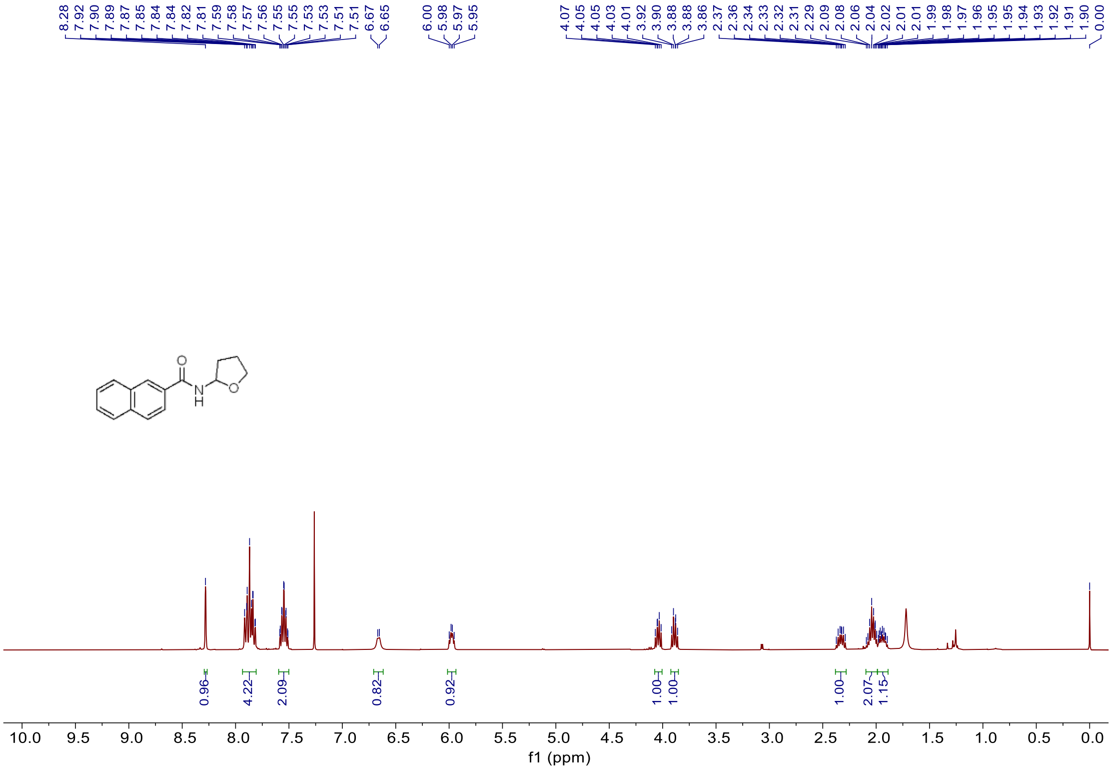
^{13}C NMR spectrum (101 MHz) of **50c** (see procedure)



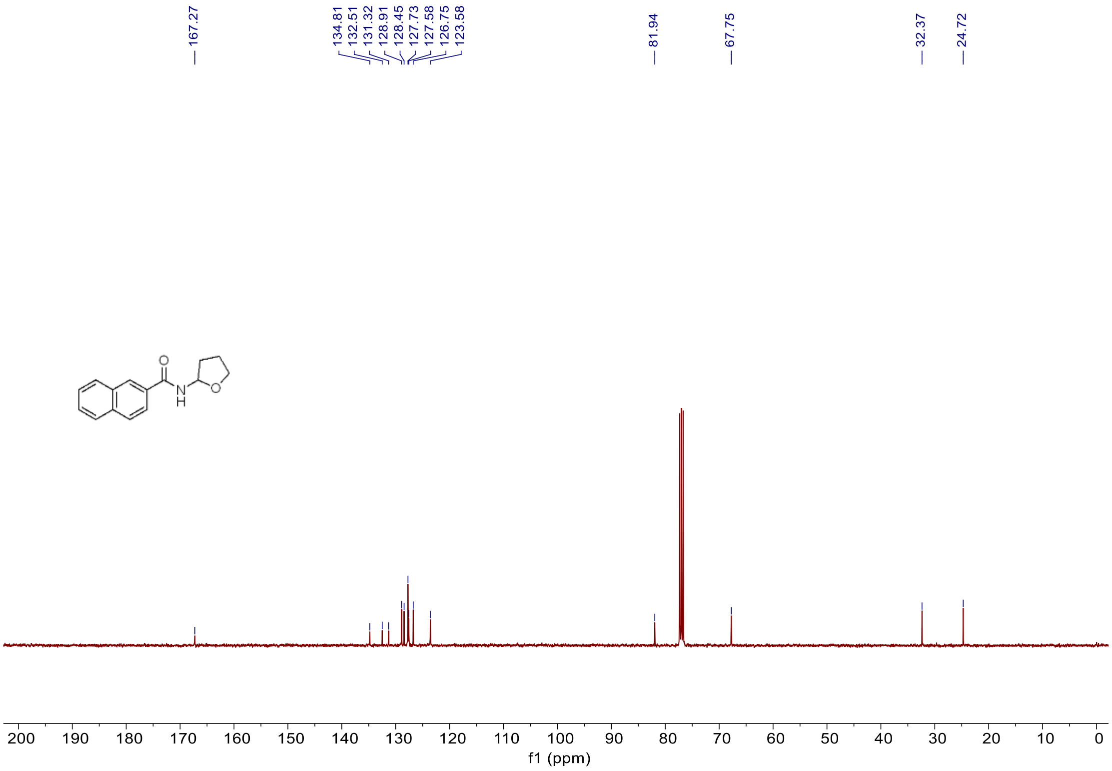
^{19}F NMR spectrum (376 MHz) of **50c** (see procedure)



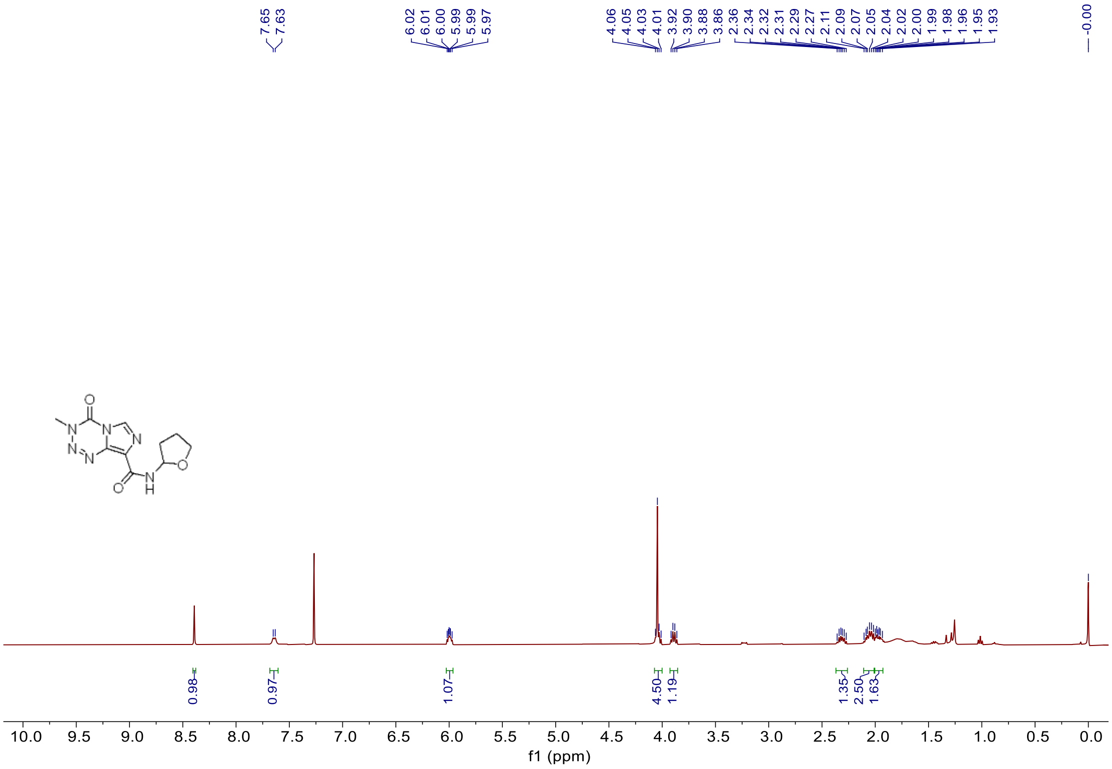
^{1}H NMR spectrum (400 MHz) of **51c** (see procedure)



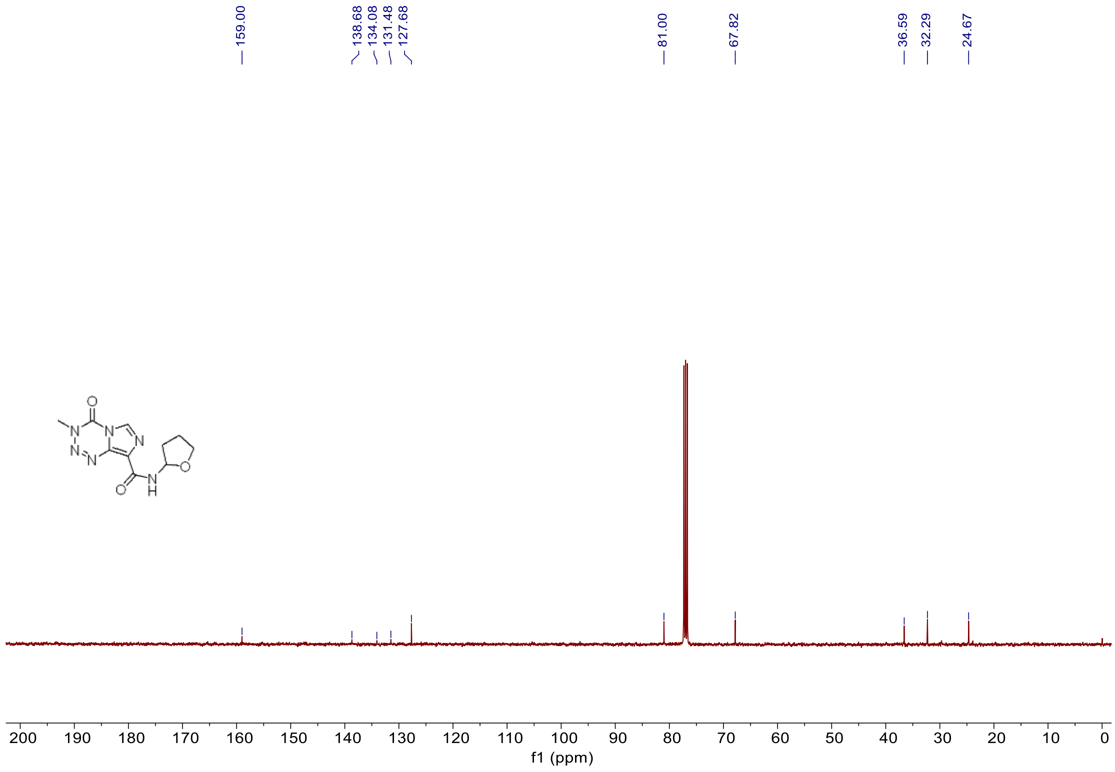
^{13}C NMR spectrum (101 MHz) of **51c** (see procedure)



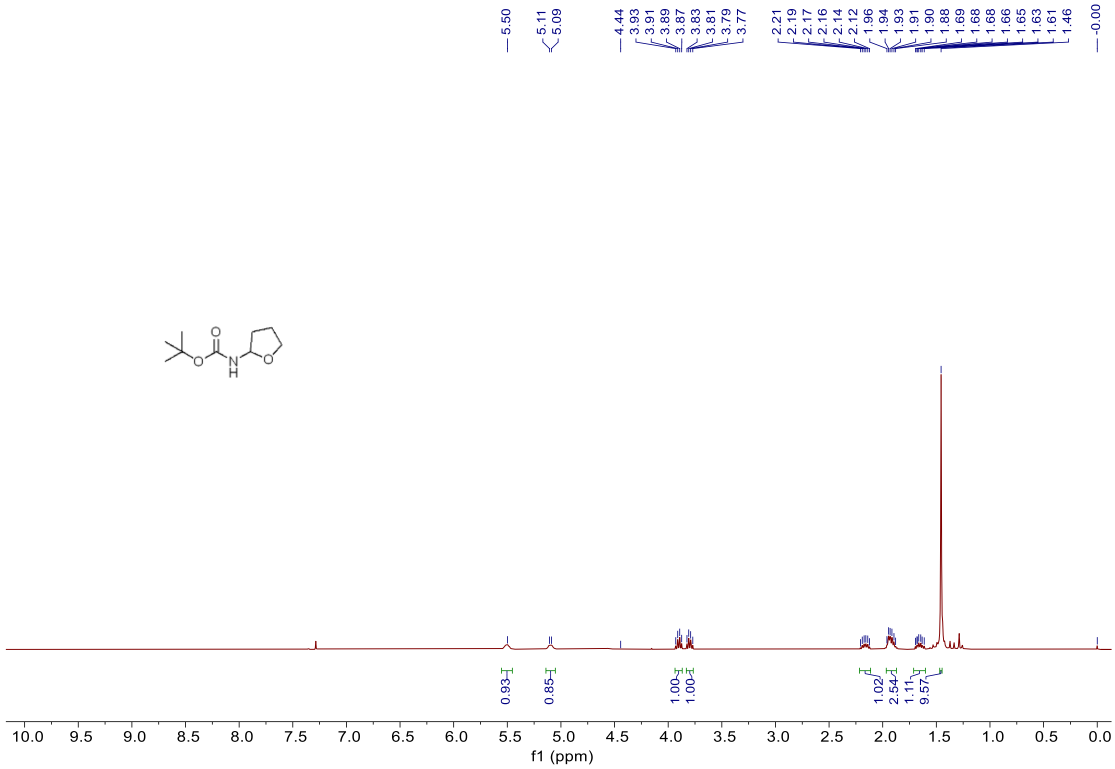
^{1}H NMR spectrum (400 MHz) of **52c** (see procedure)



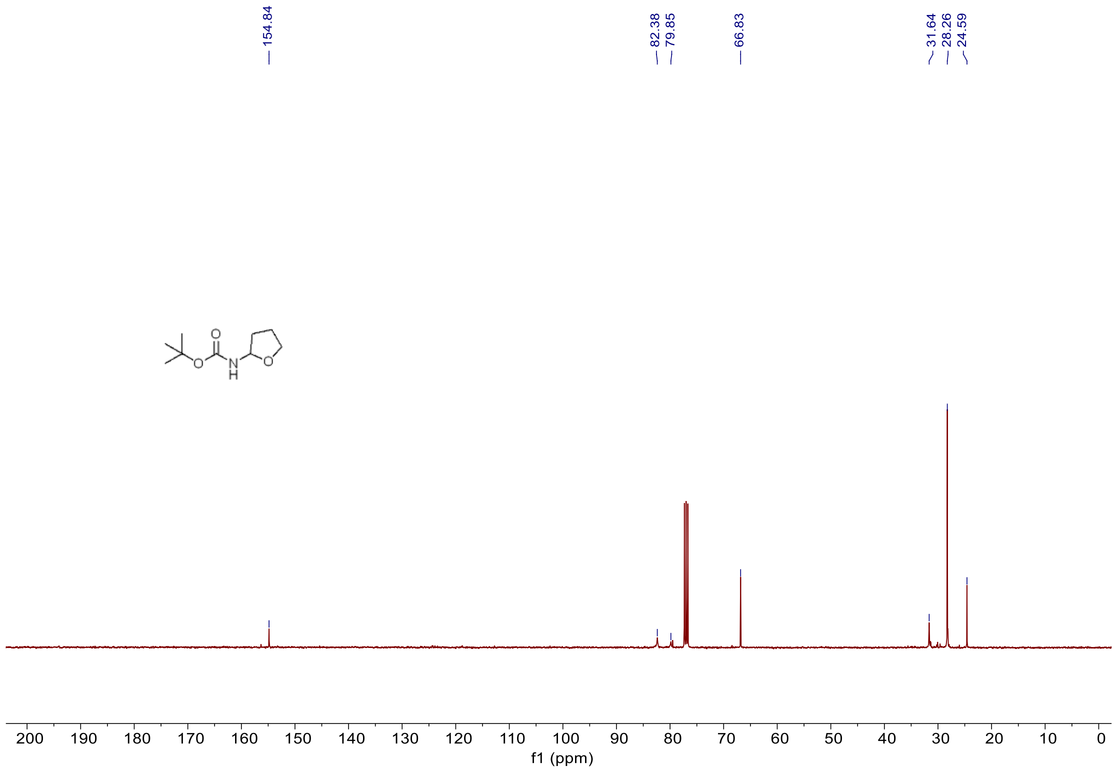
^{13}C NMR spectrum (101 MHz) of **52c** (see procedure)



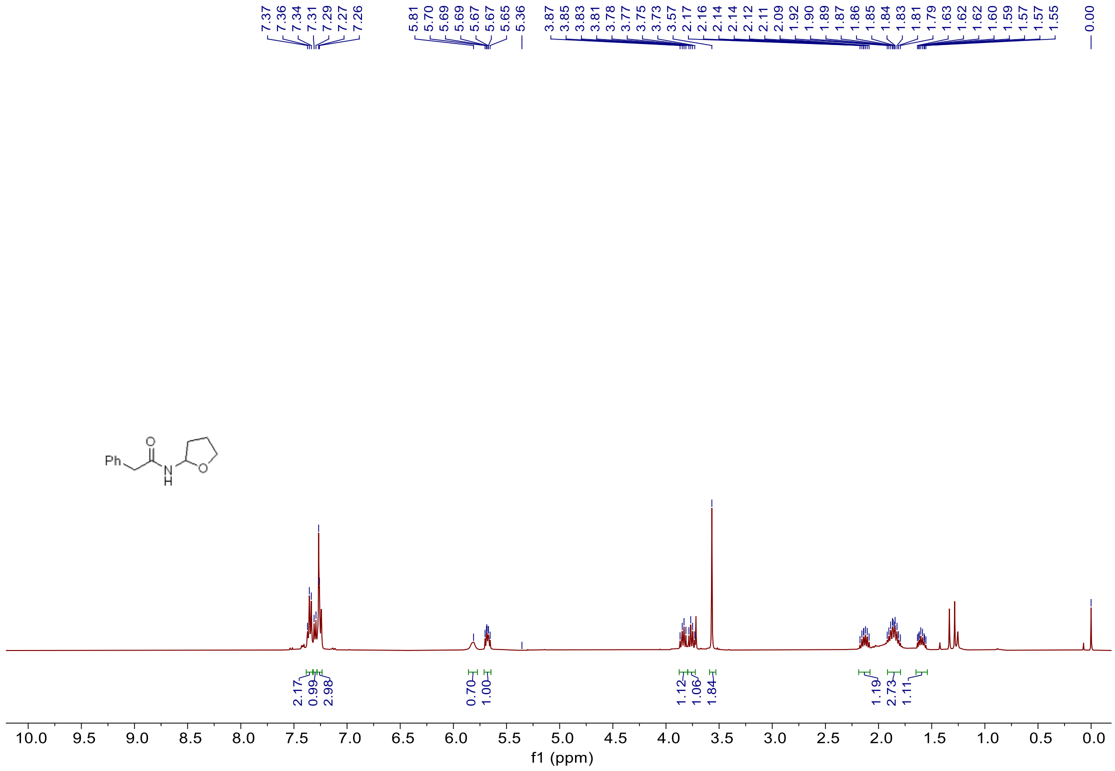
^{1}H NMR spectrum (400 MHz) of **53c** (see procedure)



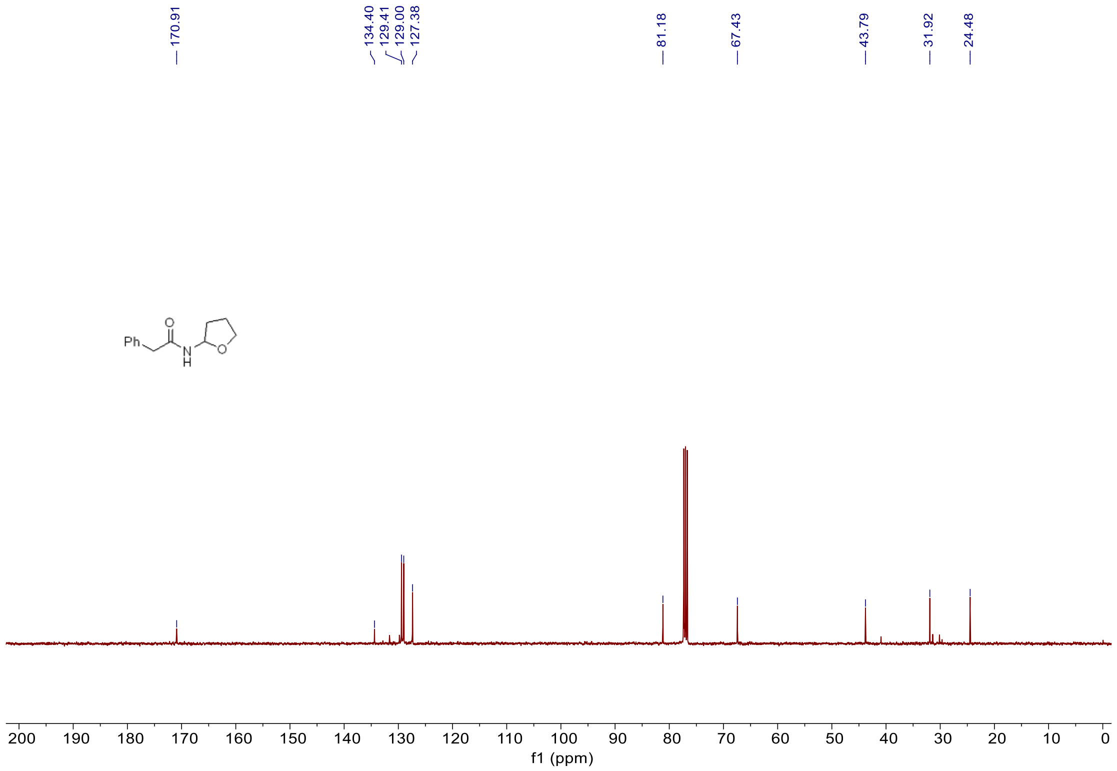
^{13}C NMR spectrum (101 MHz) of **53c** (see procedure)



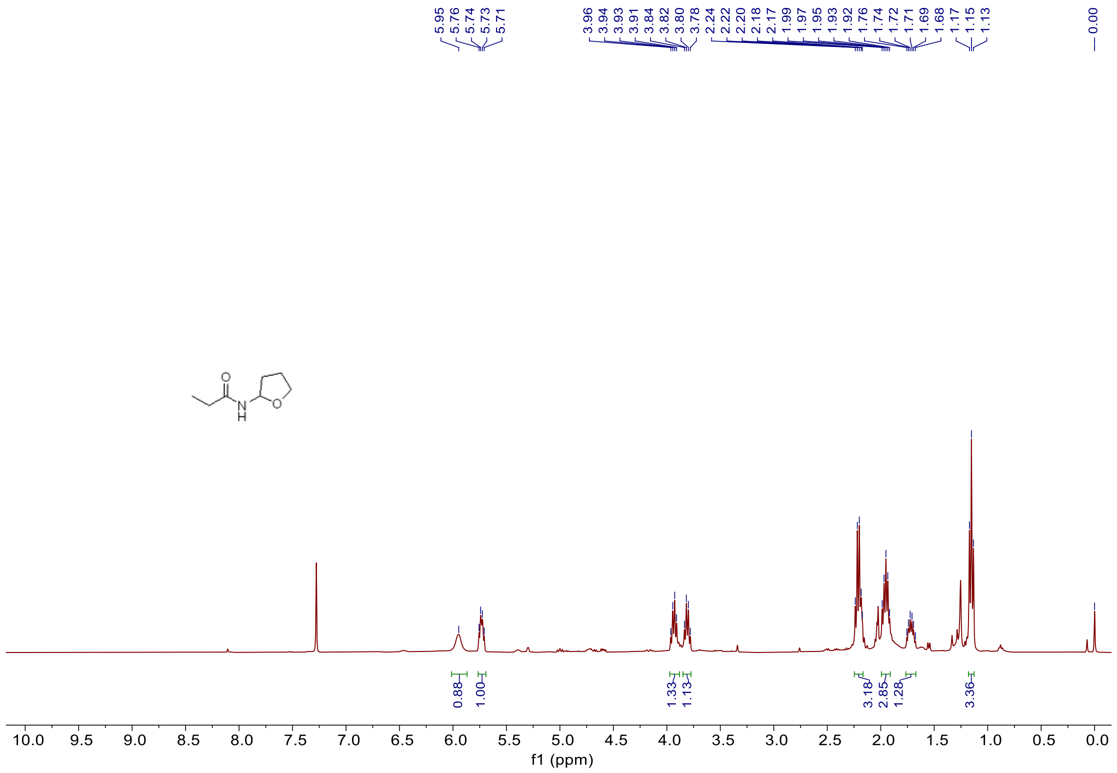
^{1}H NMR spectrum (400 MHz) of **54c** (see procedure)



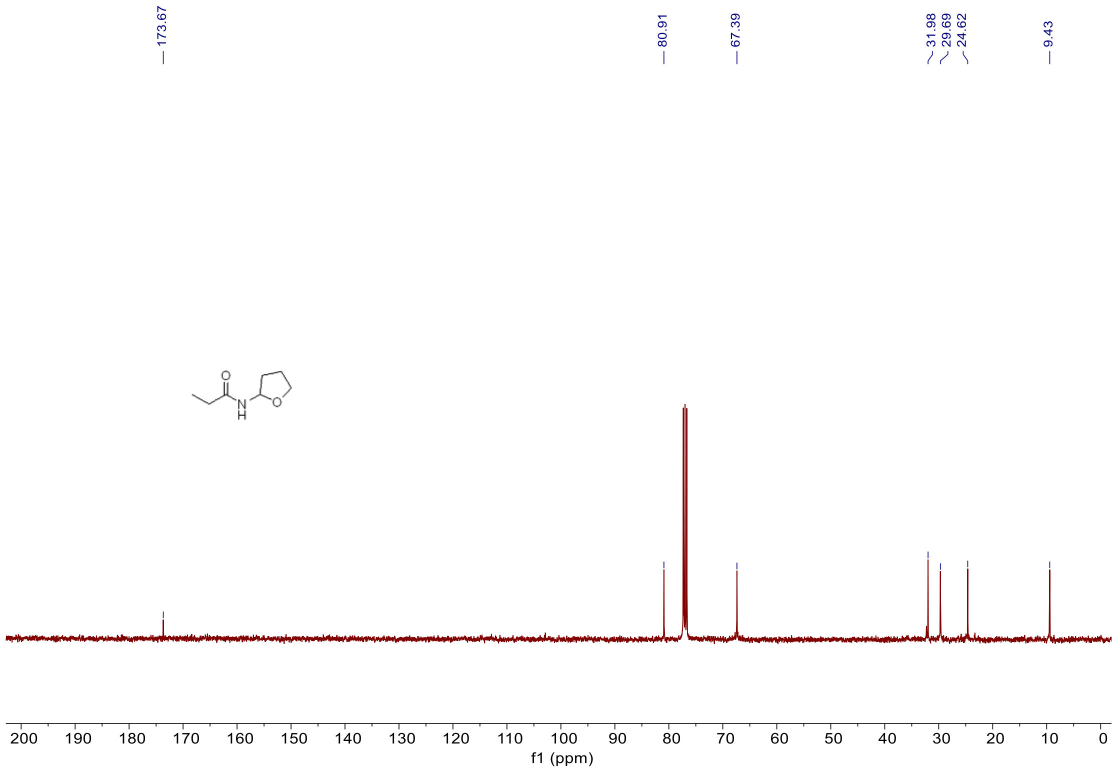
^{13}C NMR spectrum (101 MHz) of **54c** (see procedure)



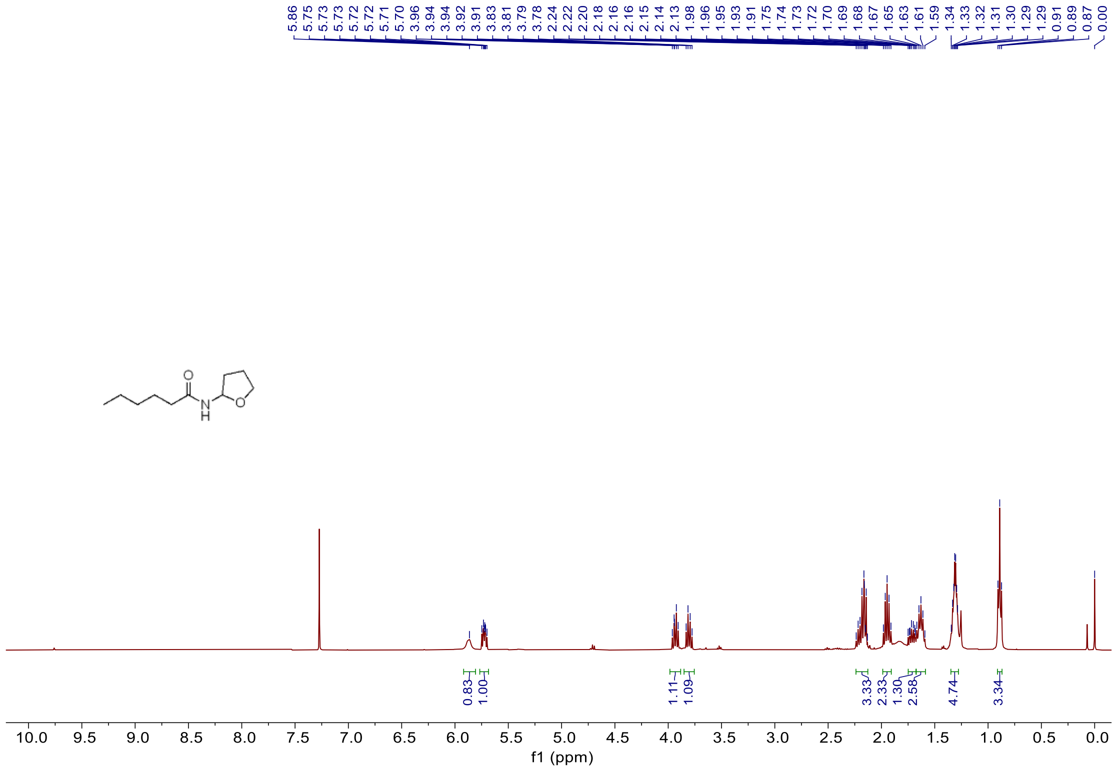
^{1}H NMR spectrum (400 MHz) of **55c** (see procedure)



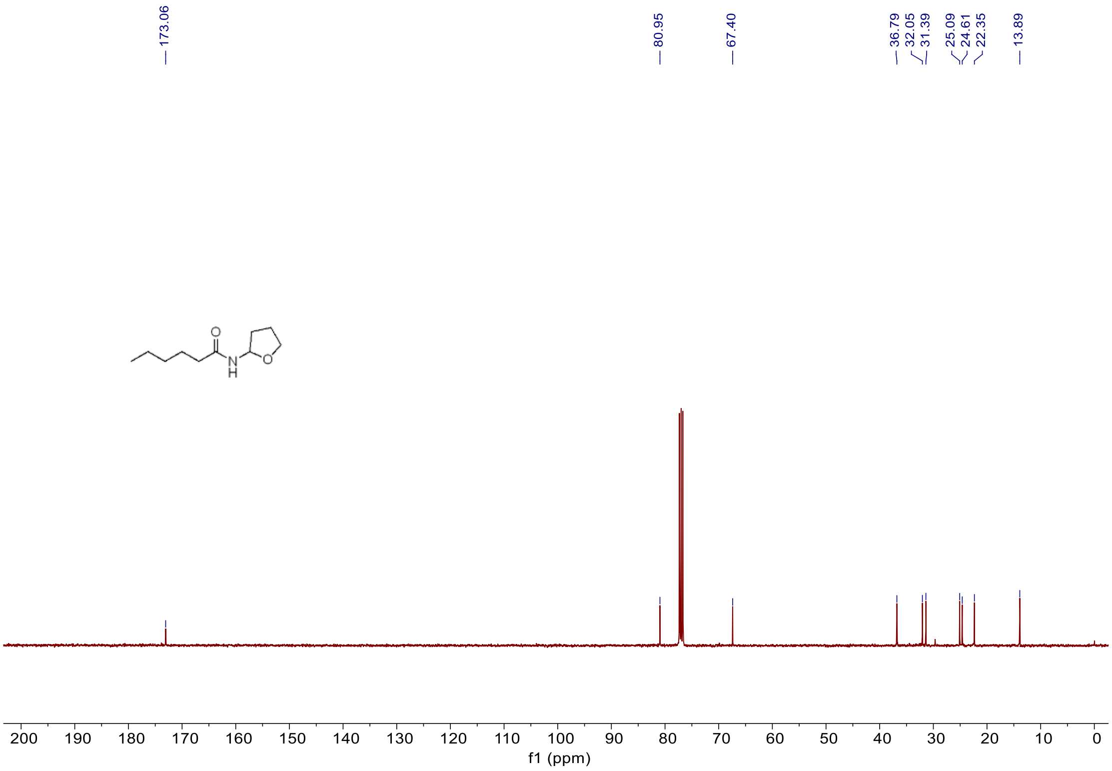
^{13}C NMR spectrum (101 MHz) of **55c** (see procedure)



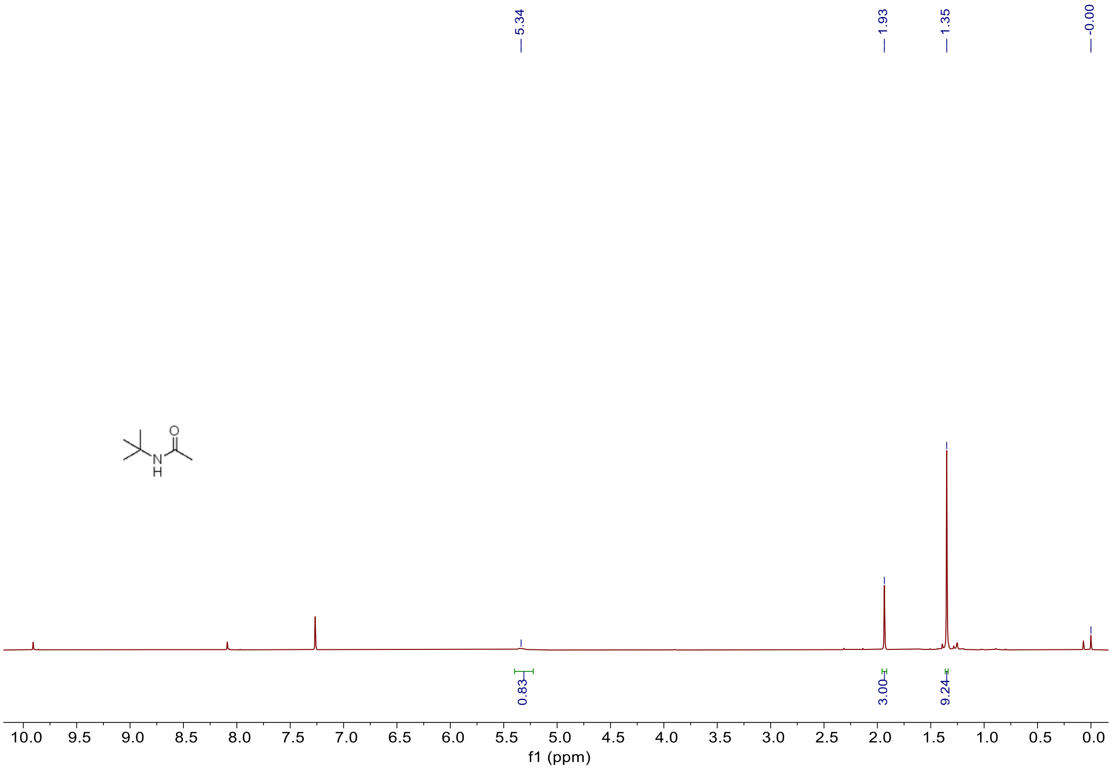
^{1}H NMR spectrum (400 MHz) of **56c** (see procedure)



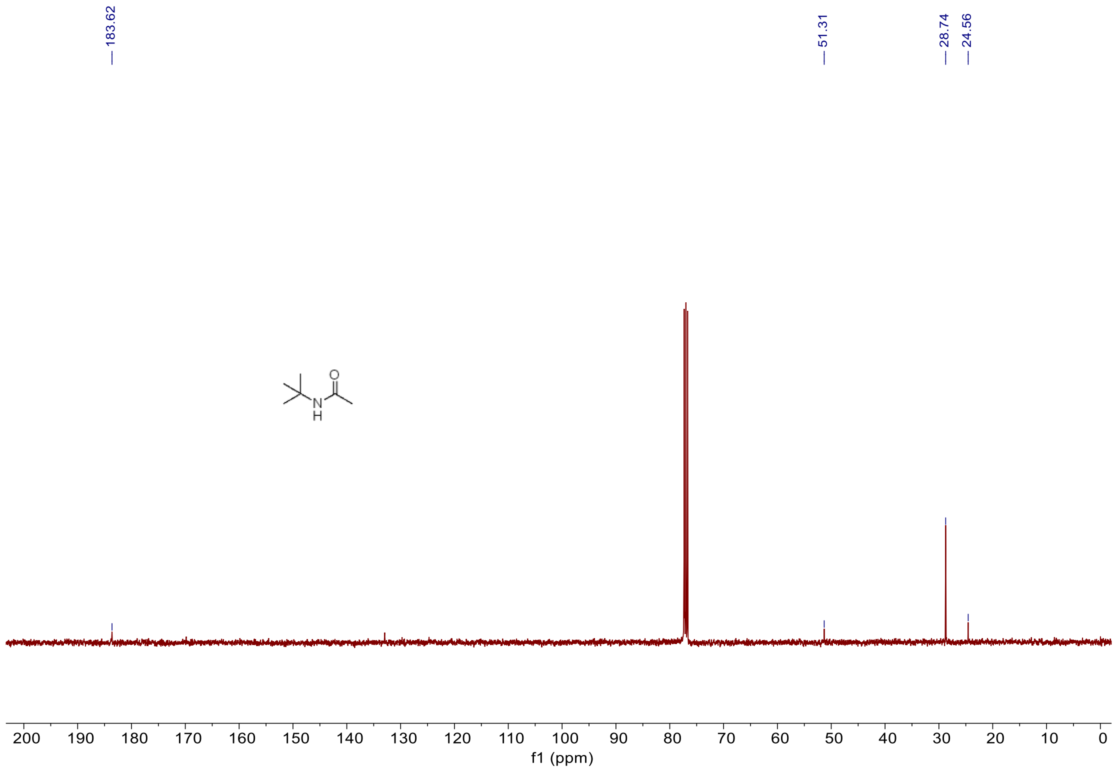
^{13}C NMR spectrum (101 MHz) of **56c** (see procedure)



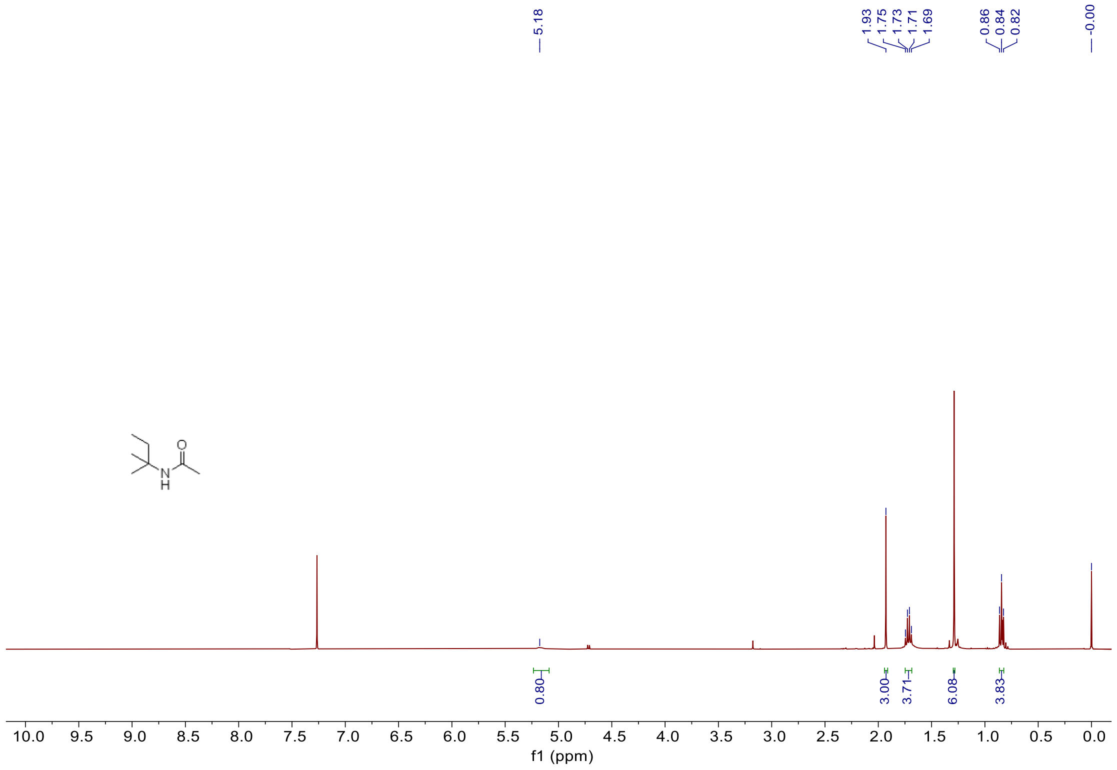
^{1}H NMR spectrum (400 MHz) of **57c** (see procedure)



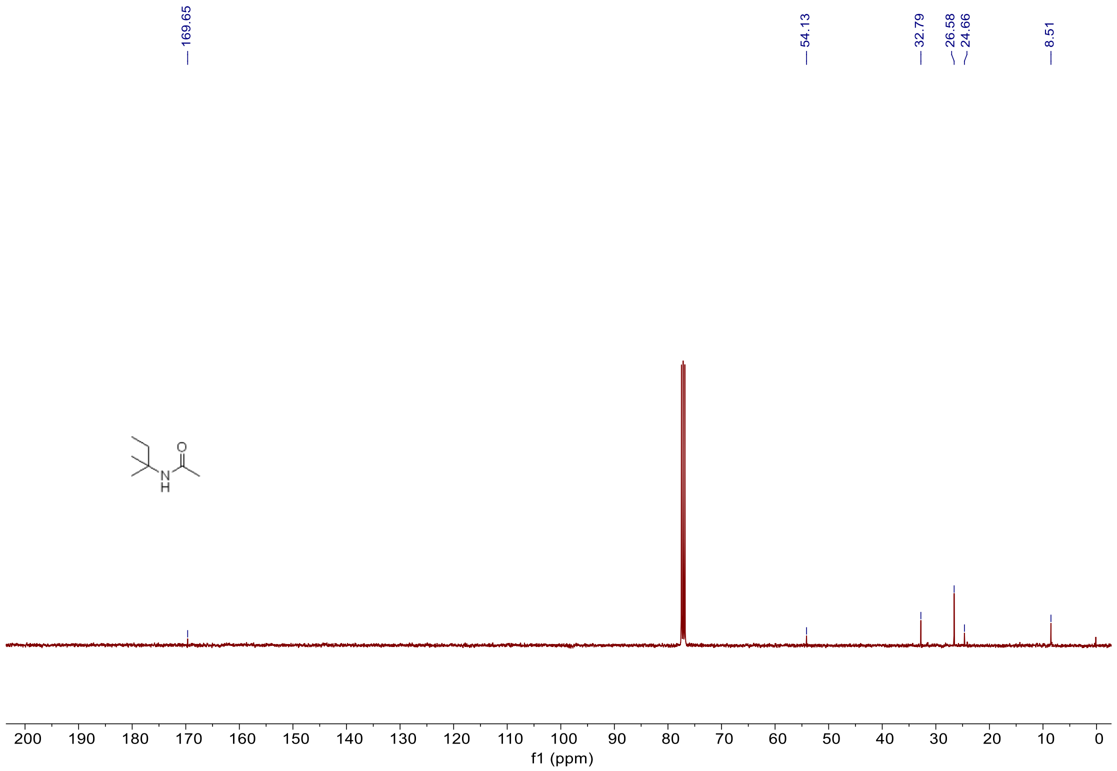
^{13}C NMR spectrum (101 MHz) of **57c** (see procedure)



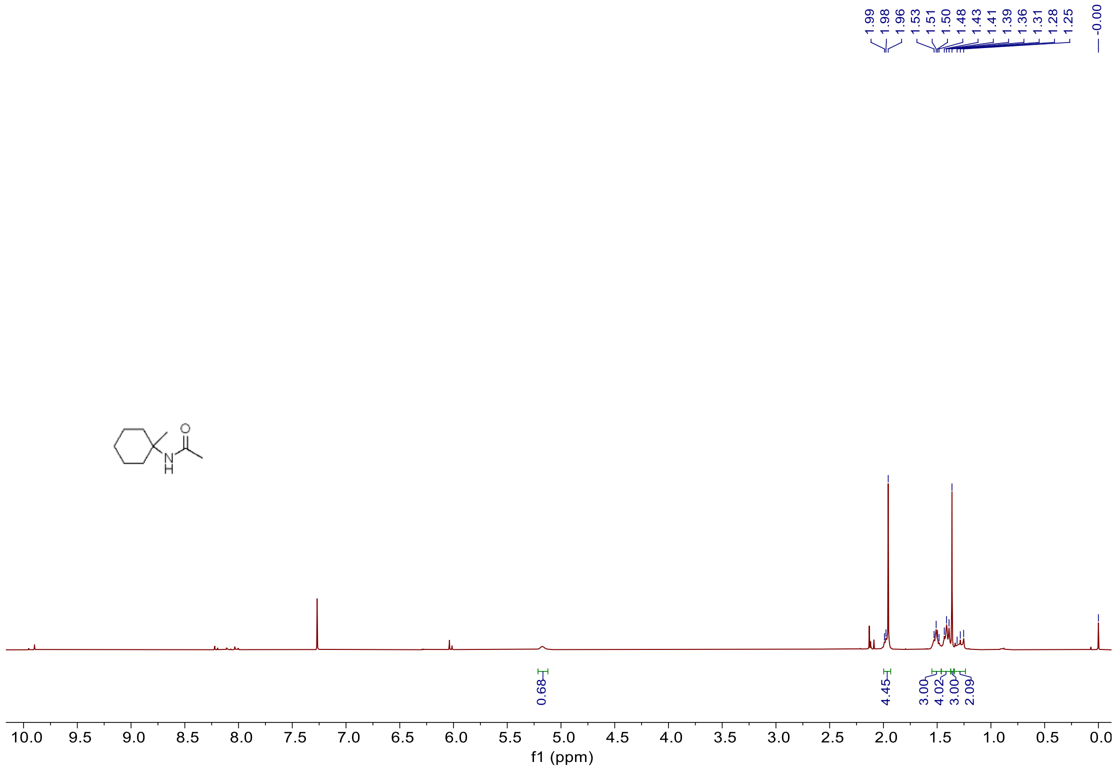
^{1}H NMR spectrum (400 MHz) of **58c** (see procedure)



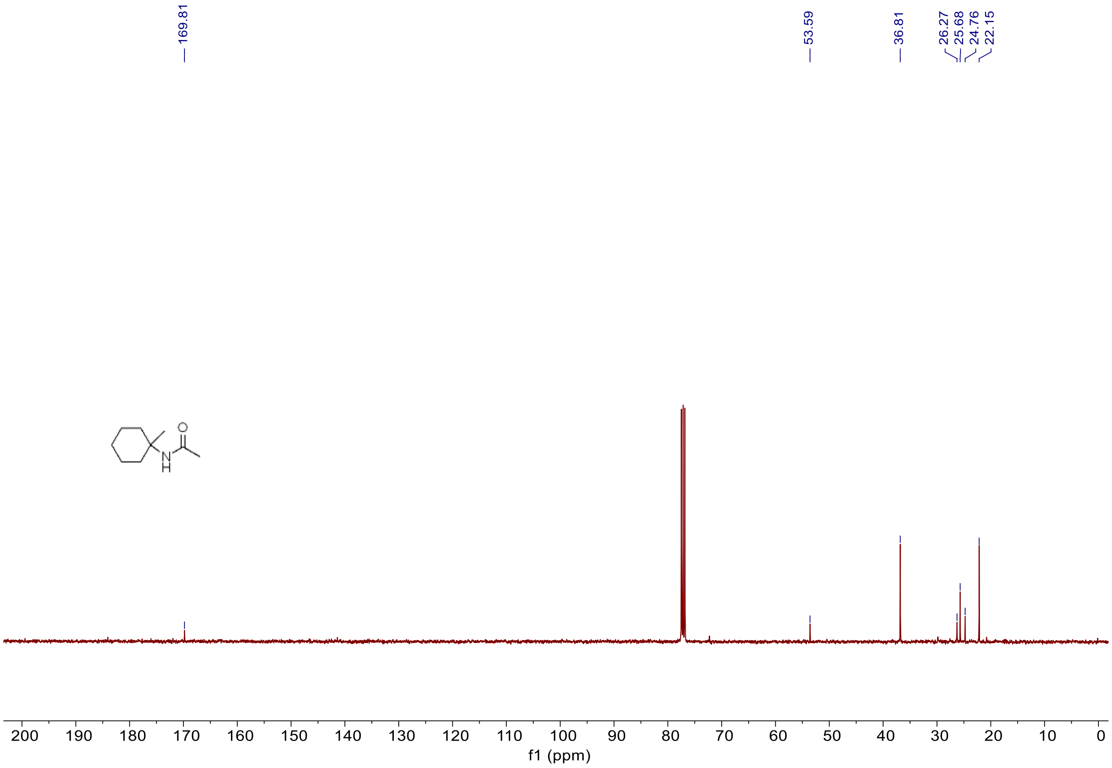
^{13}C NMR spectrum (101 MHz) of **58c** (see procedure)



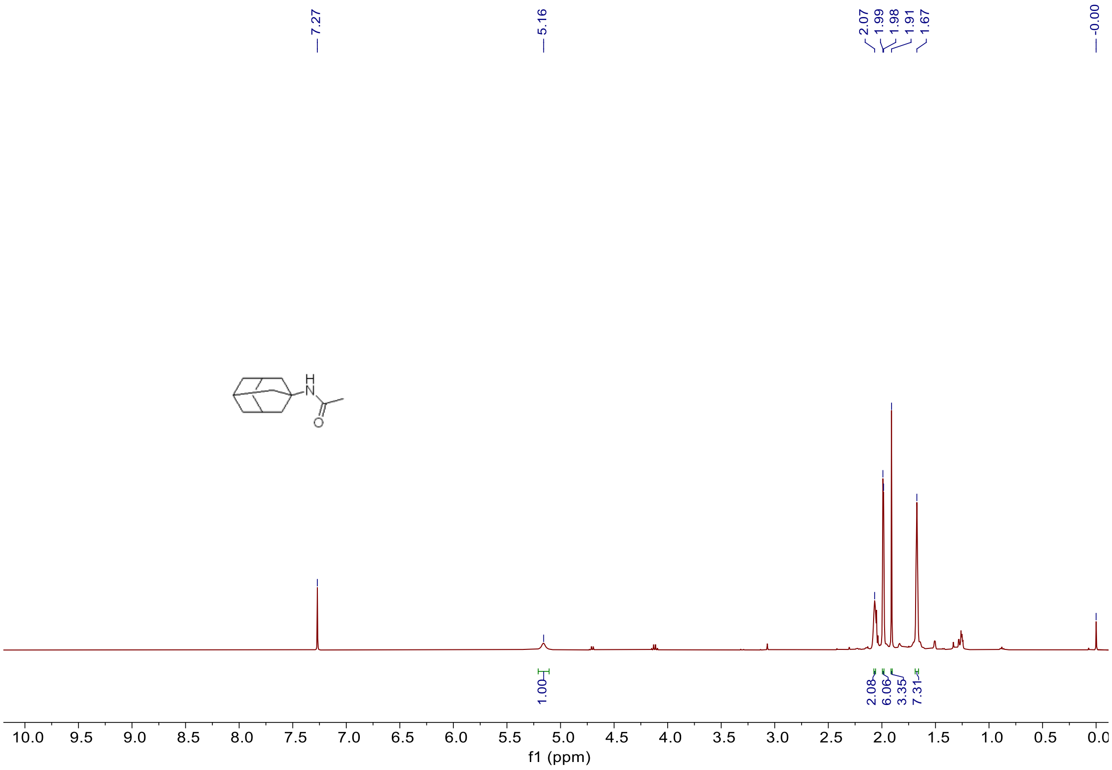
^{1}H NMR spectrum (400 MHz) of **59c** (see procedure)



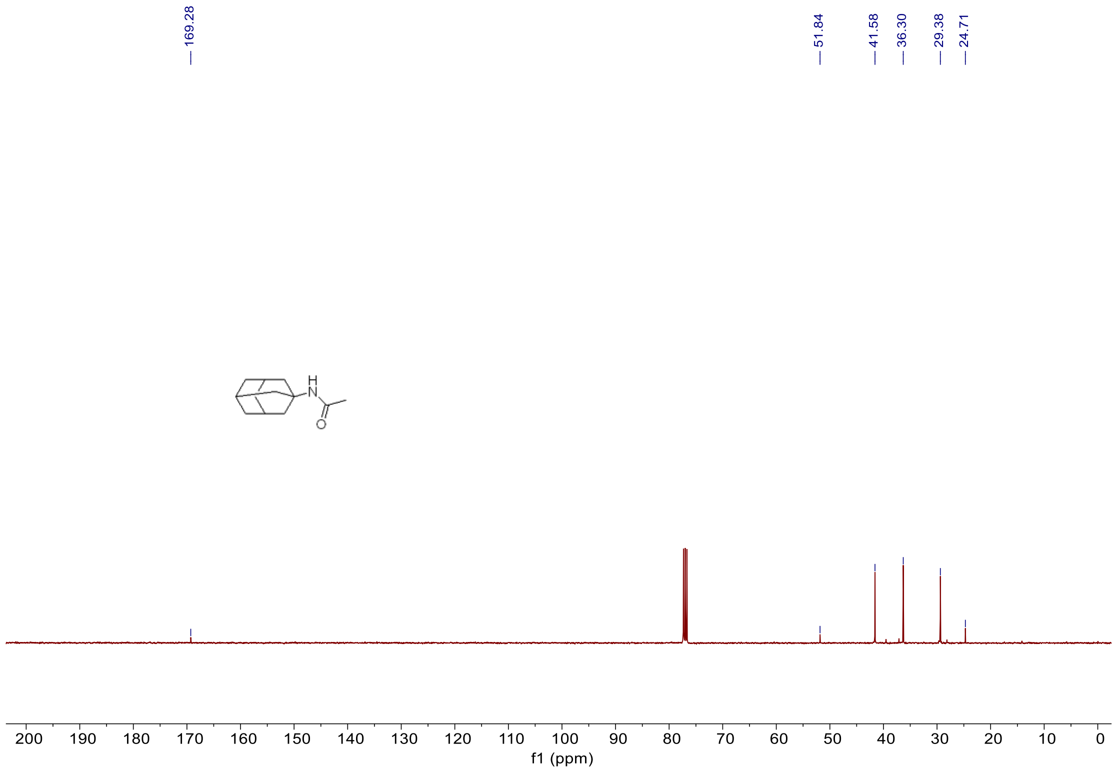
^{13}C NMR spectrum (101 MHz) of **59c** (see procedure)



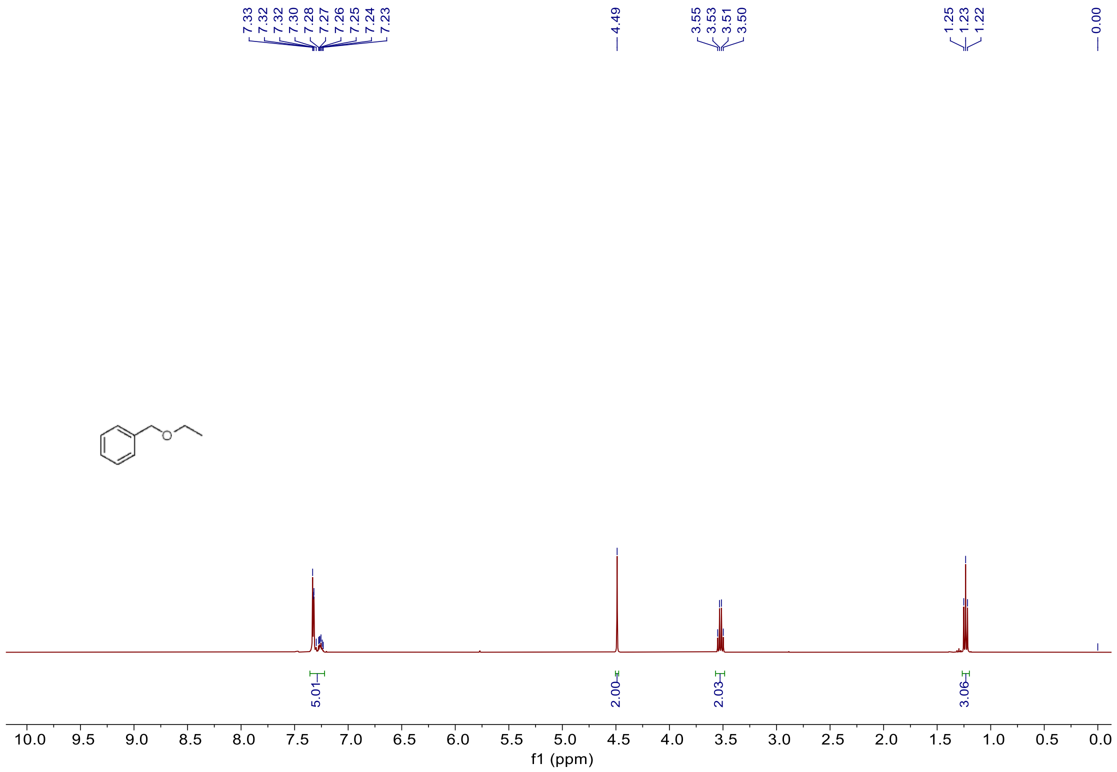
^{1}H NMR spectrum (400 MHz) of **60c** (see procedure)



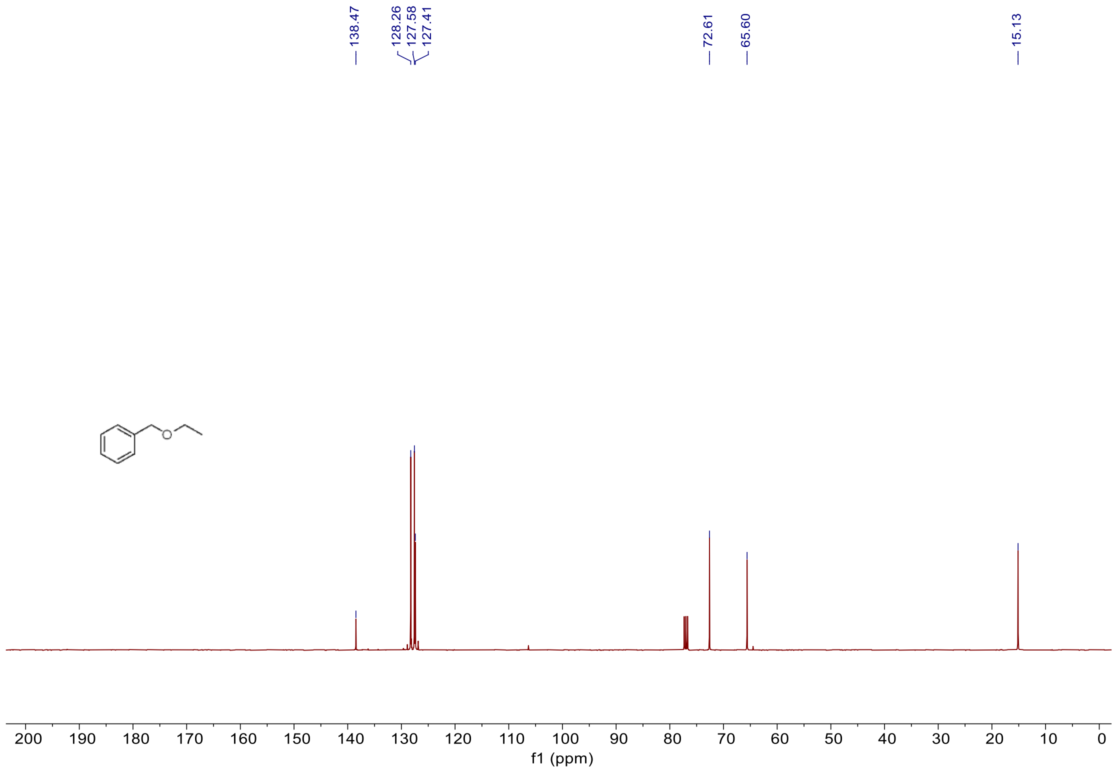
^{13}C NMR spectrum (101 MHz) of **60c** (see procedure)



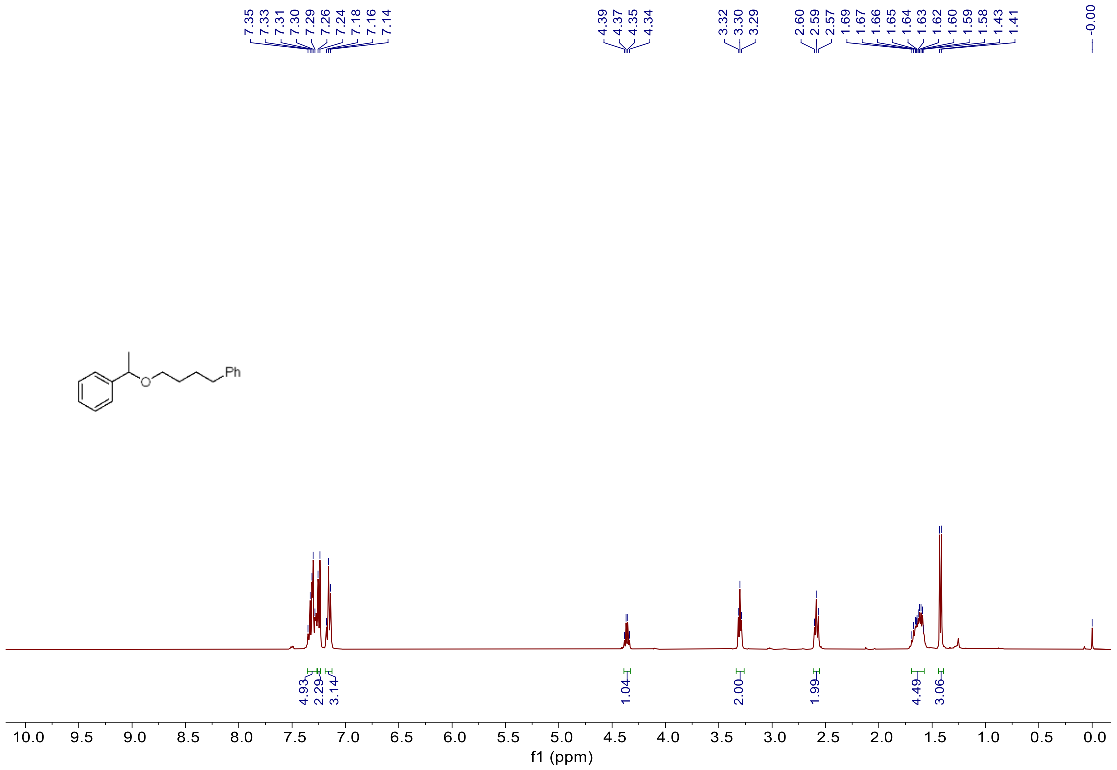
^{1}H NMR spectrum (400 MHz) of **61c** (see procedure)



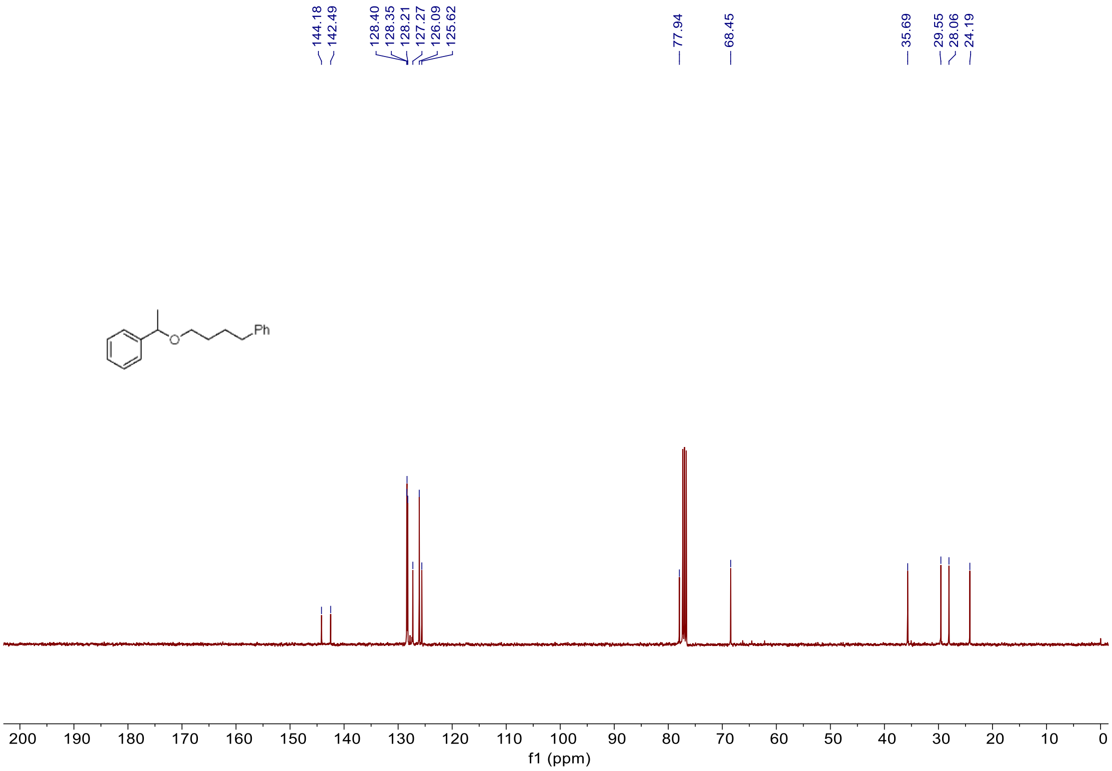
^{13}C NMR spectrum (101 MHz) of **61c** (see procedure)



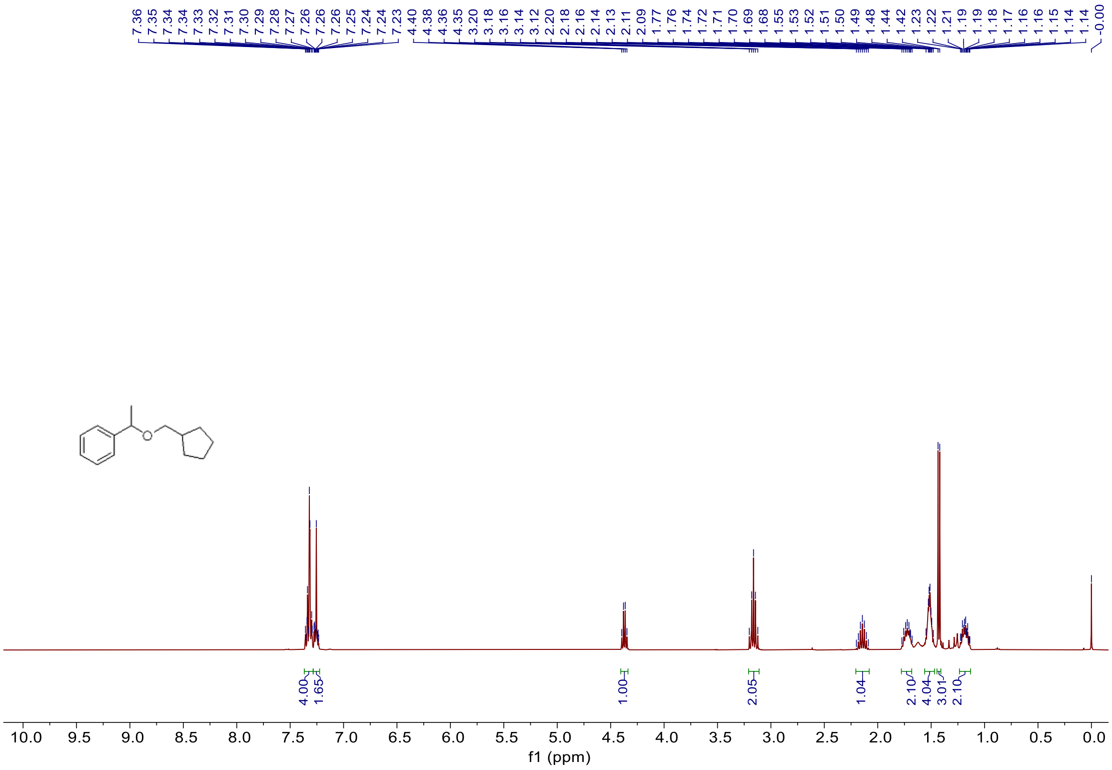
^{1}H NMR spectrum (400 MHz) of **62c** (see procedure)



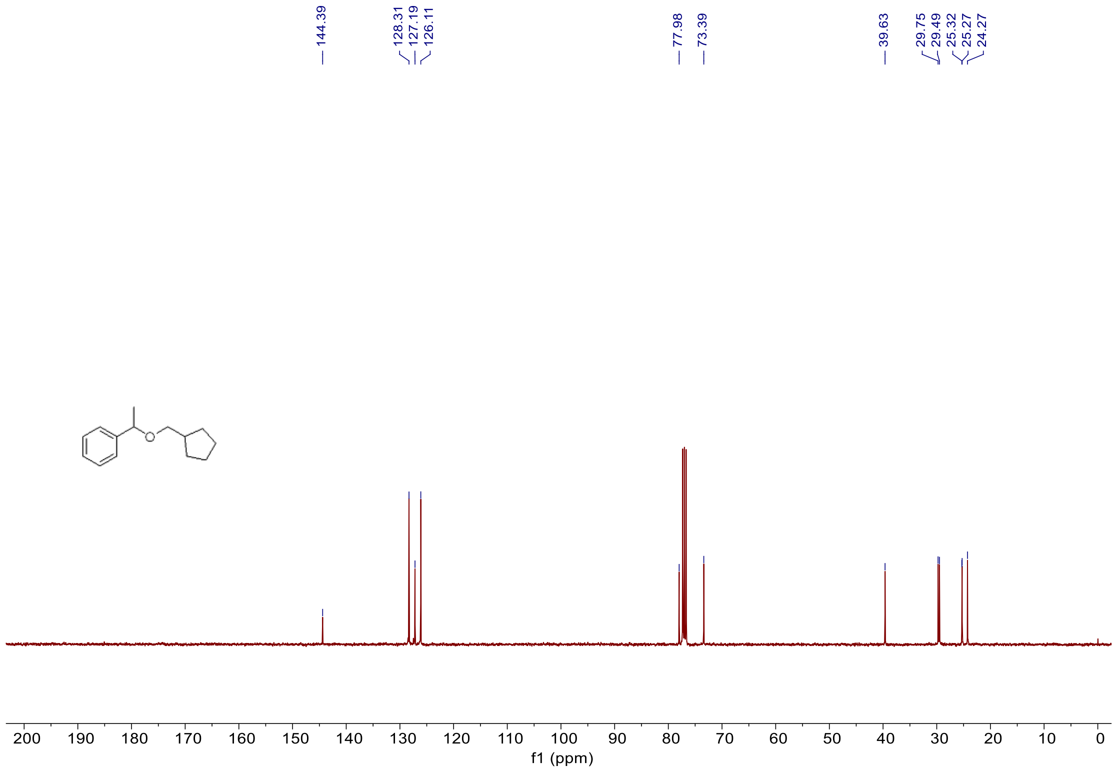
^{13}C NMR spectrum (101 MHz) of **62c** (see procedure)



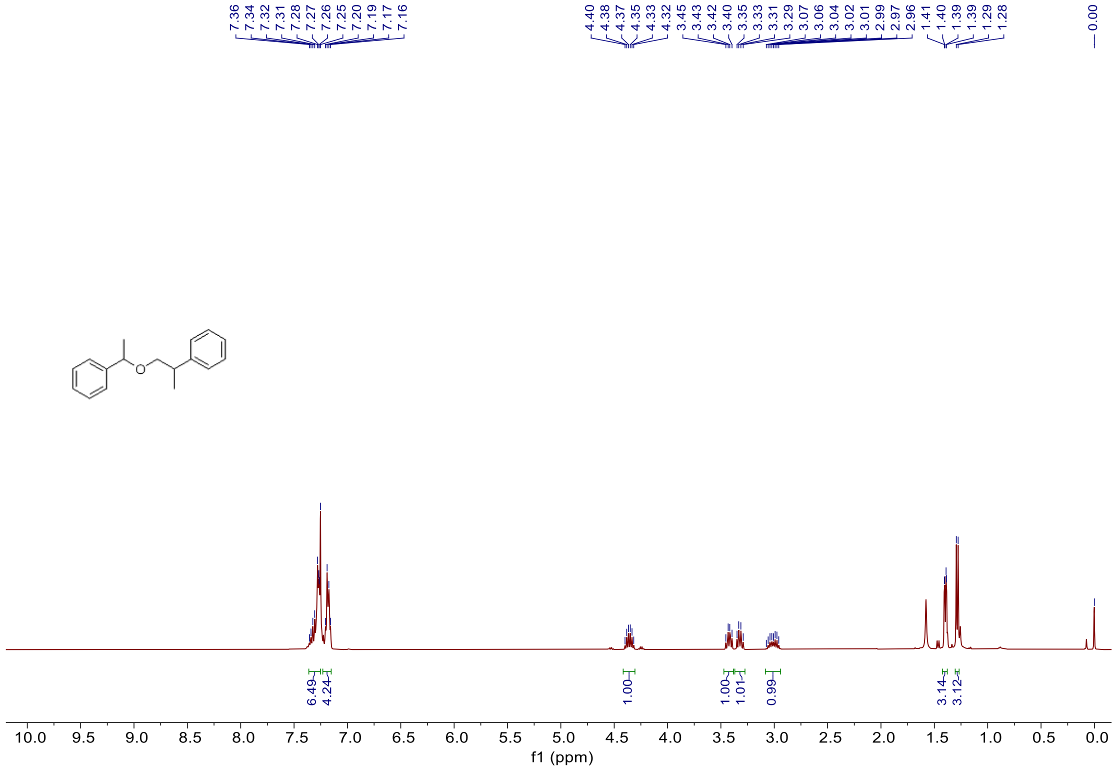
^{1}H NMR spectrum (400 MHz) of **63c** (see procedure)



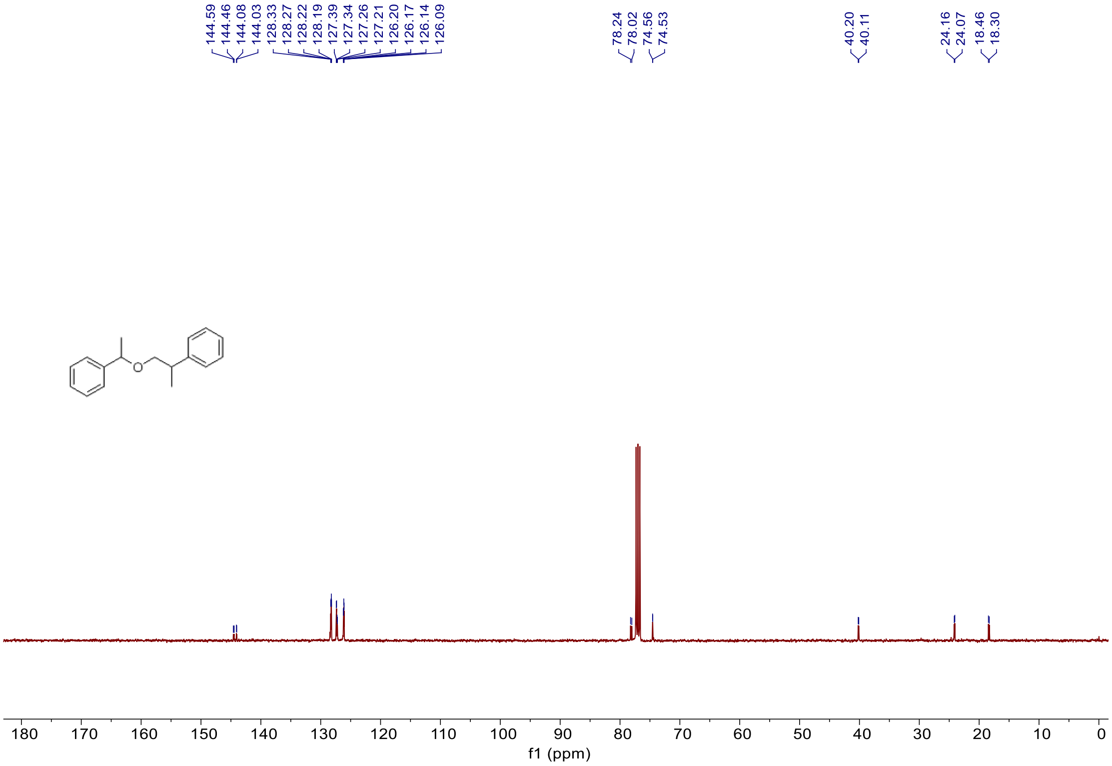
^{13}C NMR spectrum (101 MHz) of **63c** (see procedure)



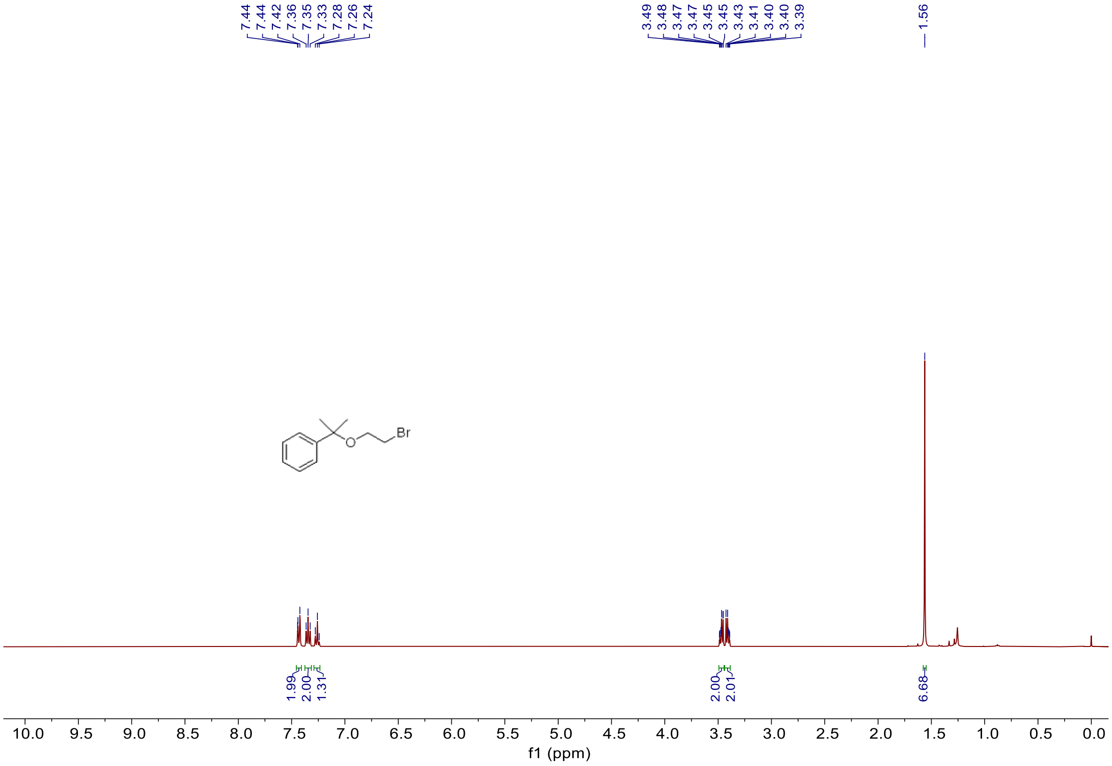
^{1}H NMR spectrum (400 MHz) of **64c** (see procedure)



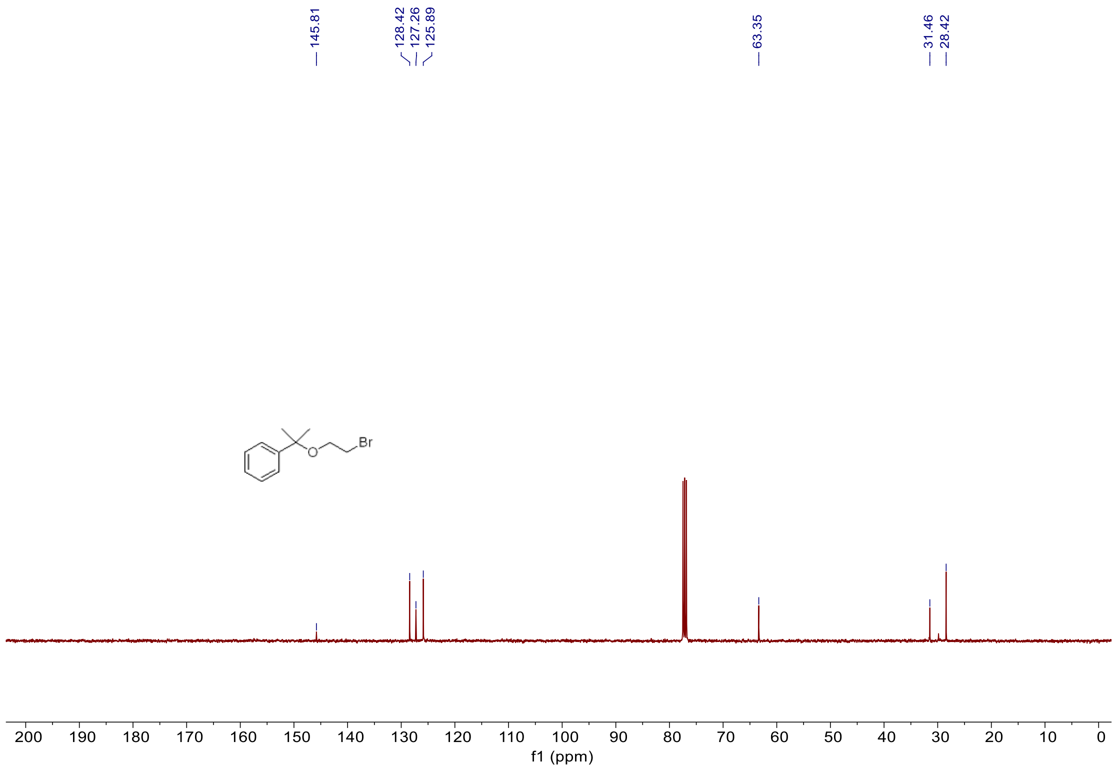
^{13}C NMR spectrum (101 MHz) of **64c** (see procedure)



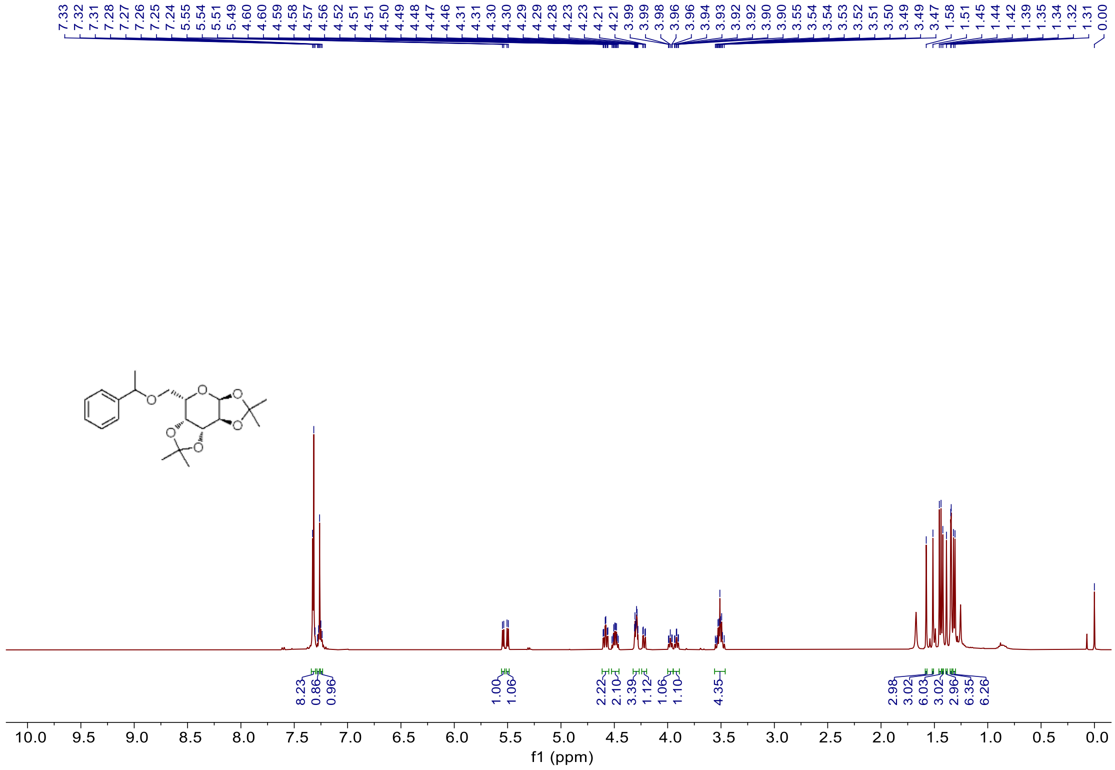
^{1}H NMR spectrum (400 MHz) of **65c** (see procedure)



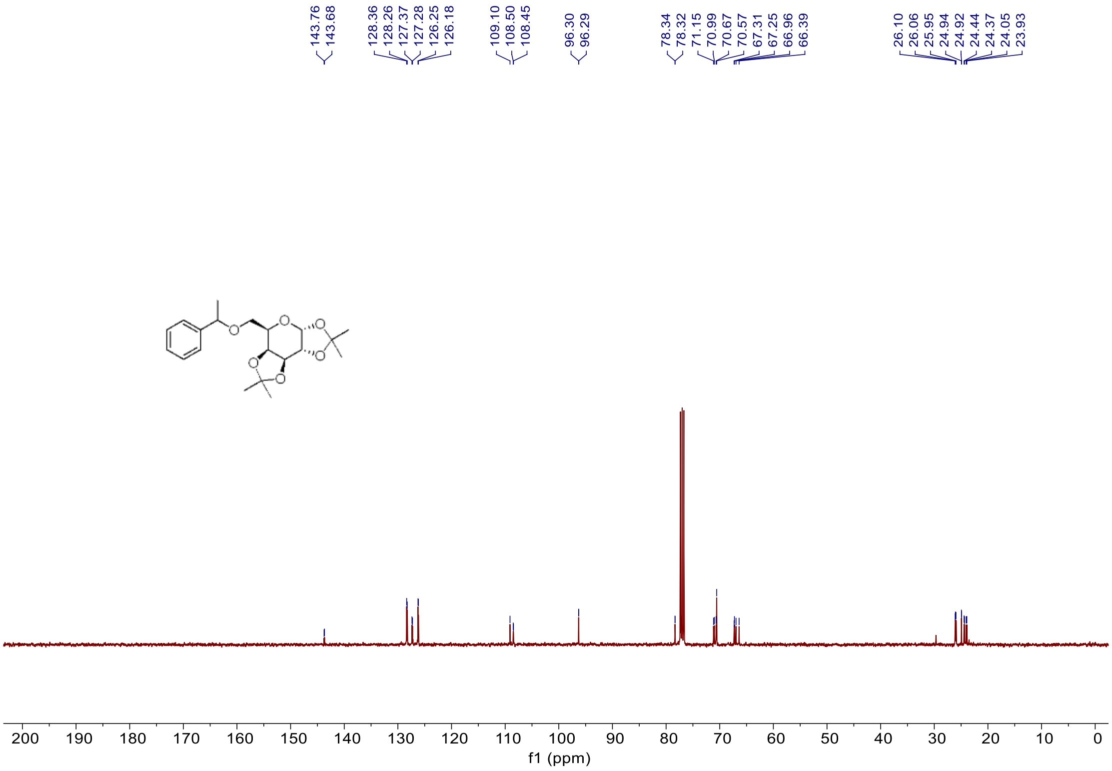
^{13}C NMR spectrum (101 MHz) of **65c** (see procedure)



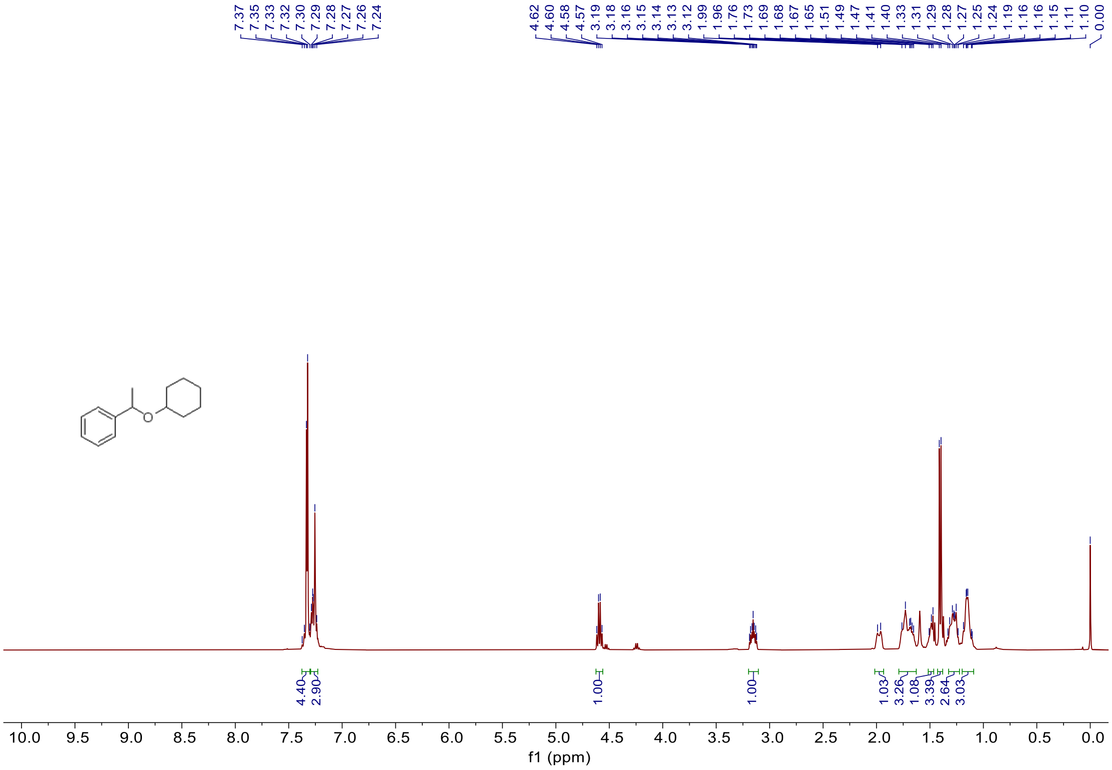
^{1}H NMR spectrum (400 MHz) of **66c** (see procedure)



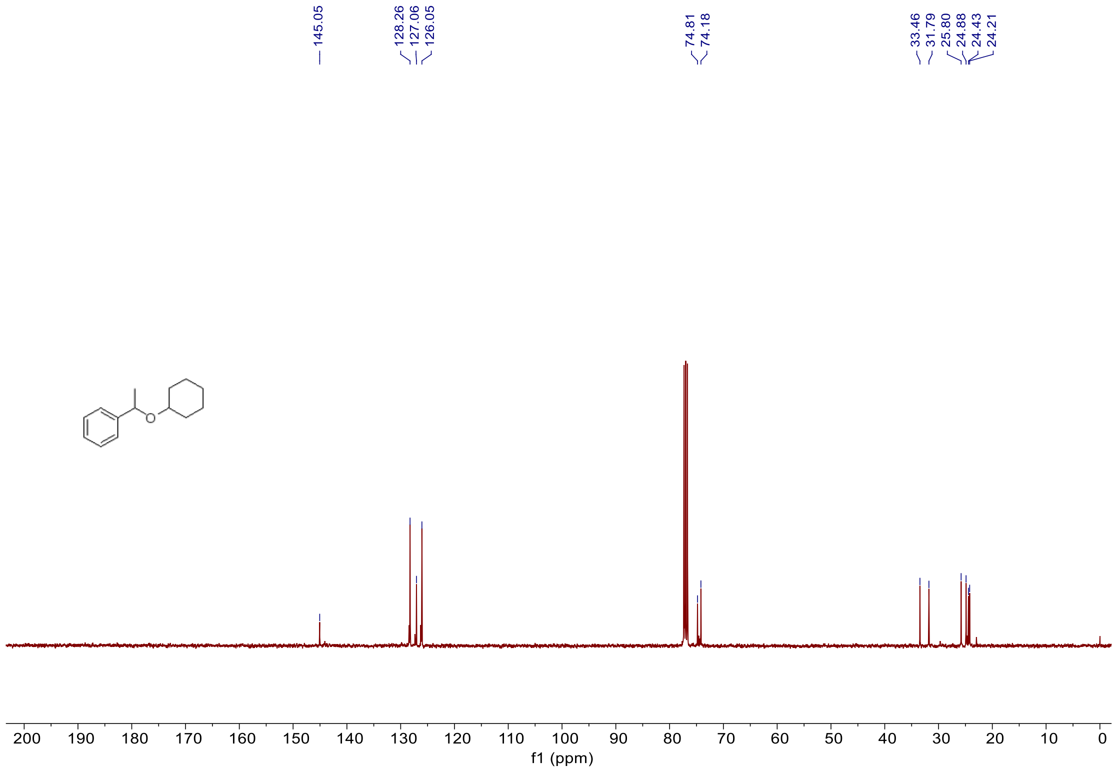
^{13}C NMR spectrum (101 MHz) of **66c** (see procedure)



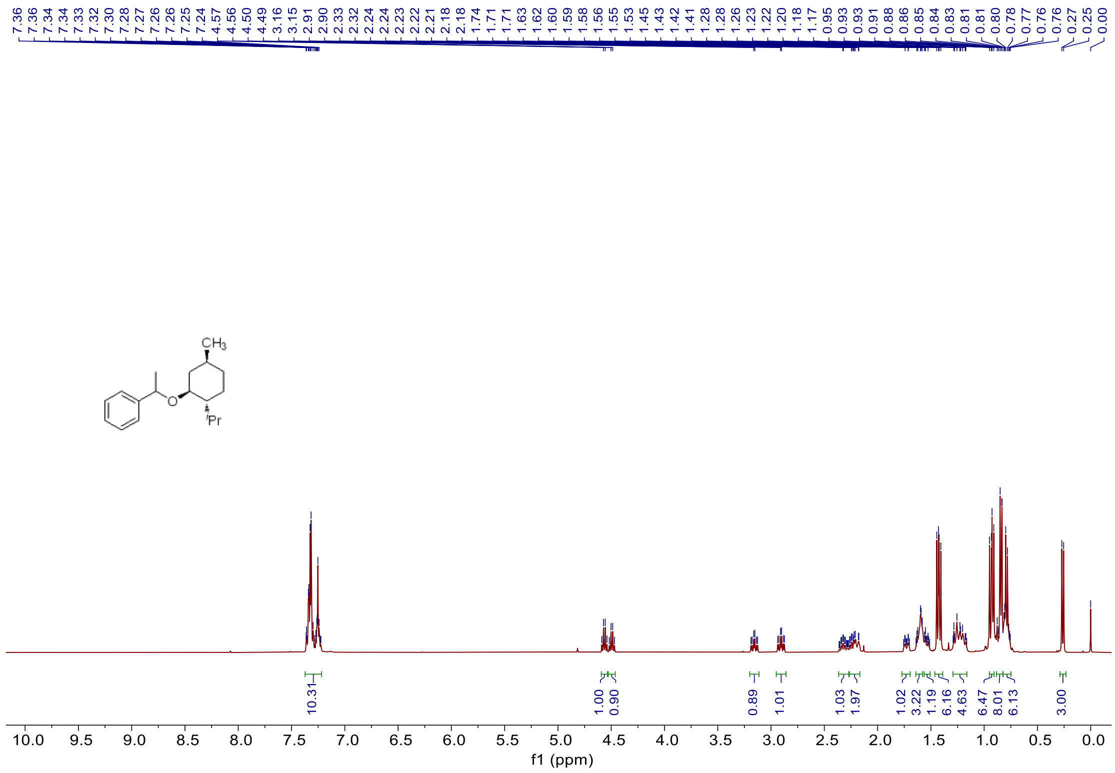
^{1}H NMR spectrum (400 MHz) of **67c** (see procedure)



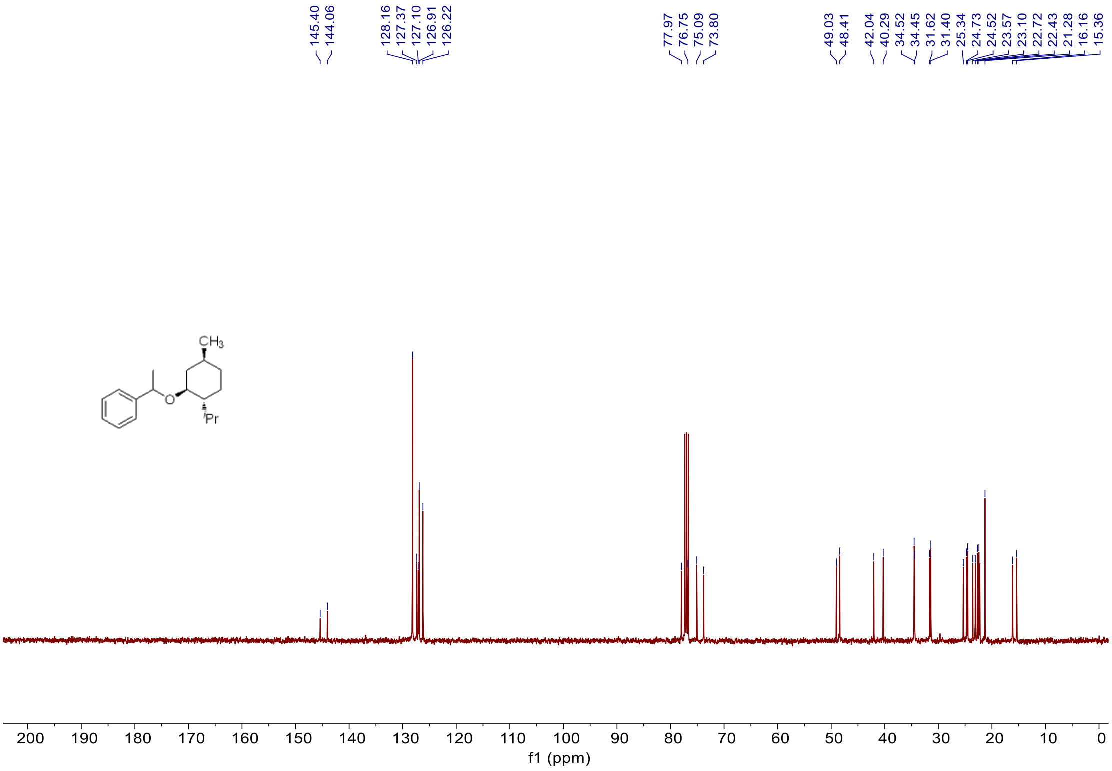
^{13}C NMR spectrum (101 MHz) of **67c** (see procedure)



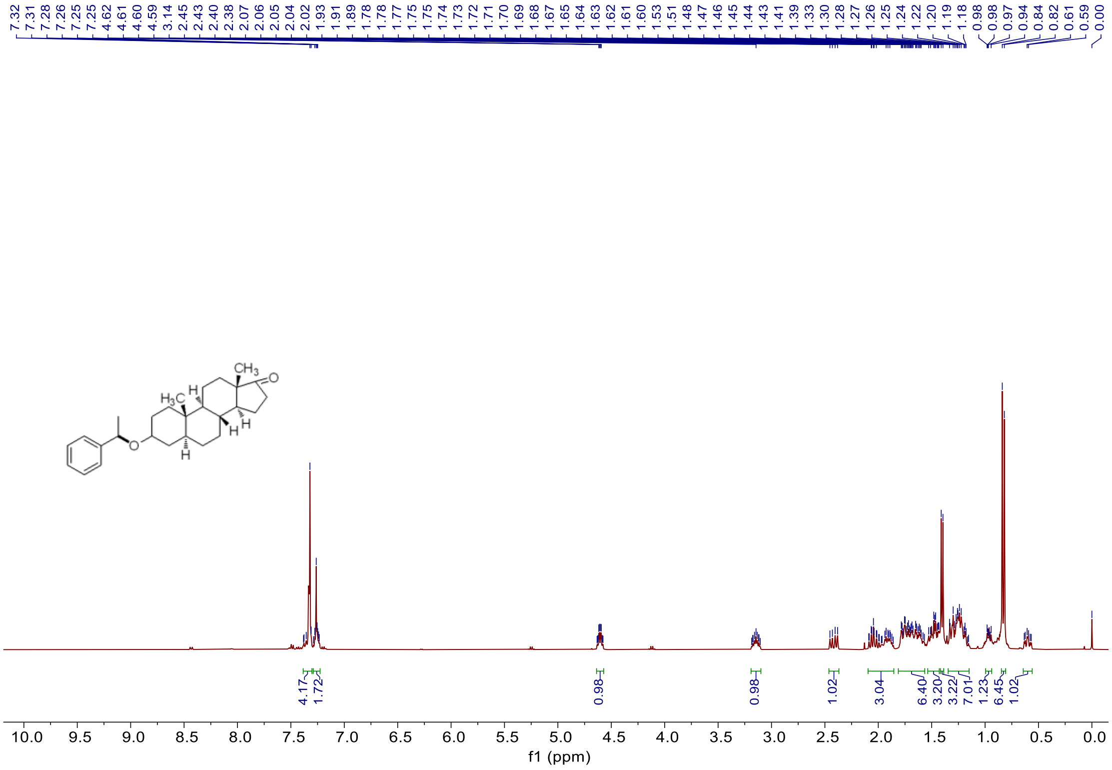
^{1}H NMR spectrum (400 MHz) of **68c** (see procedure)



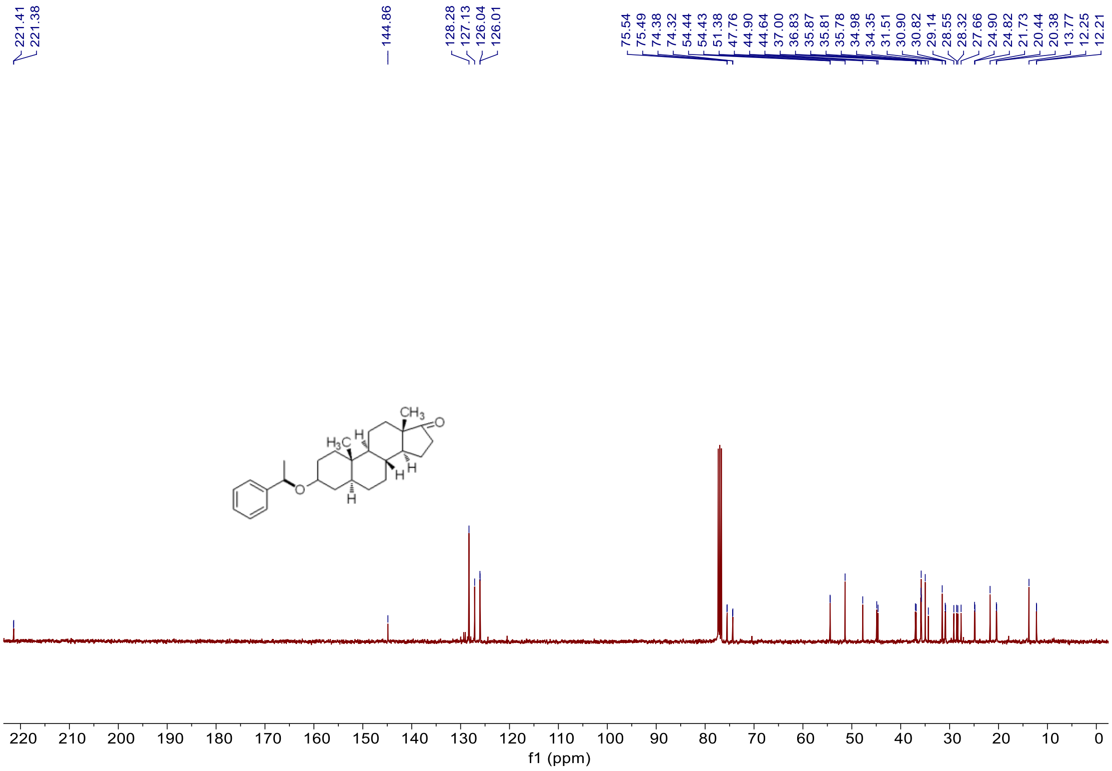
^{13}C NMR spectrum (101 MHz) of **68c** (see procedure)



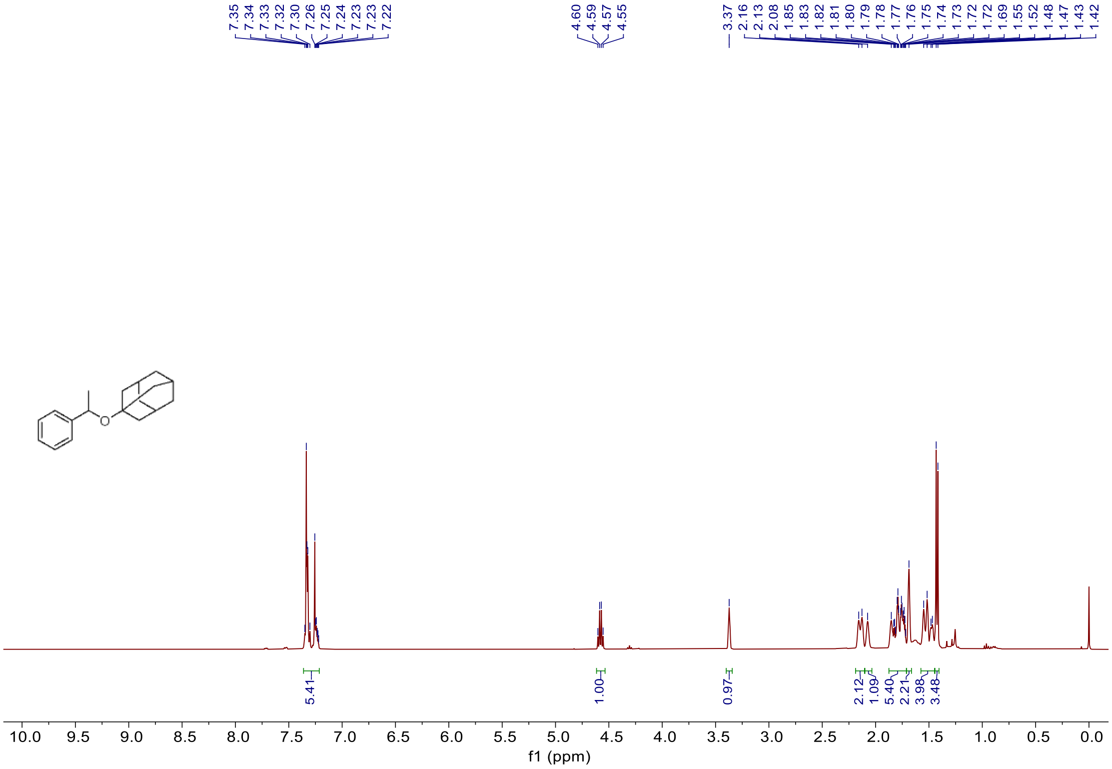
^{1}H NMR spectrum (400 MHz) of **69c** (see procedure)



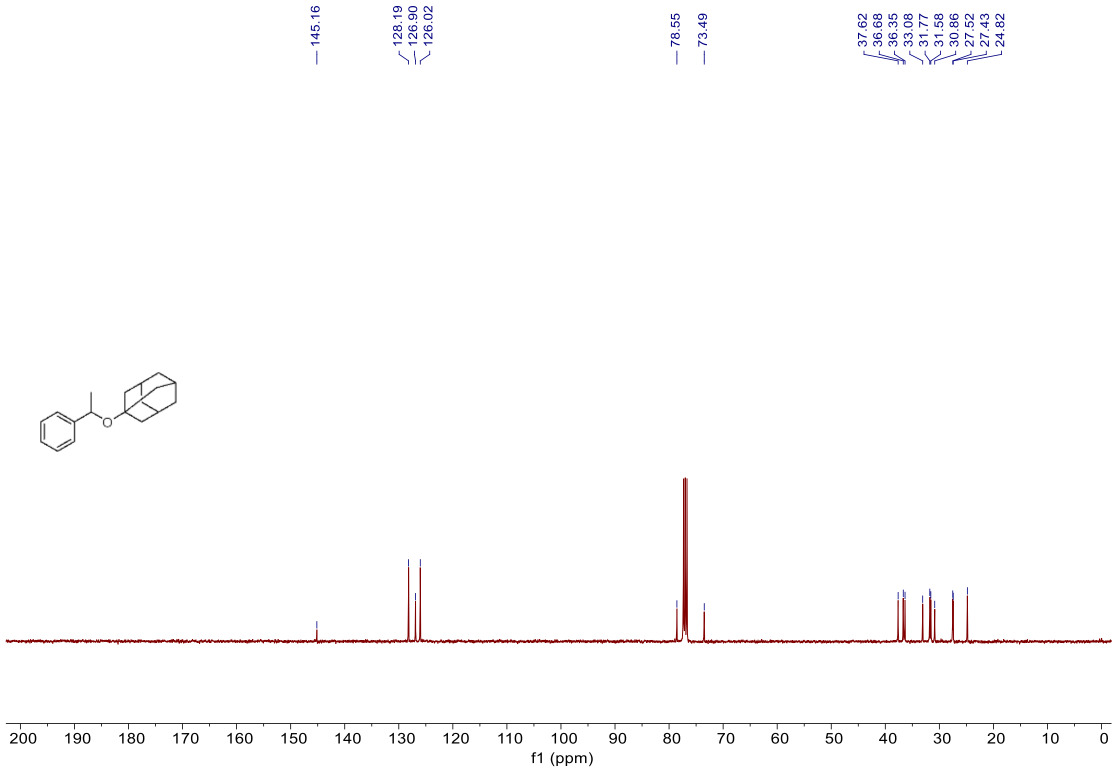
^{13}C NMR spectrum (101 MHz) of **69c** (see procedure)



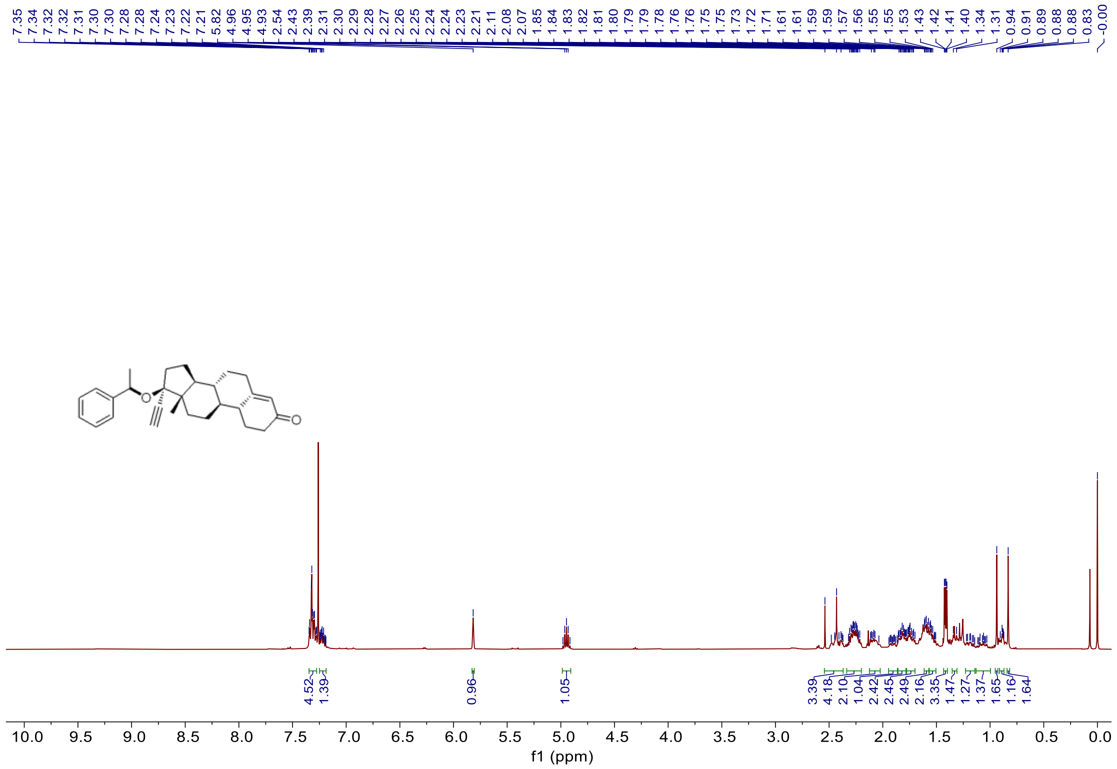
^{1}H NMR spectrum (400 MHz) of **70c** (see procedure)



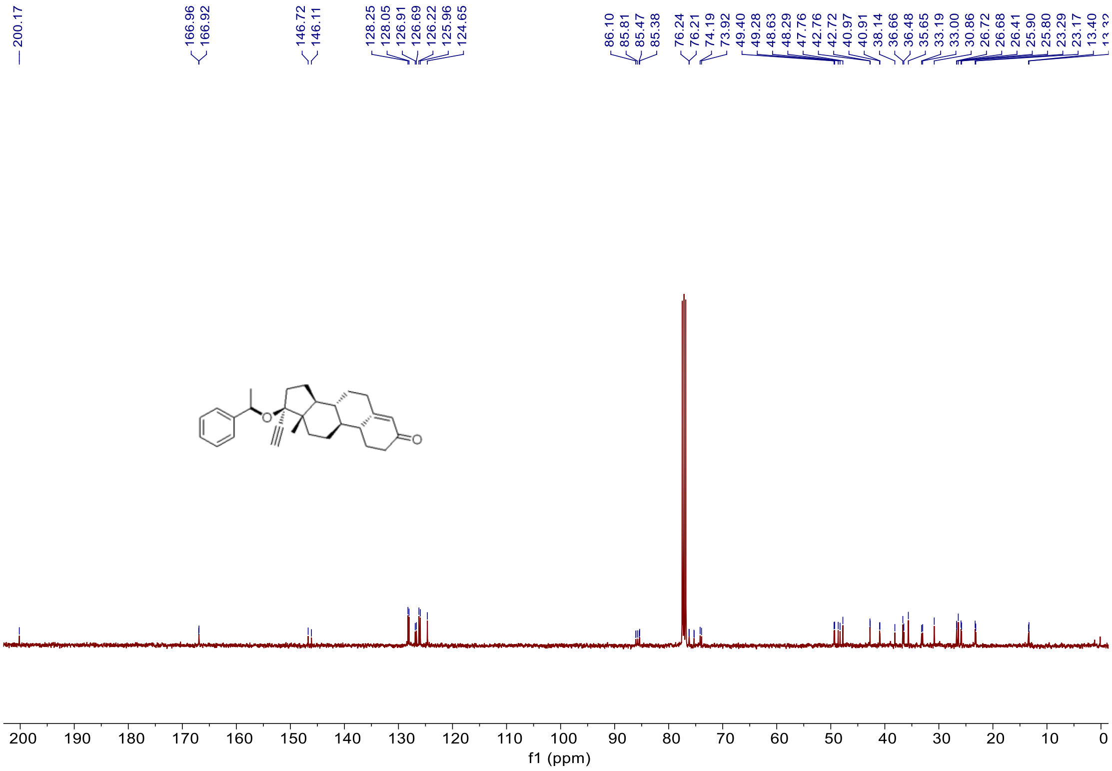
^{13}C NMR spectrum (101 MHz) of **70c** (see procedure)



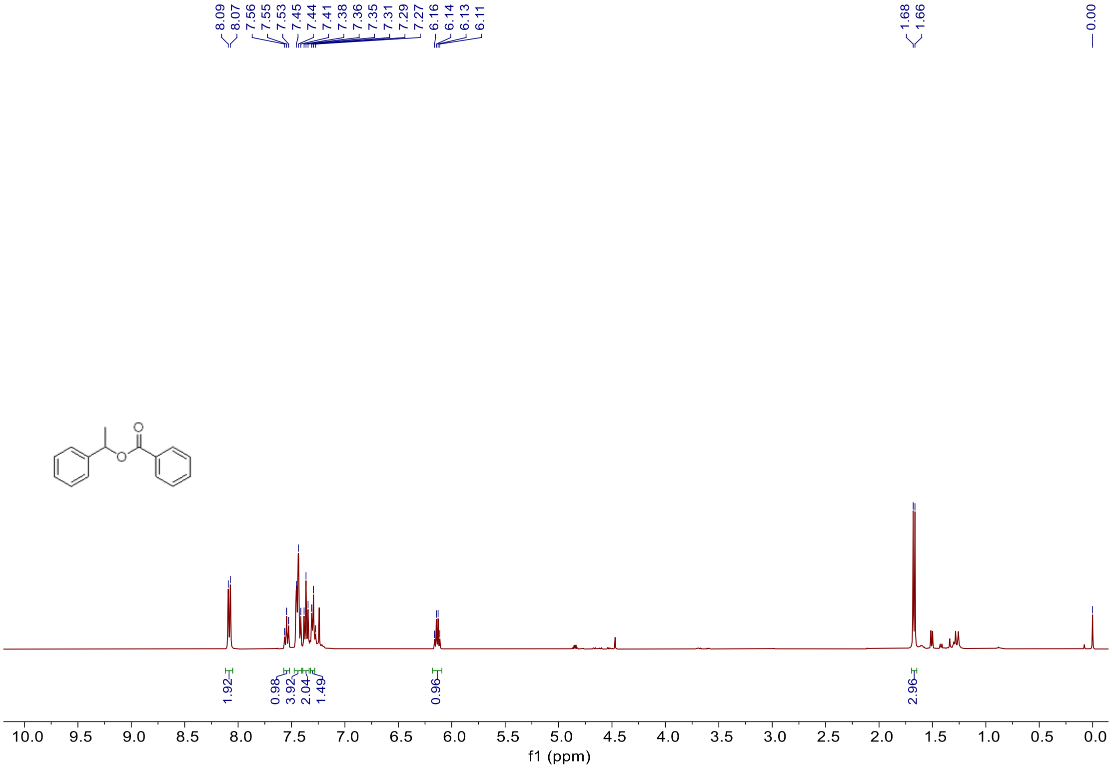
^{1}H NMR spectrum (400 MHz) of **71c** (see procedure)



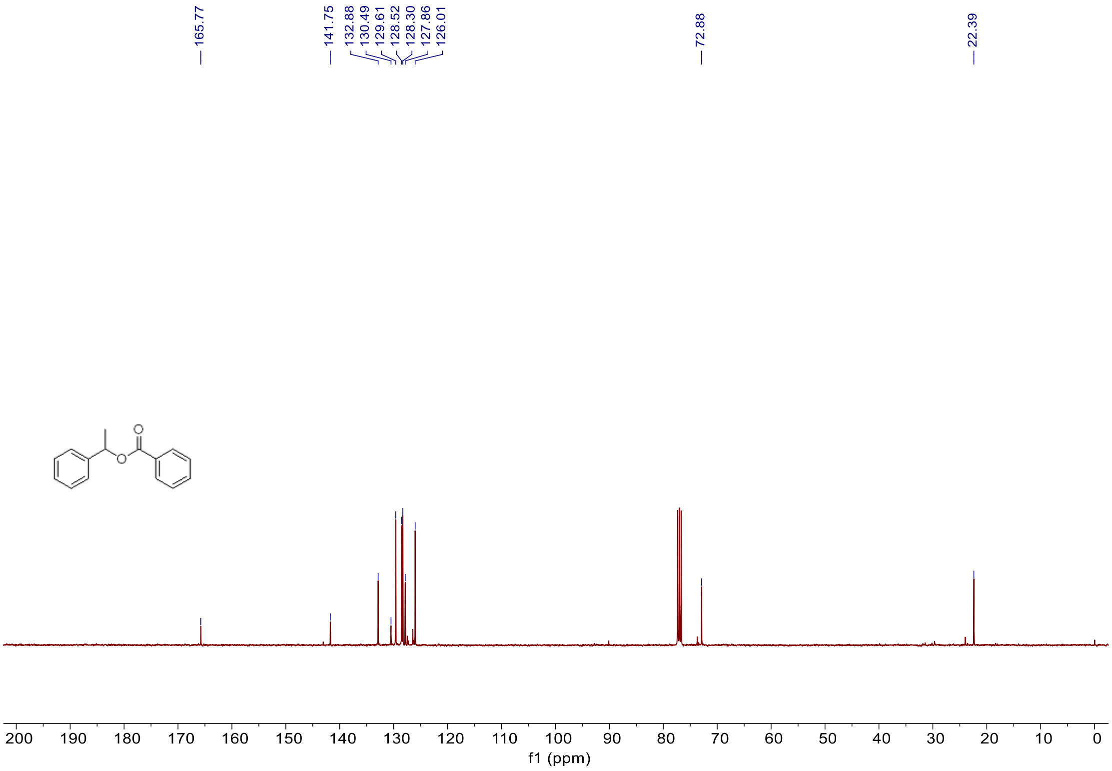
^{13}C NMR spectrum (101 MHz) of **71c** (see procedure)



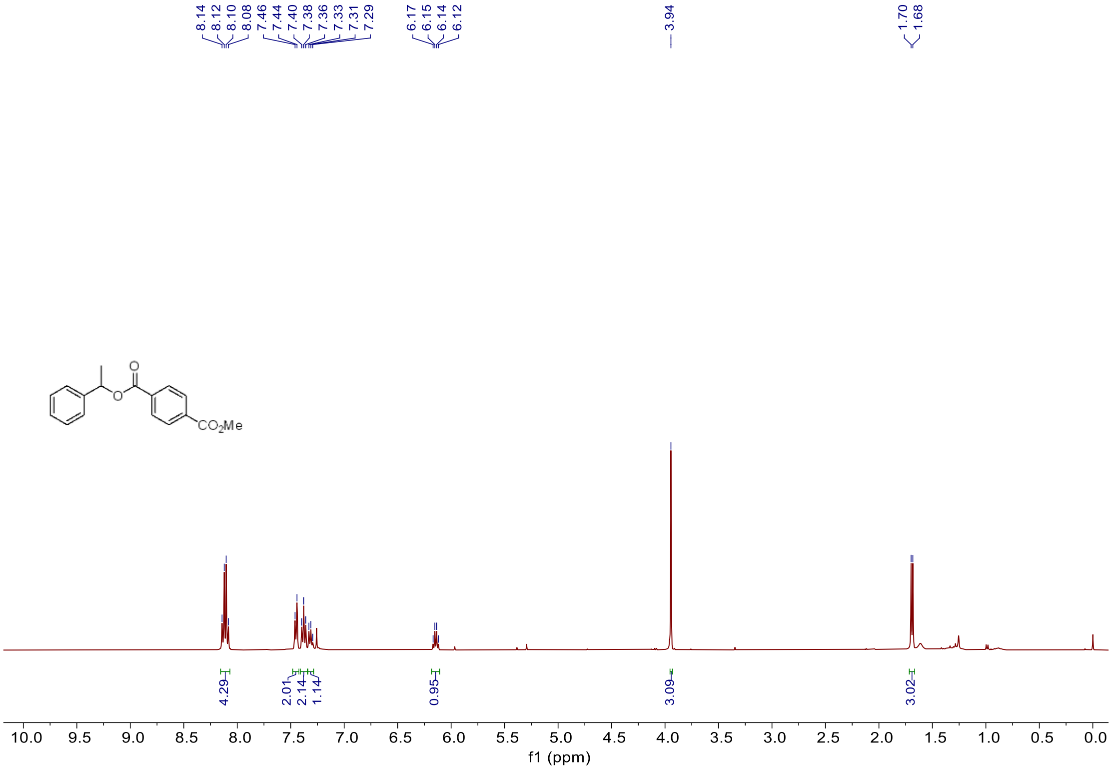
^{1}H NMR spectrum (400 MHz) of **72c** (see procedure)



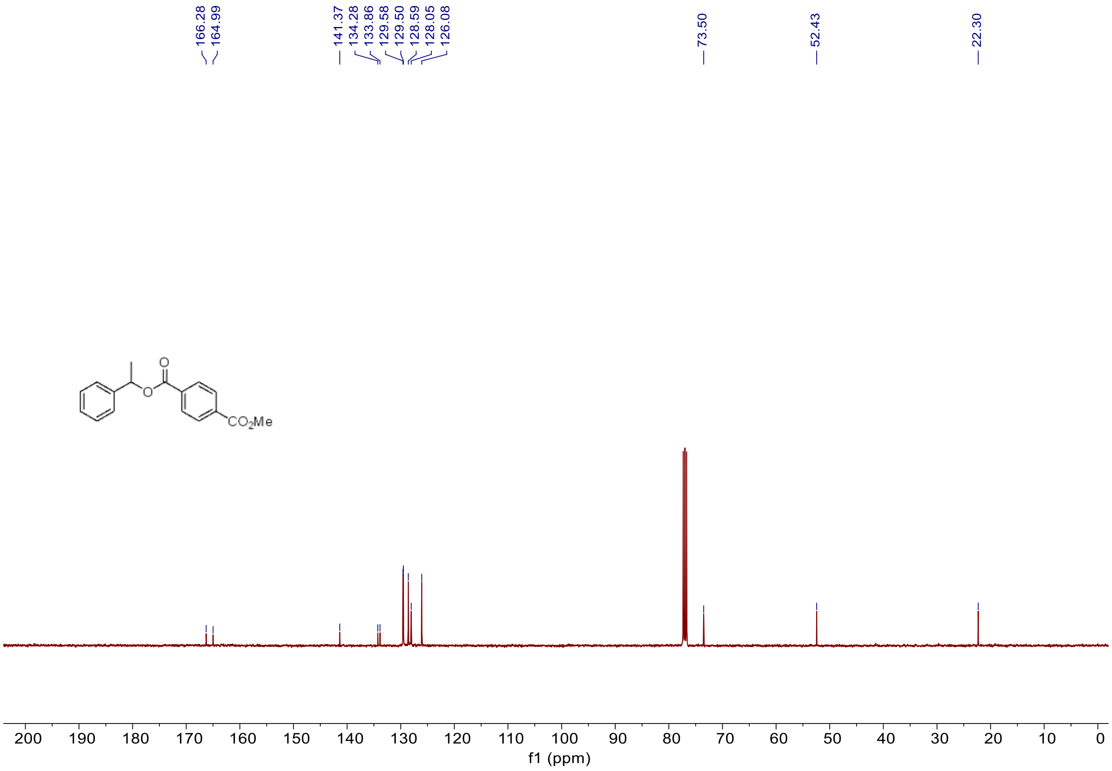
^{13}C NMR spectrum (101 MHz) of **72c** (see procedure)



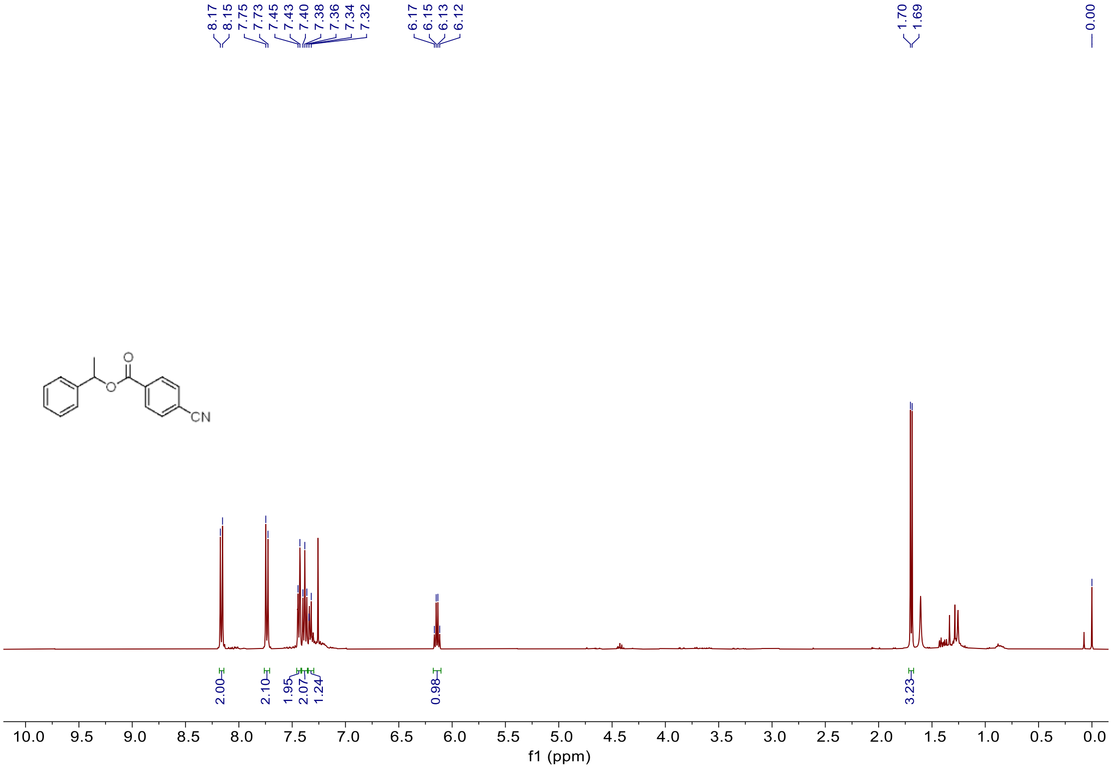
^{1}H NMR spectrum (400 MHz) of **73c** (see procedure)



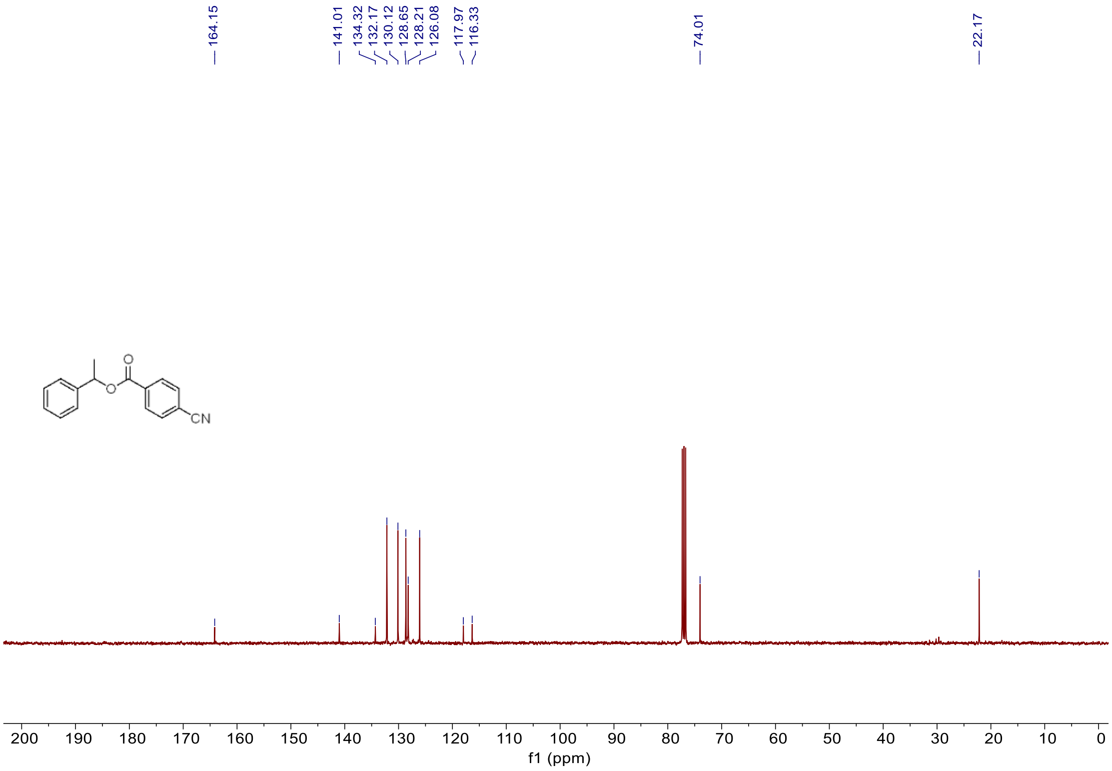
^{13}C NMR spectrum (101 MHz) of **73c** (see procedure)



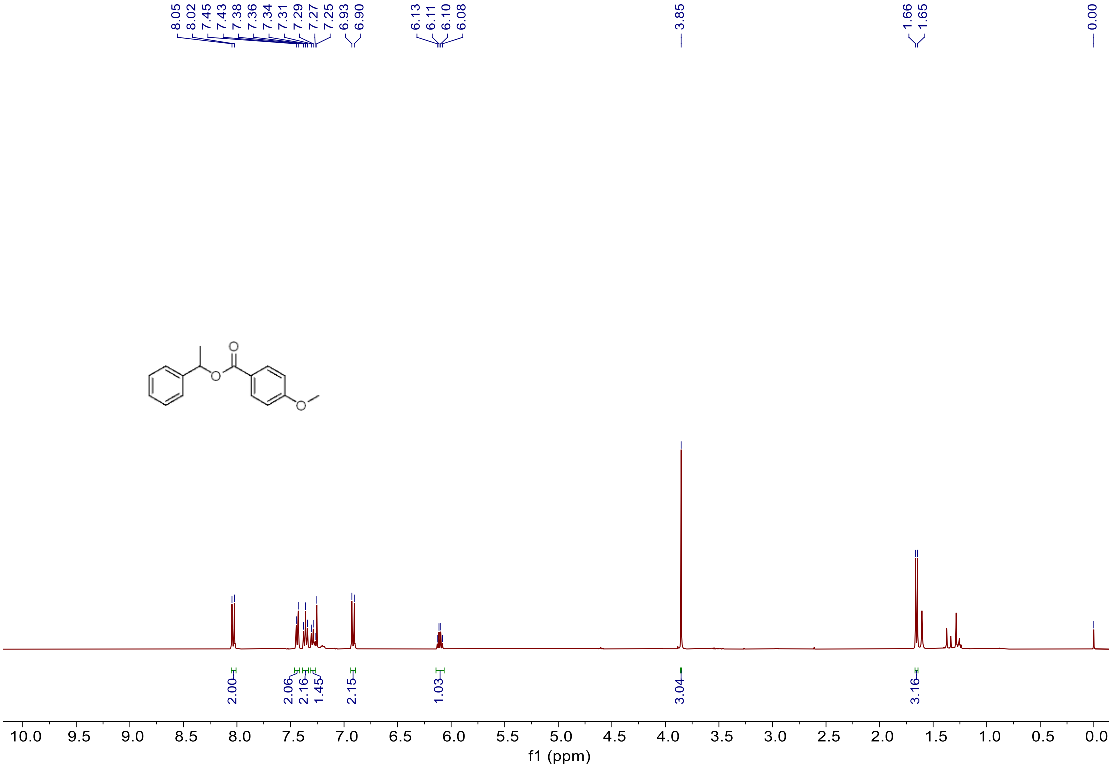
^{1}H NMR spectrum (400 MHz) of **74c** (see procedure)



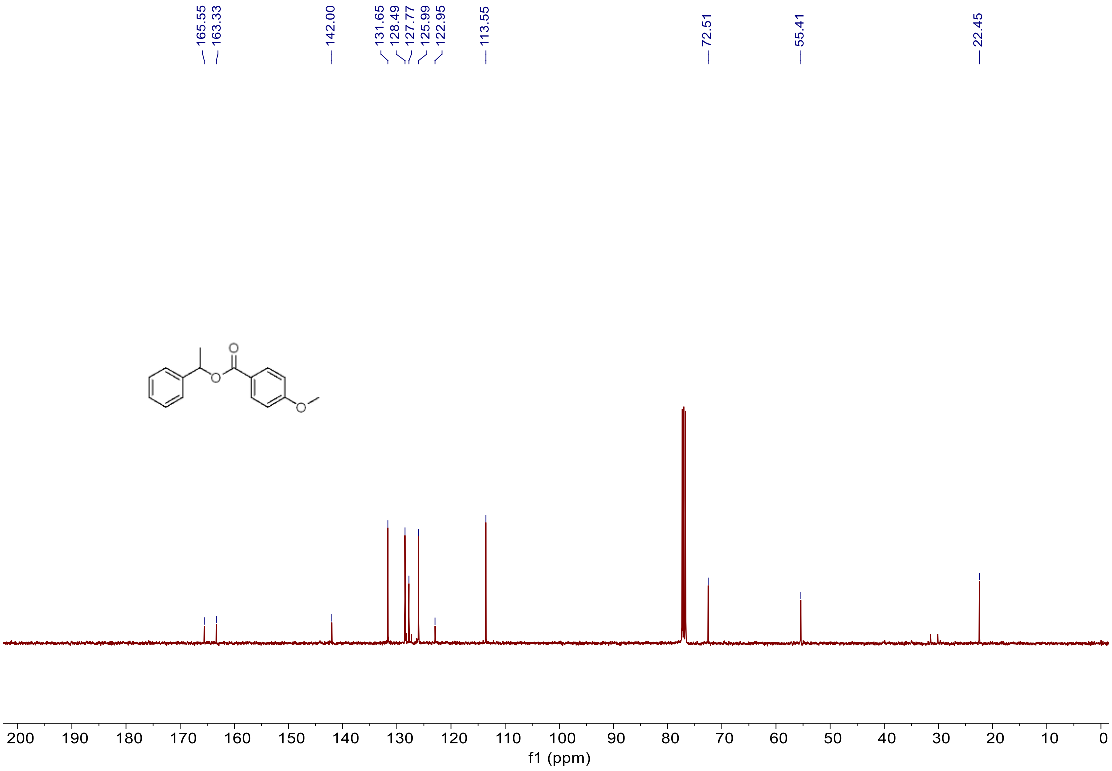
^{13}C NMR spectrum (101 MHz) of **74c** (see procedure)



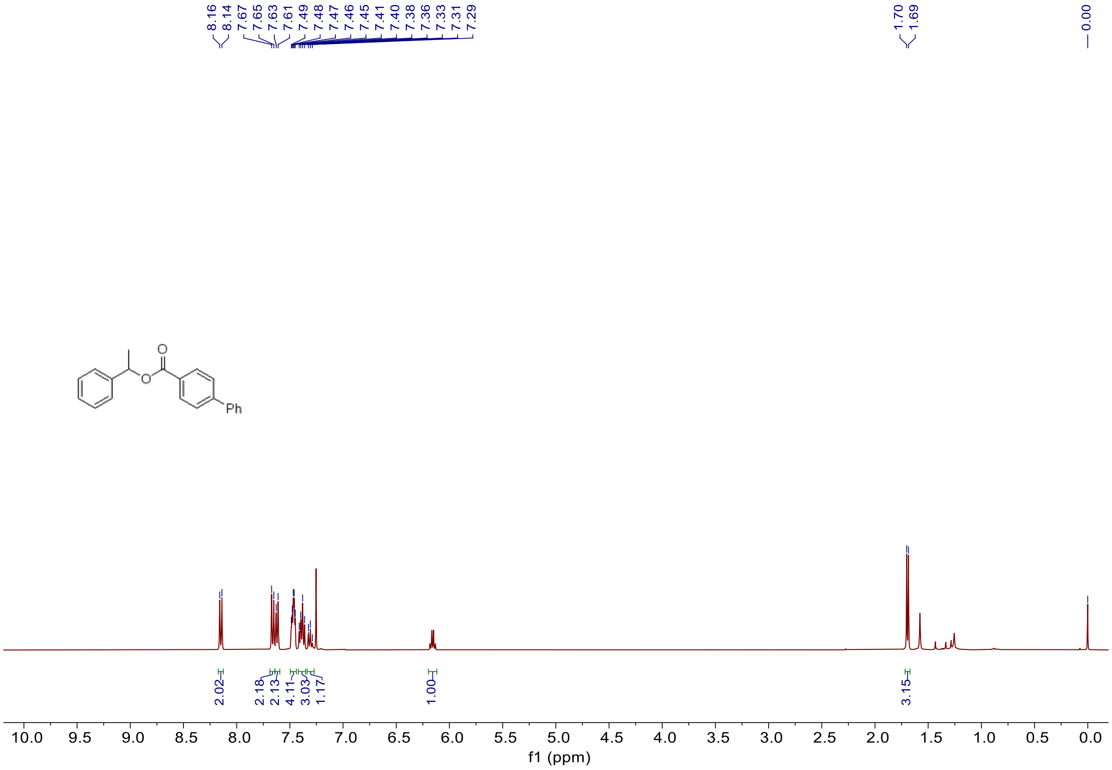
^{1}H NMR spectrum (400 MHz) of **75c** (see procedure)



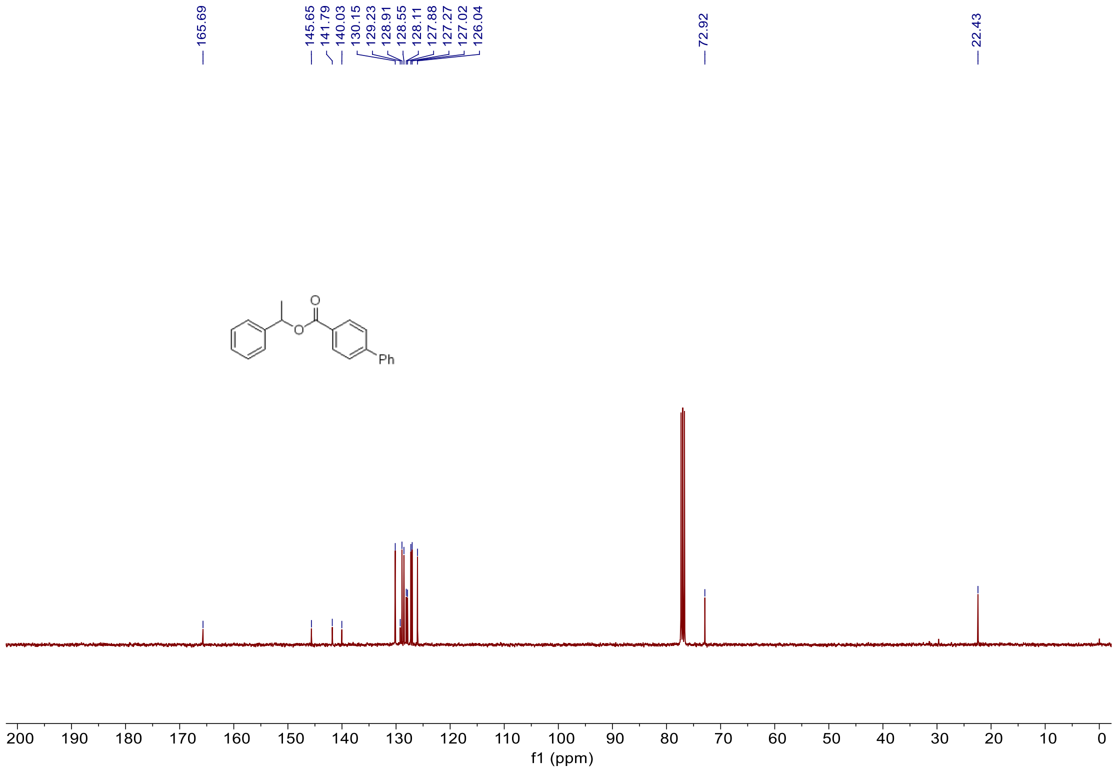
^{13}C NMR spectrum (101 MHz) of **75c** (see procedure)



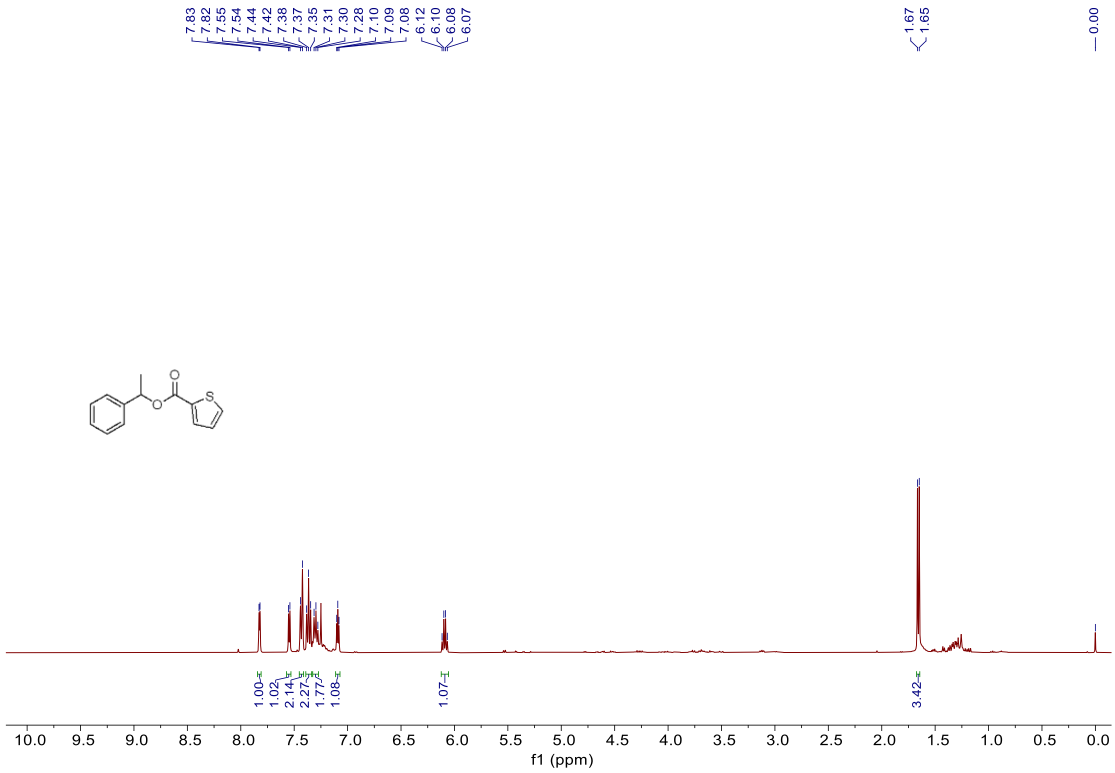
^{1}H NMR spectrum (400 MHz) of **76c** (see procedure)



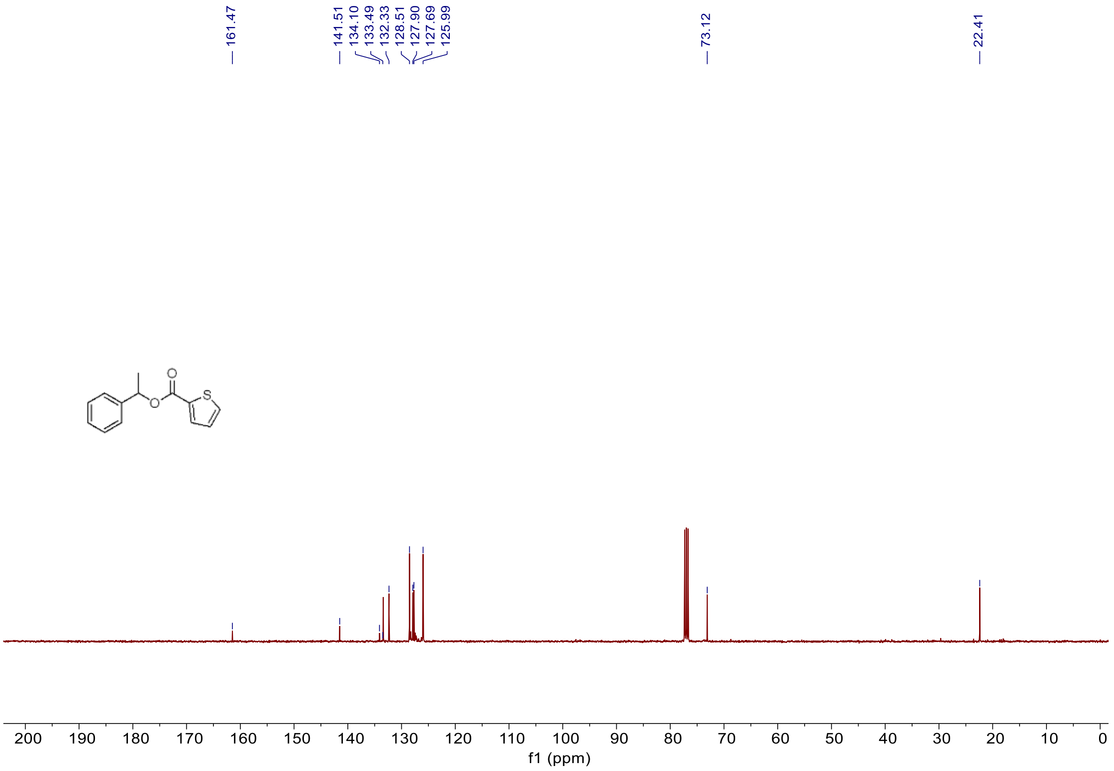
^{13}C NMR spectrum (101 MHz) of **76c** (see procedure)



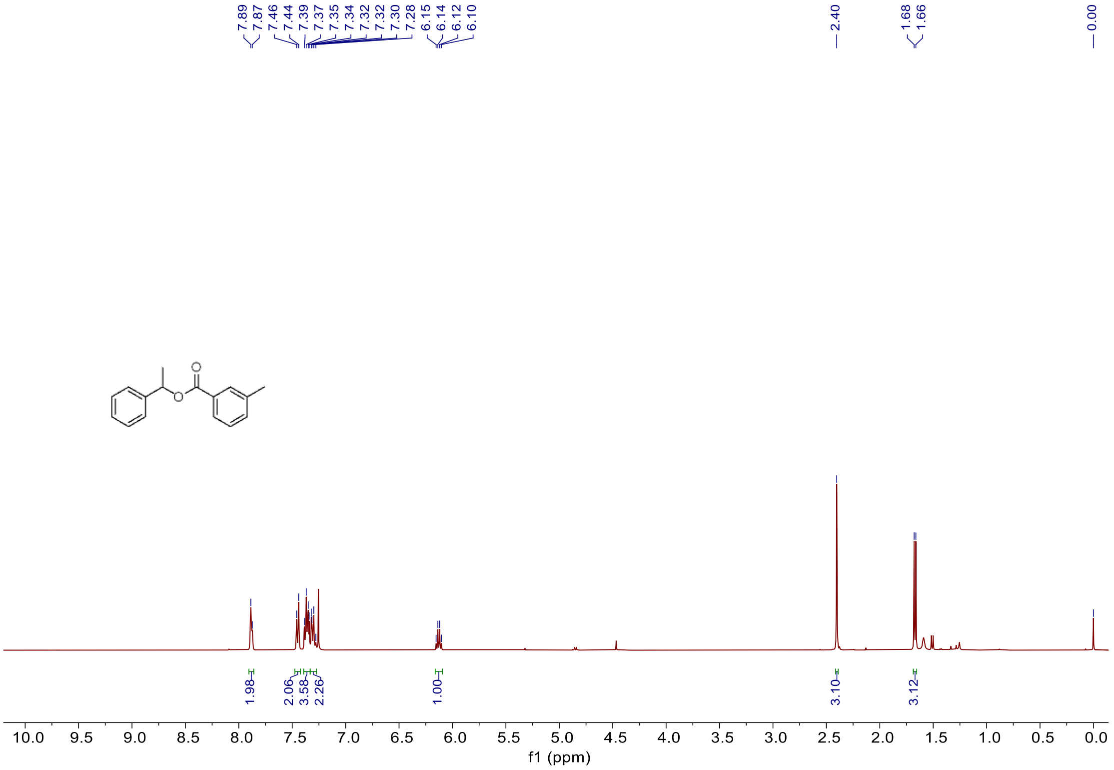
^{1}H NMR spectrum (400 MHz) of **77c** (see procedure)



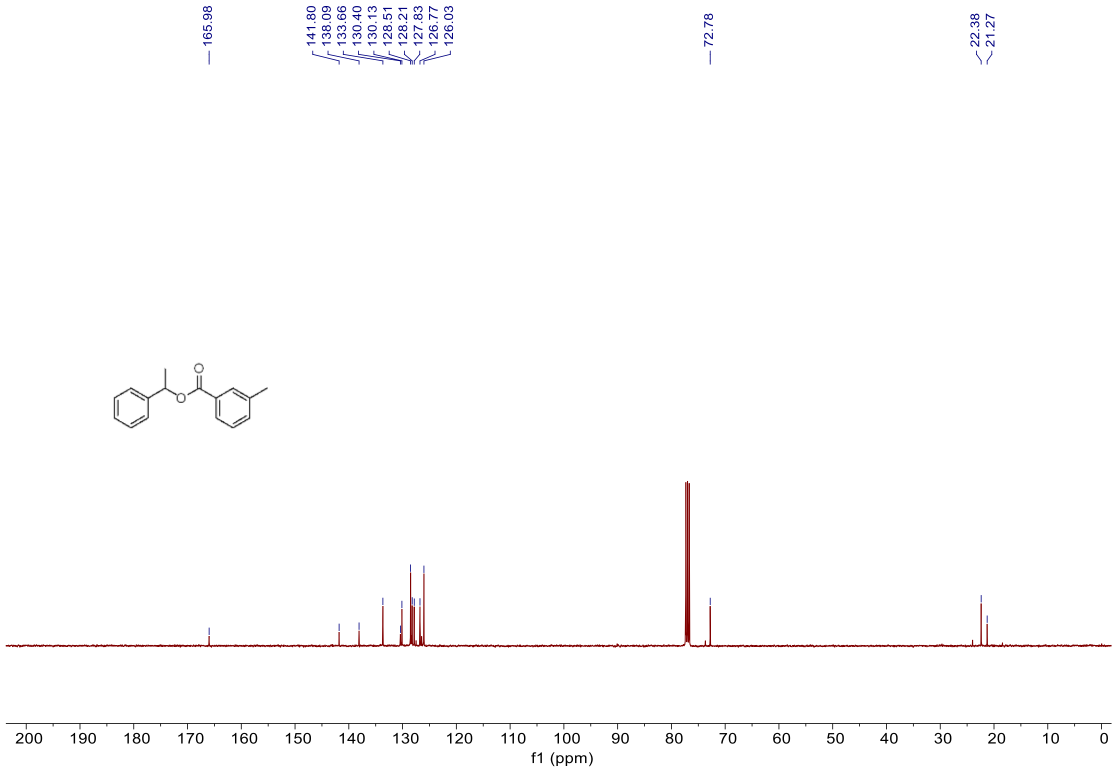
^{13}C NMR spectrum (101 MHz) of **77c** (see procedure)



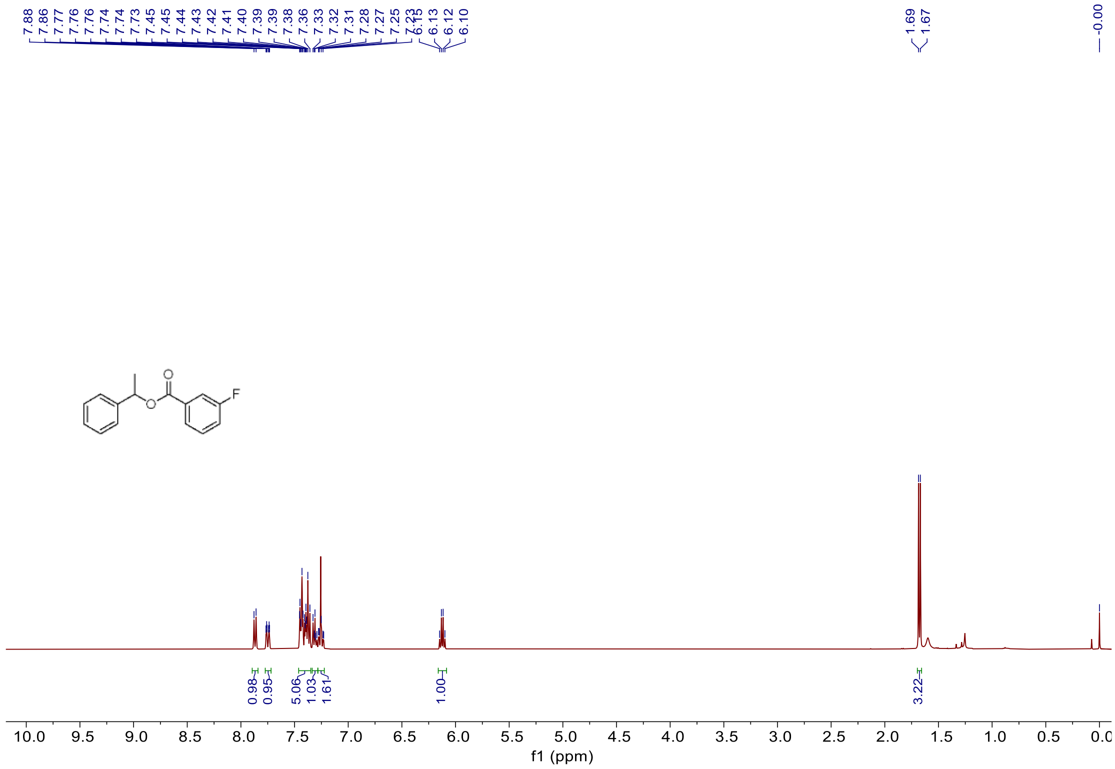
^{1}H NMR spectrum (400 MHz) of **78c** (see procedure)



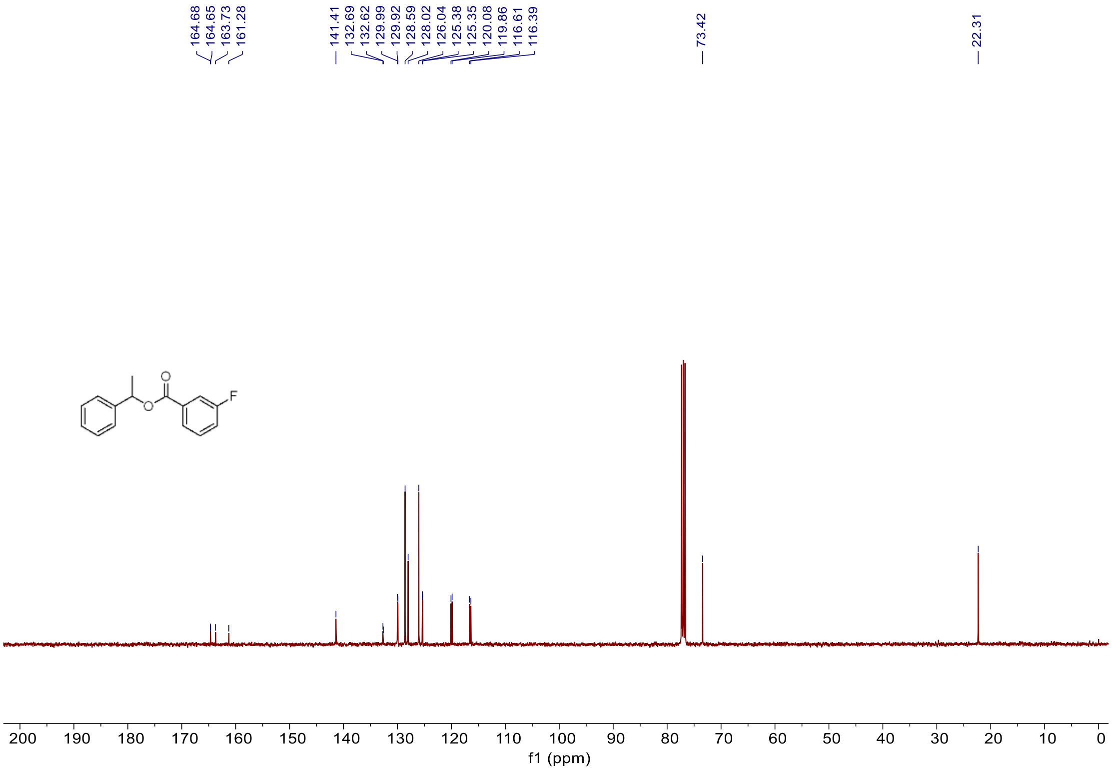
^{13}C NMR spectrum (101 MHz) of **78c** (see procedure)



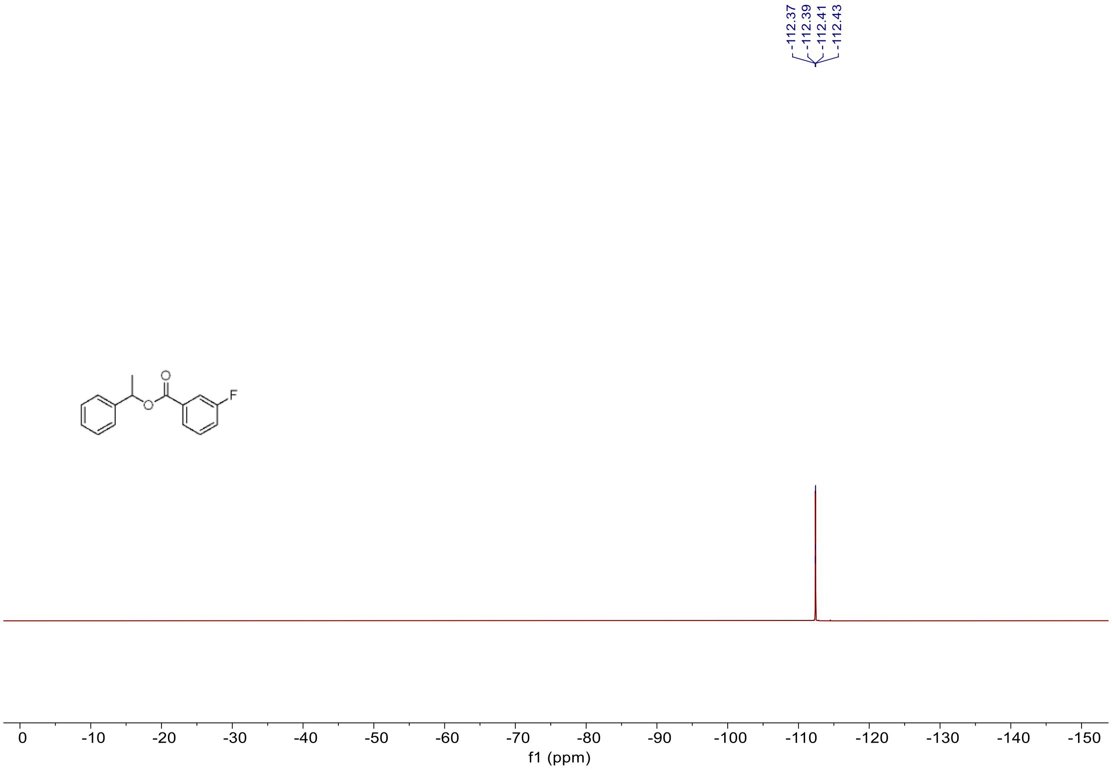
^{1}H NMR spectrum (400 MHz) of **79c** (see procedure)



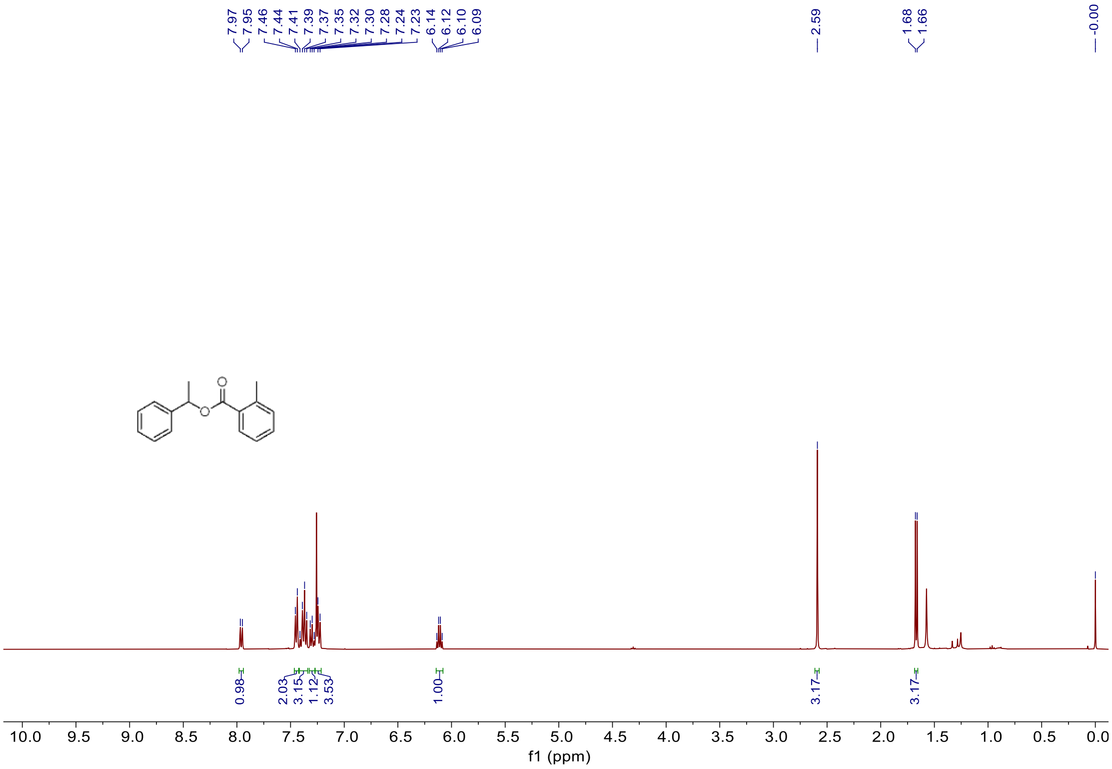
^{13}C NMR spectrum (101 MHz) of **79c** (see procedure)



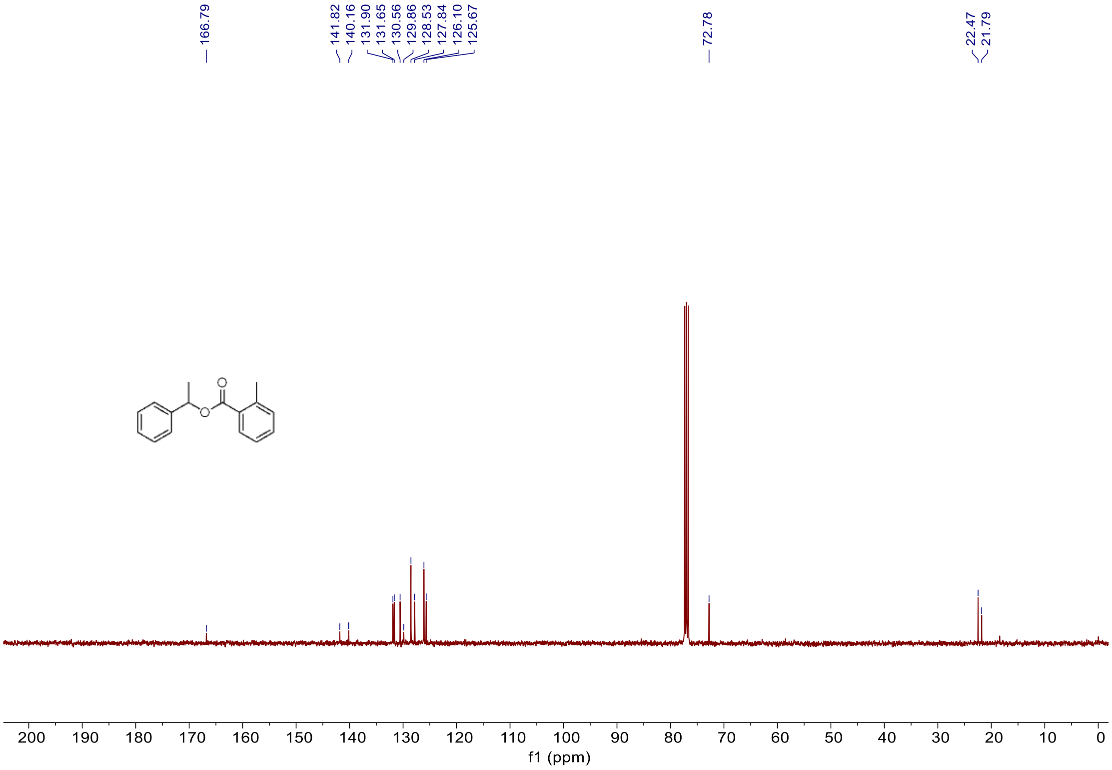
^{19}F NMR spectrum (376 MHz) of **79c** (see procedure)



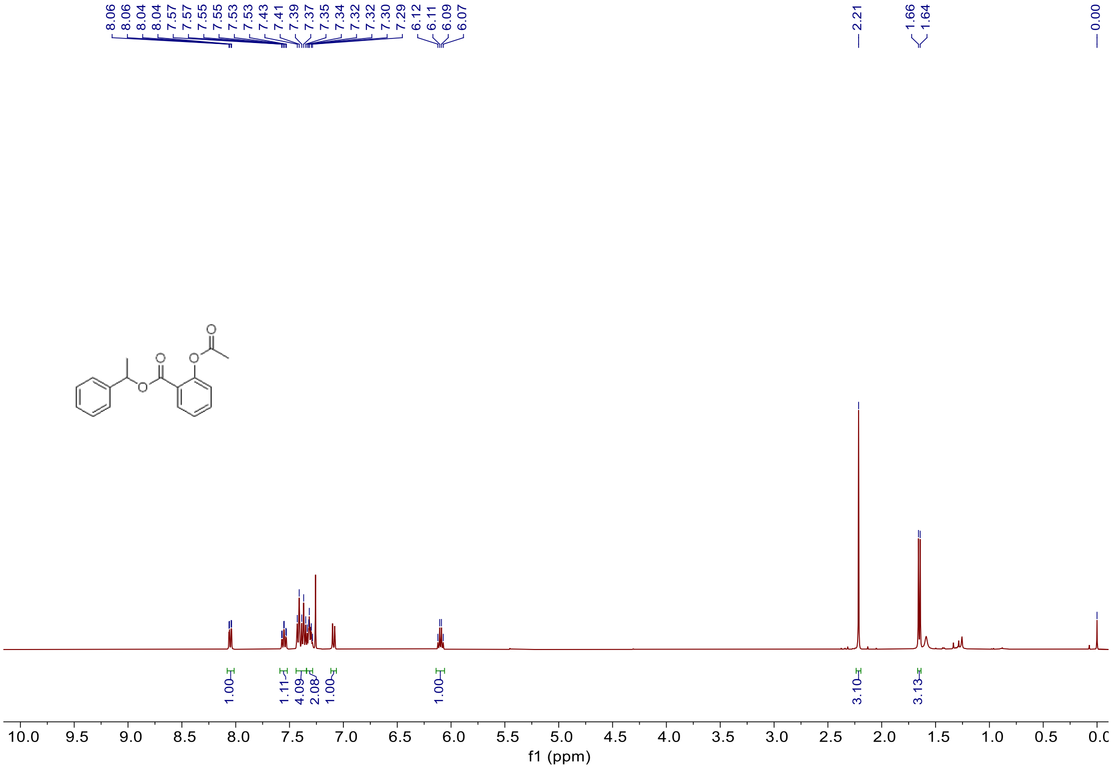
^{1}H NMR spectrum (400 MHz) of **80c** (see procedure)



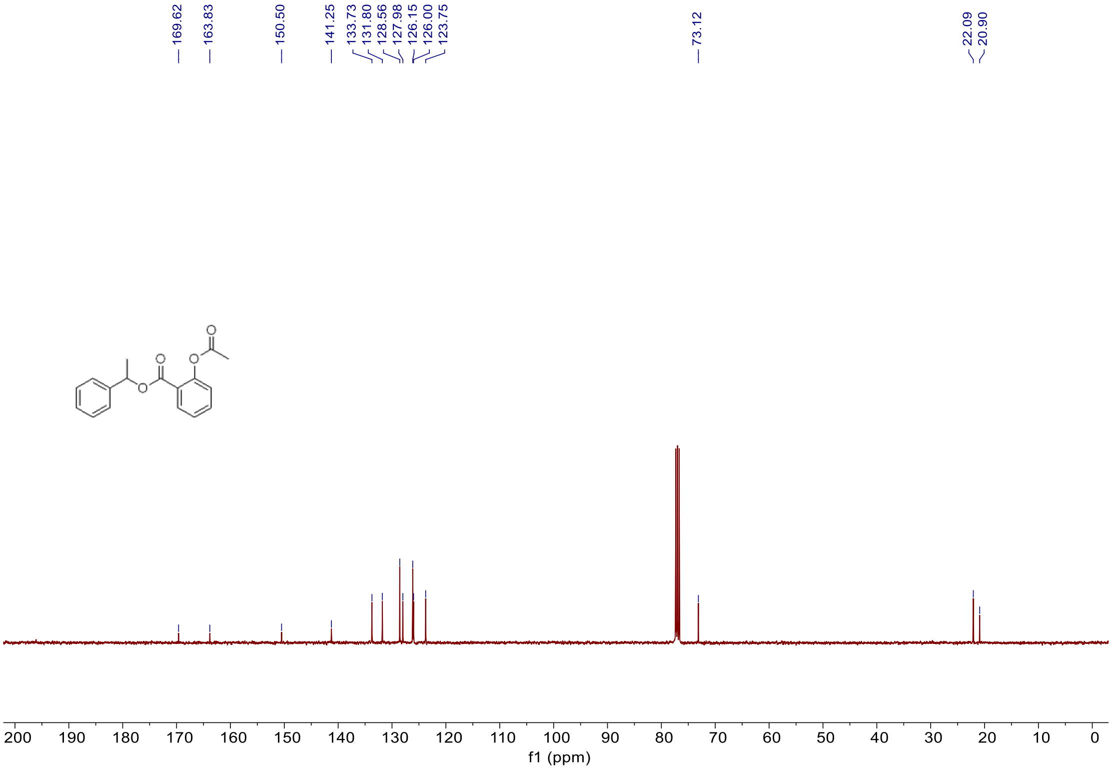
^{13}C NMR spectrum (101 MHz) of **80c** (see procedure)



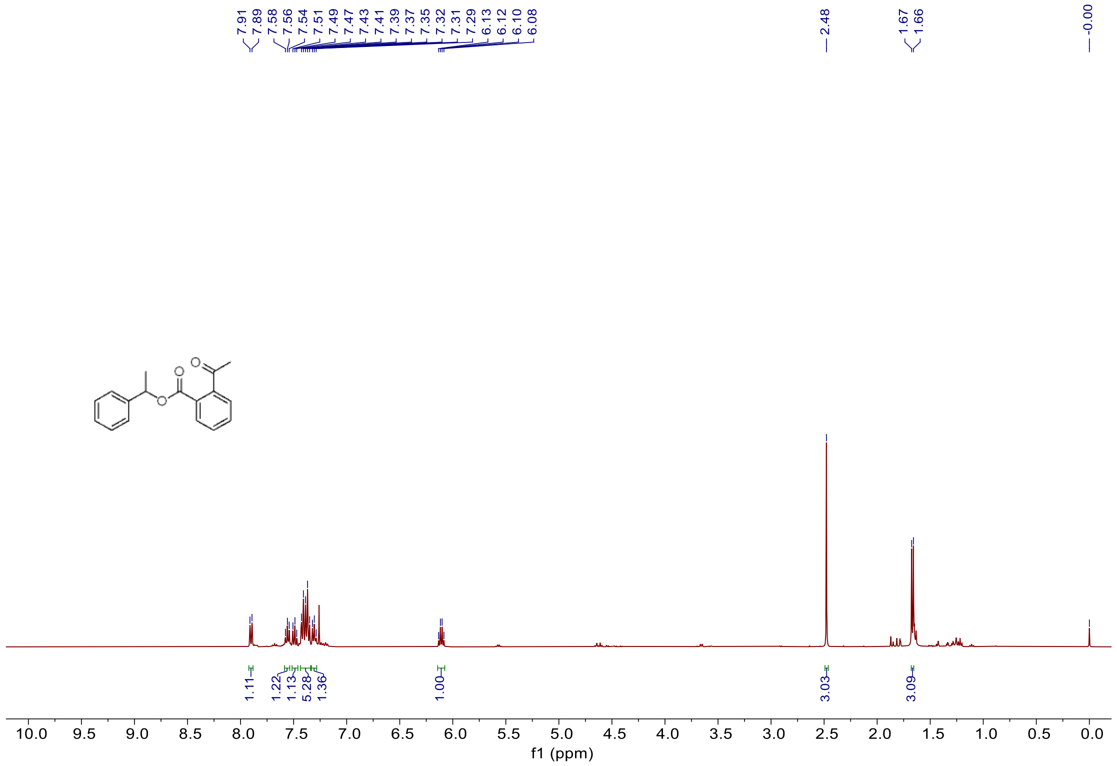
^{1}H NMR spectrum (400 MHz) of **81c** (see procedure)



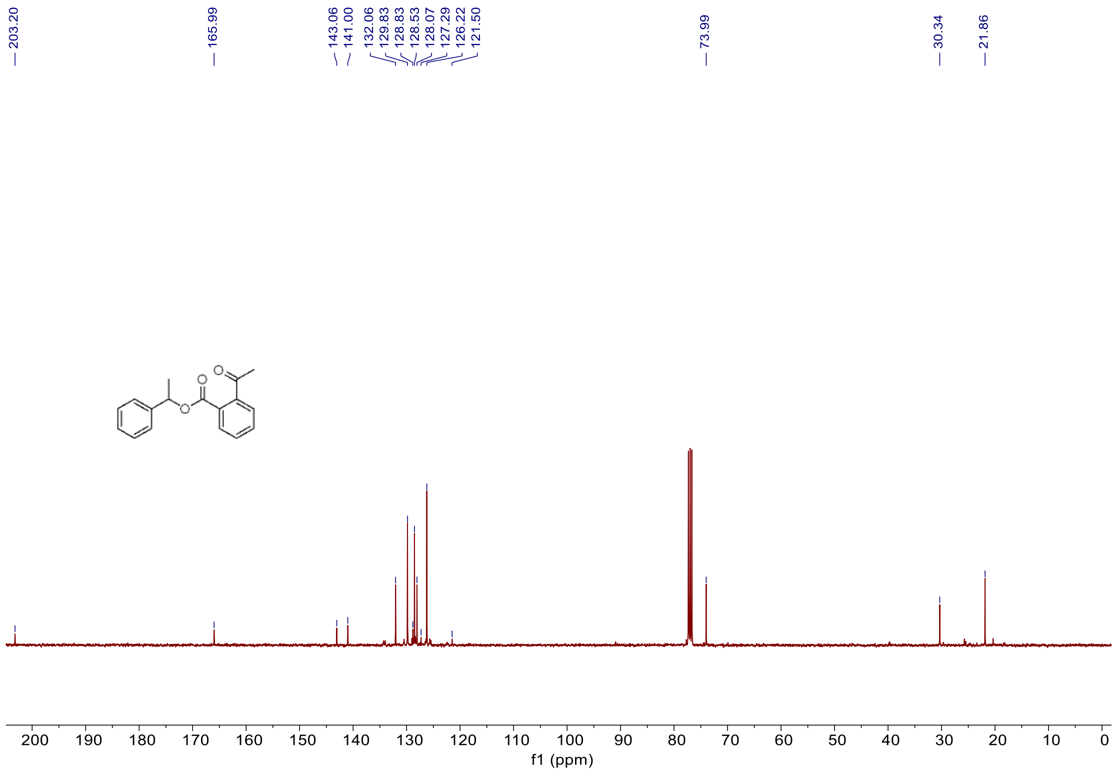
^{13}C NMR spectrum (101 MHz) of **81c** (see procedure)



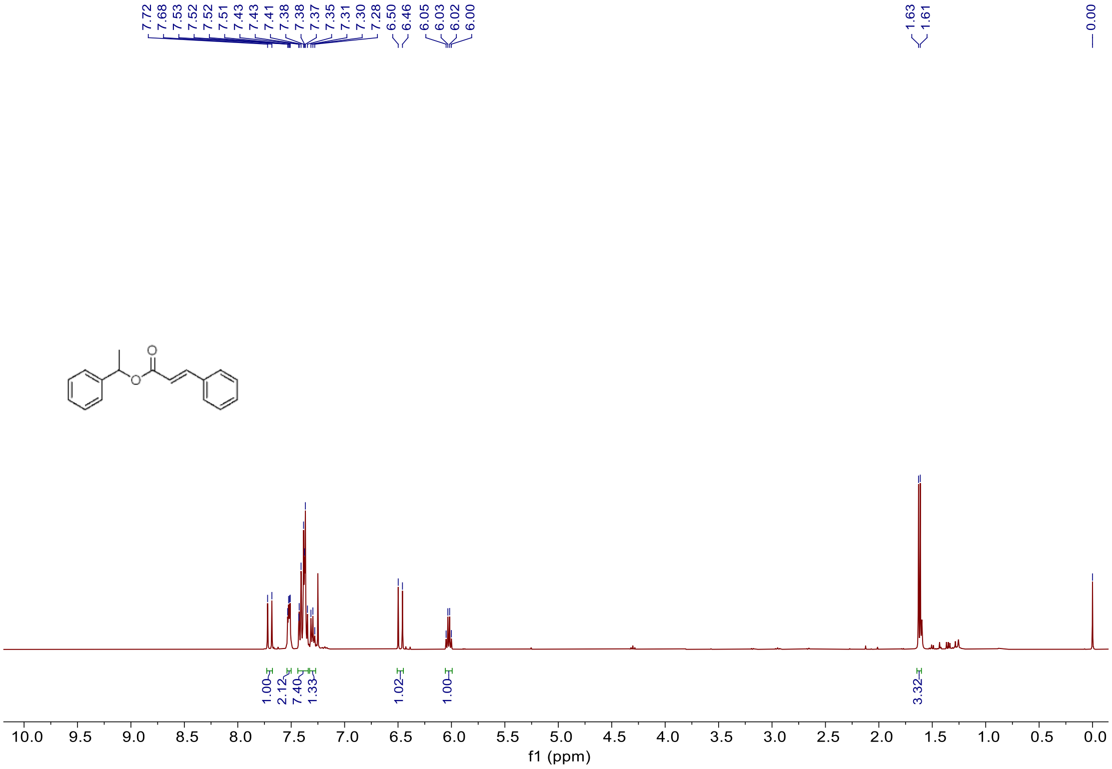
^{1}H NMR spectrum (400 MHz) of **82c** (see procedure)



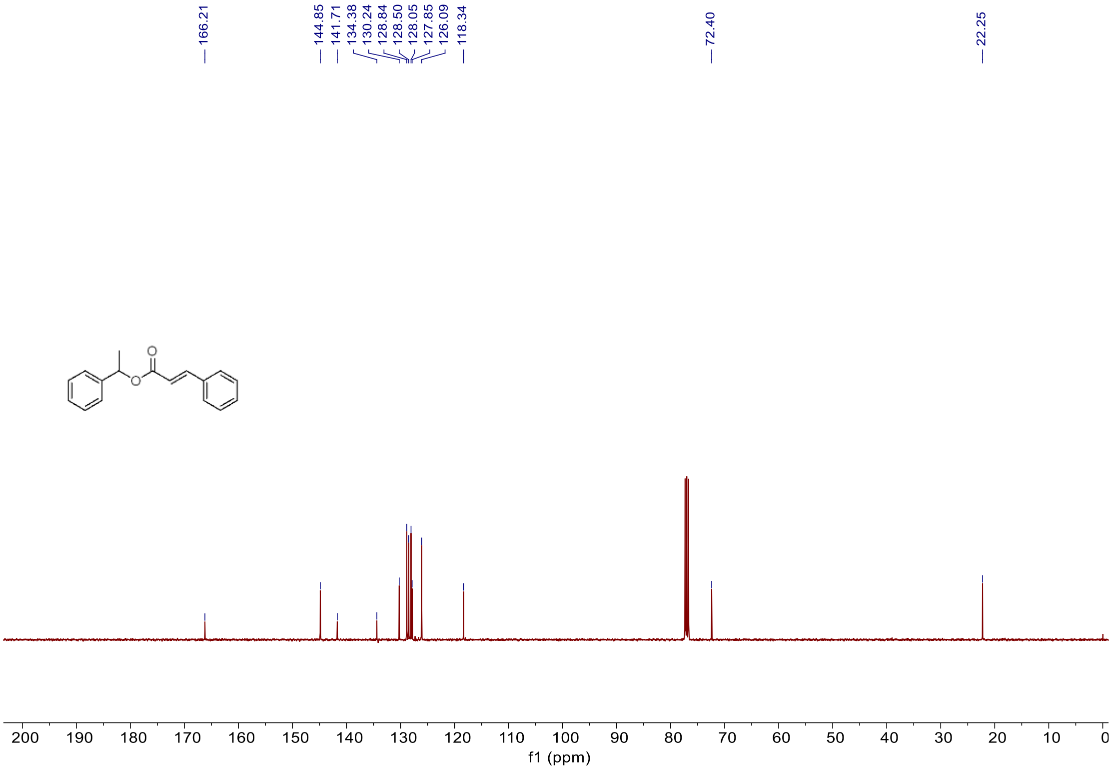
^{13}C NMR spectrum (101 MHz) of **82c** (see procedure)



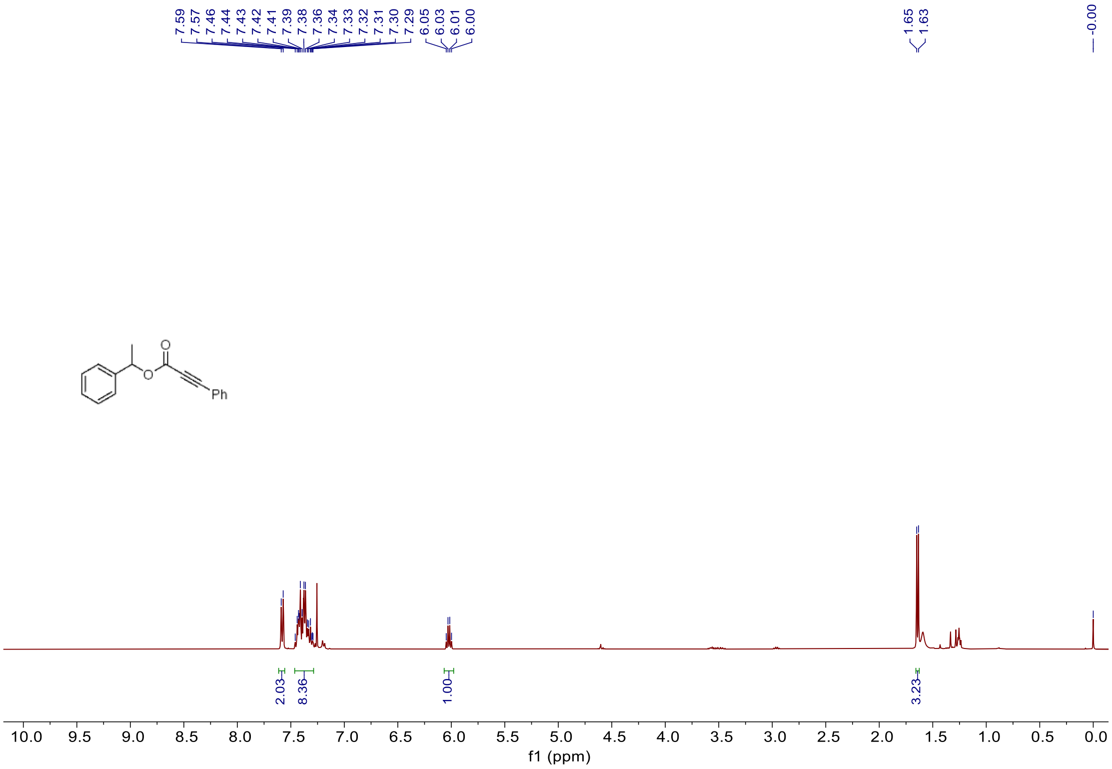
^{1}H NMR spectrum (400 MHz) of **83c** (see procedure)



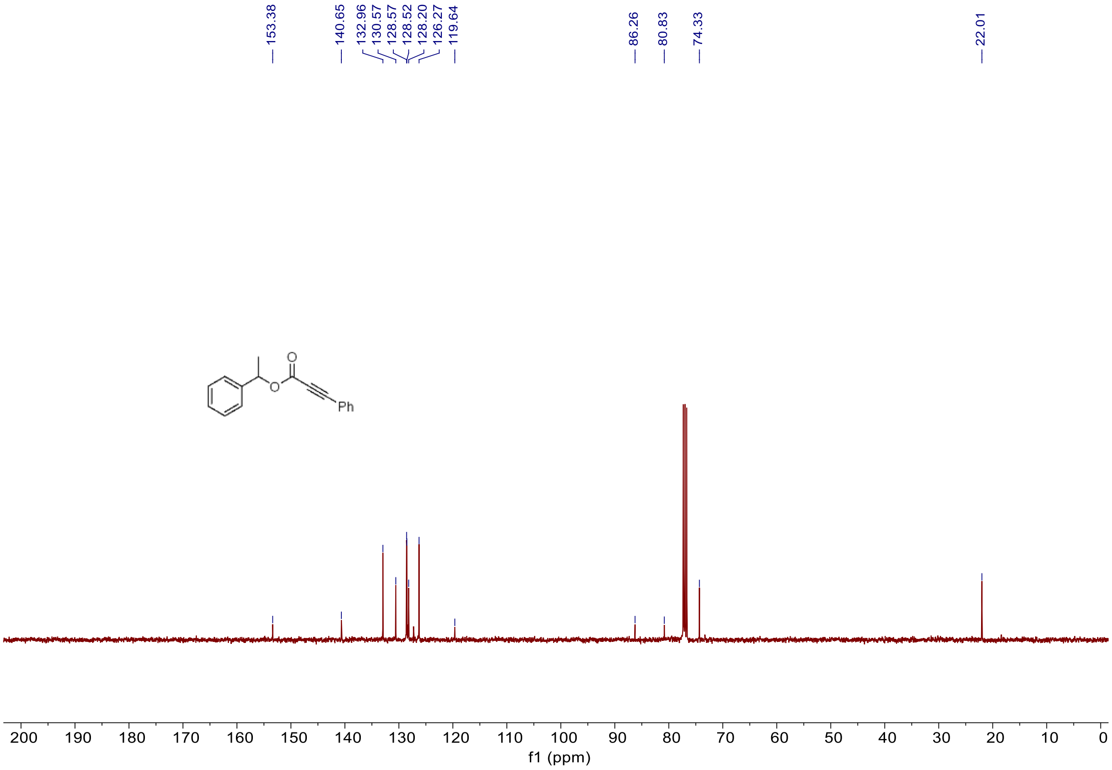
^{13}C NMR spectrum (101 MHz) of **83c** (see procedure)



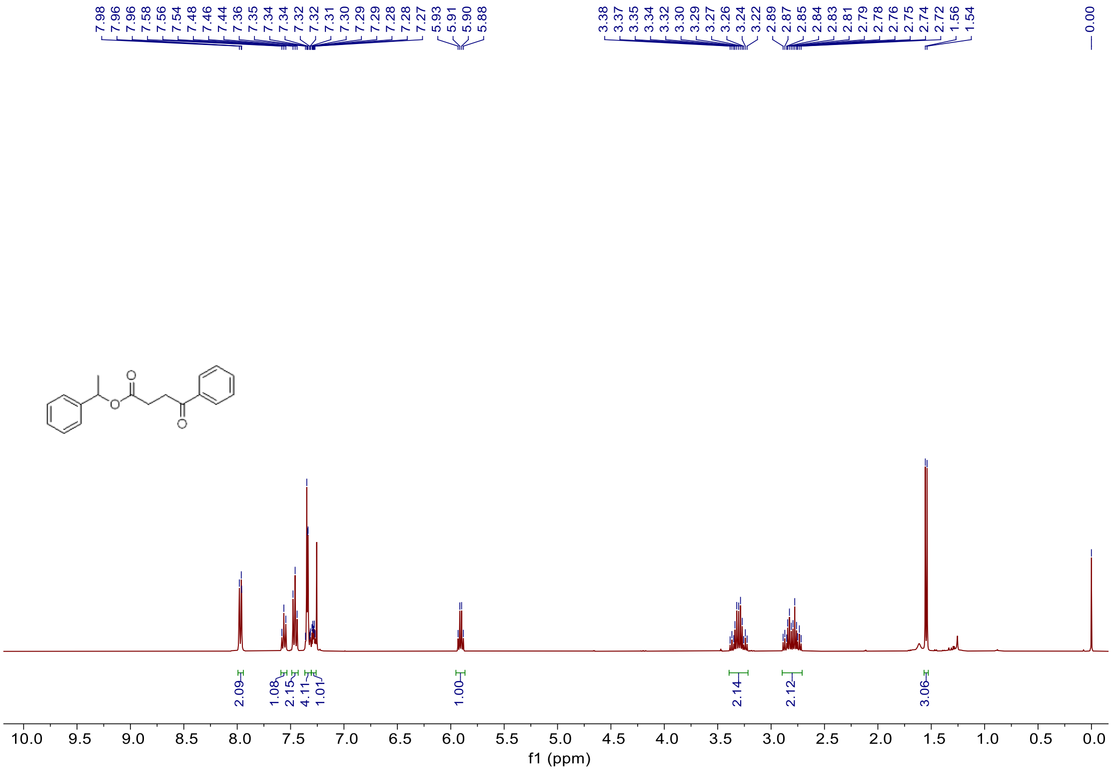
^{1}H NMR spectrum (400 MHz) of **84c** (see procedure)



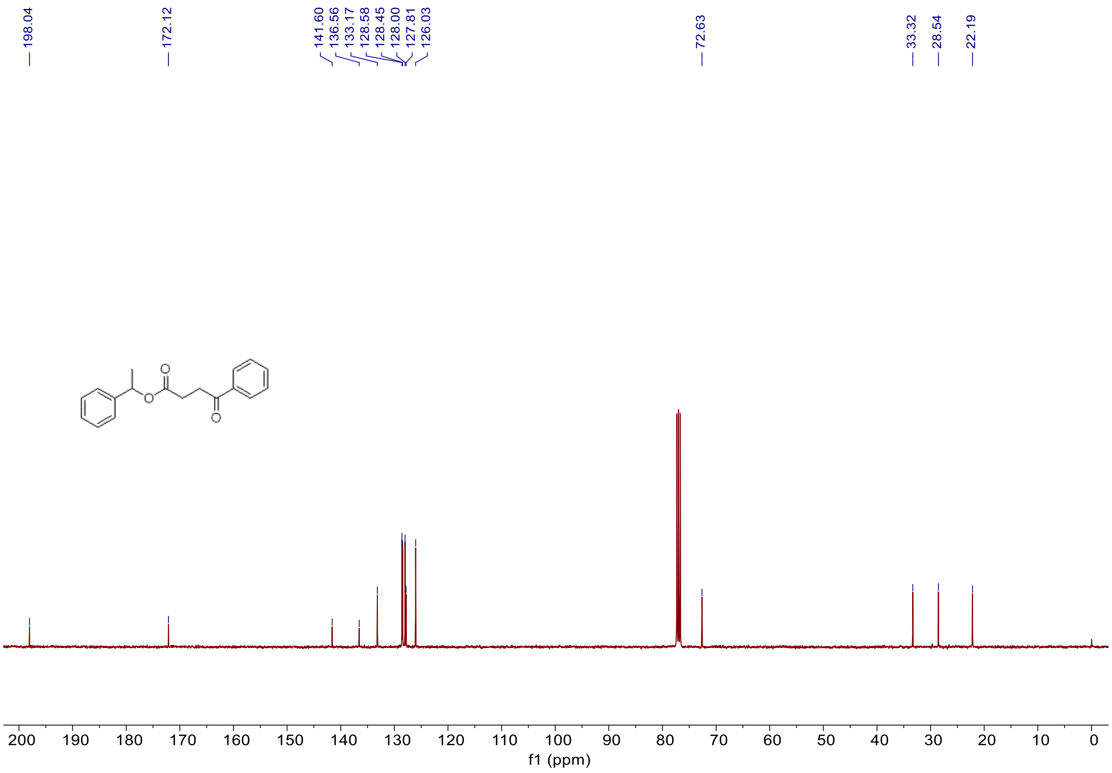
^{13}C NMR spectrum (101 MHz) of **84c** (see procedure)



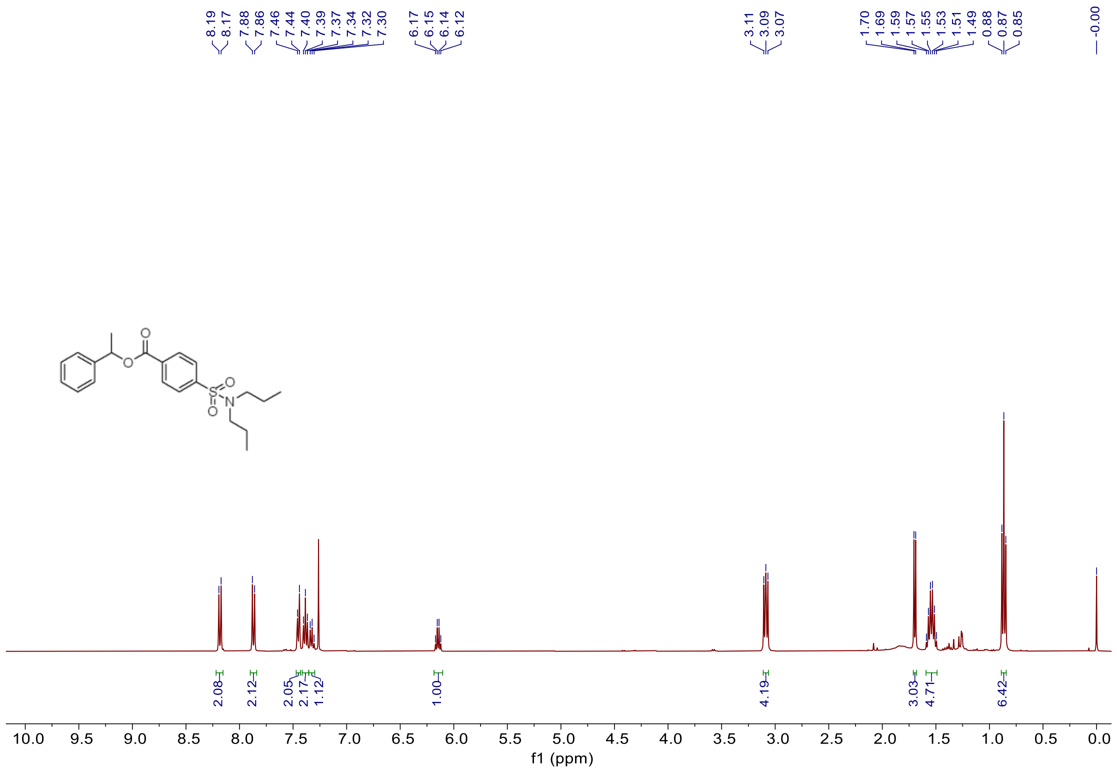
^{1}H NMR spectrum (400 MHz) of **85c** (see procedure)



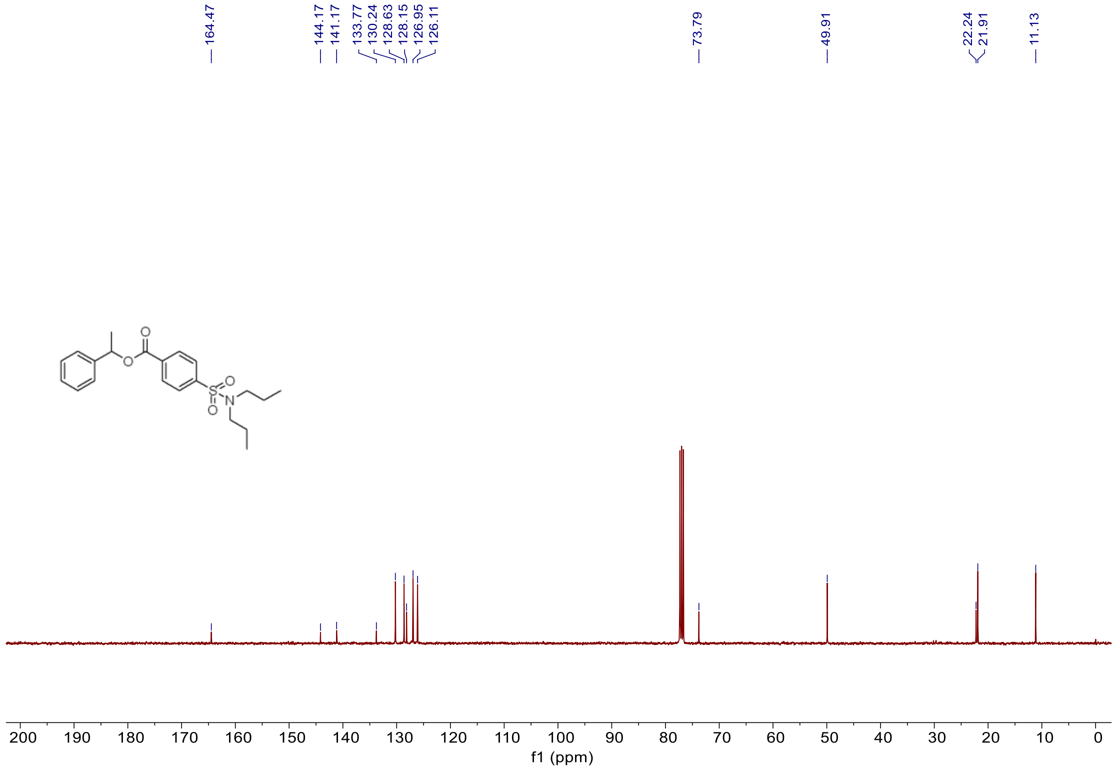
^{13}C NMR spectrum (101 MHz) of **85c** (see procedure)



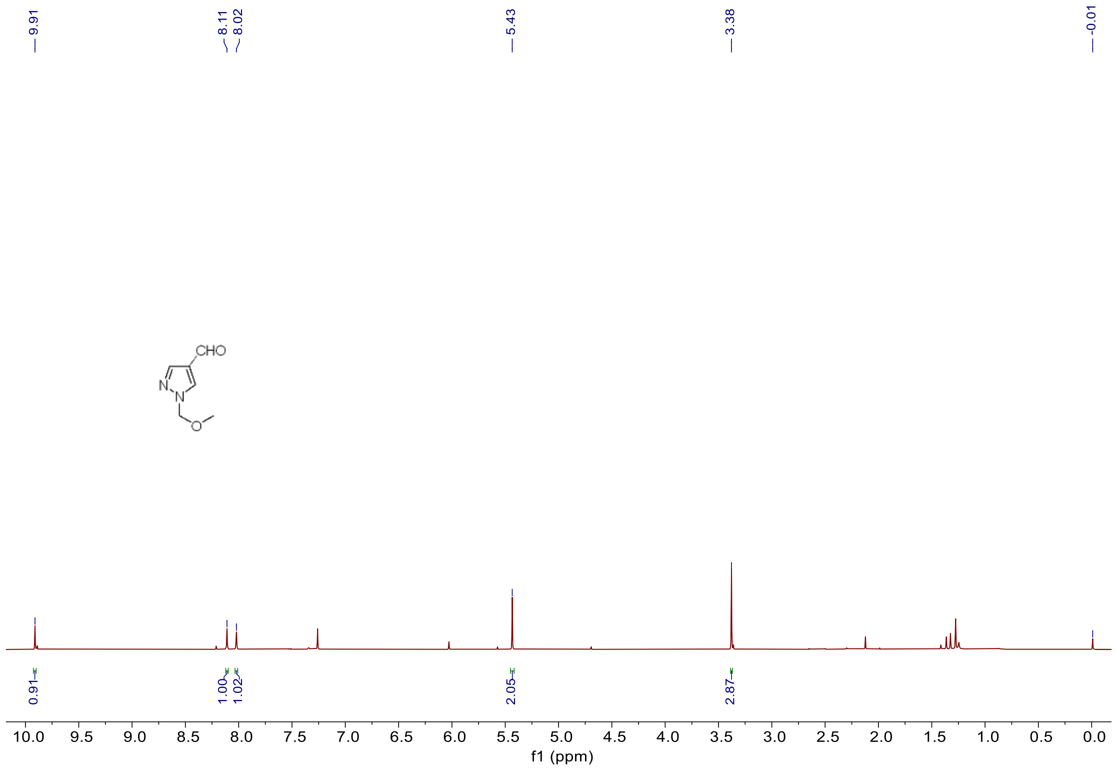
^{1}H NMR spectrum (400 MHz) of **86c** (see procedure)



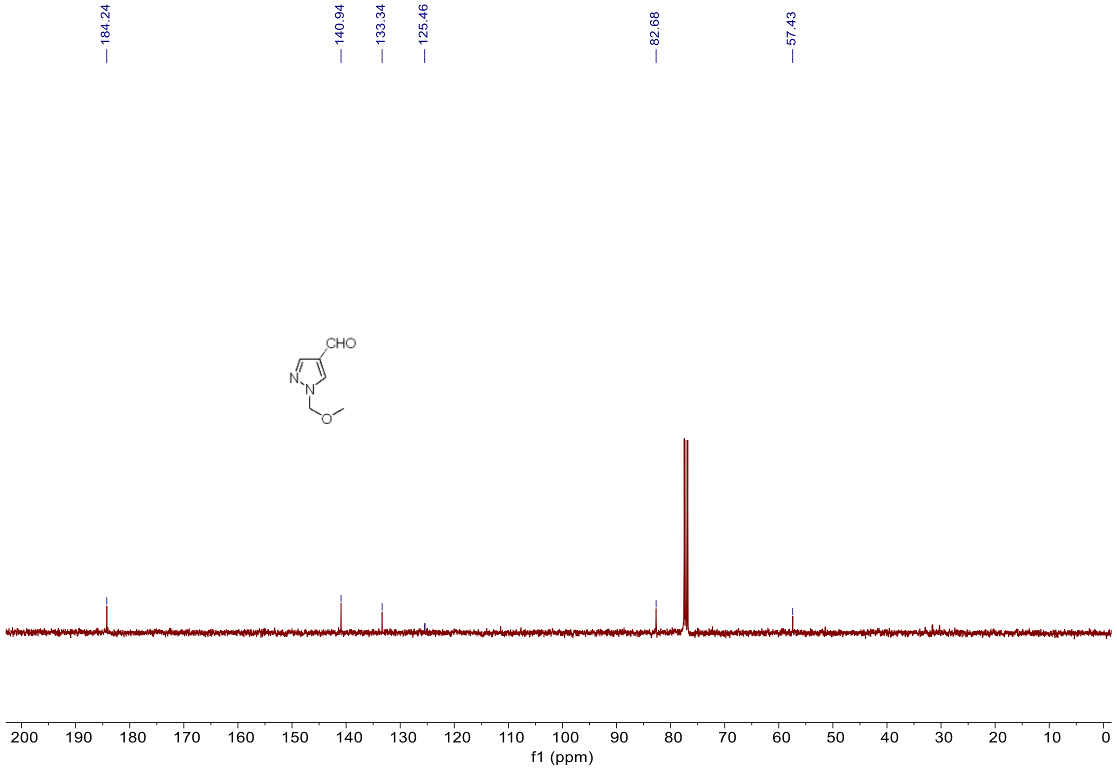
^{13}C NMR spectrum (101 MHz) of **86c** (see procedure)



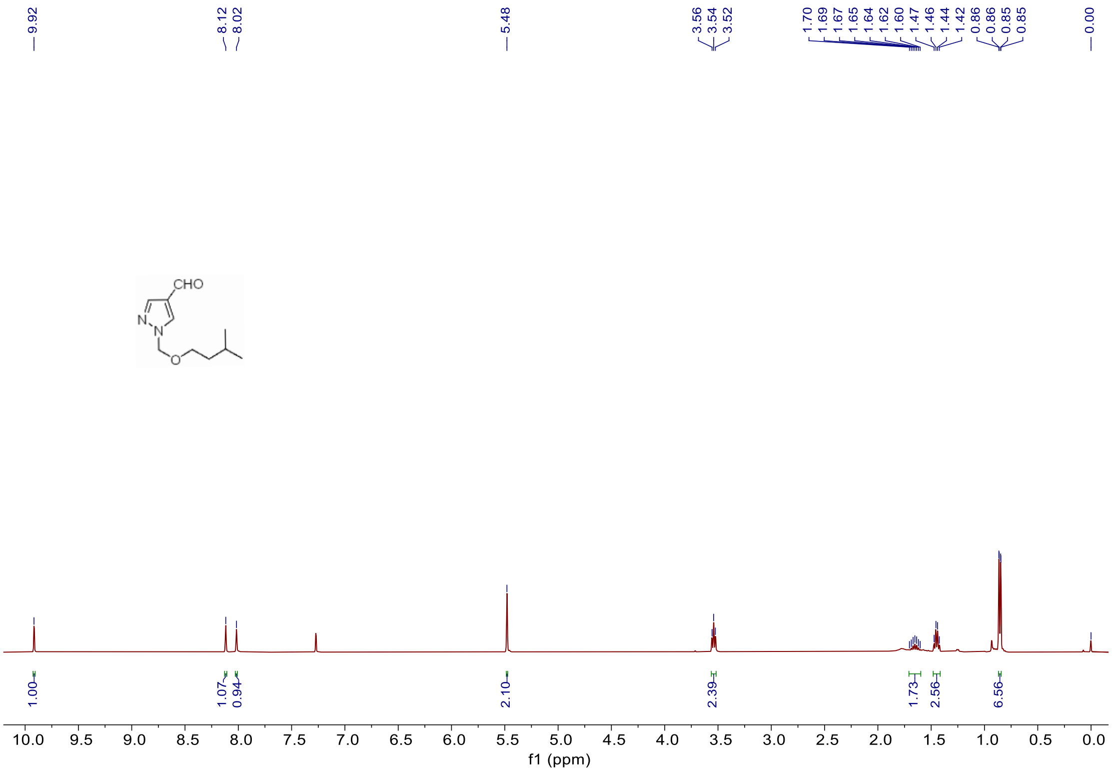
^{1}H NMR spectrum (400 MHz) of **2g** (see procedure)



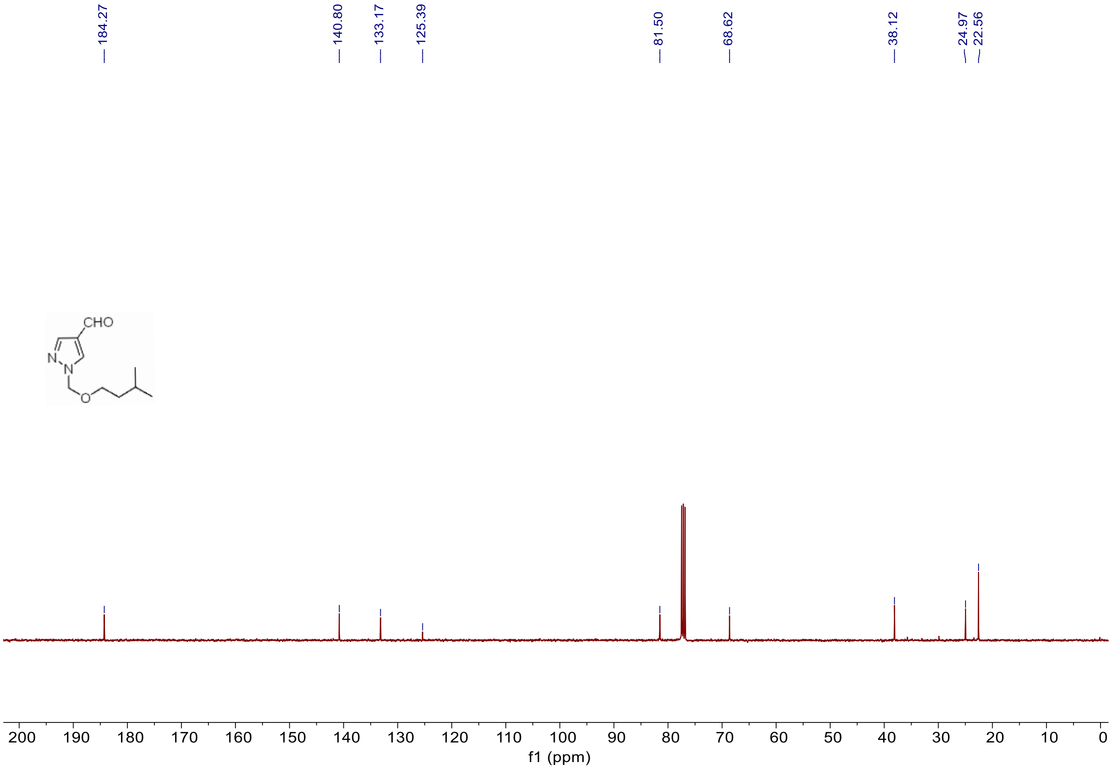
^{13}C NMR spectrum (101 MHz) of **2g** (see procedure)



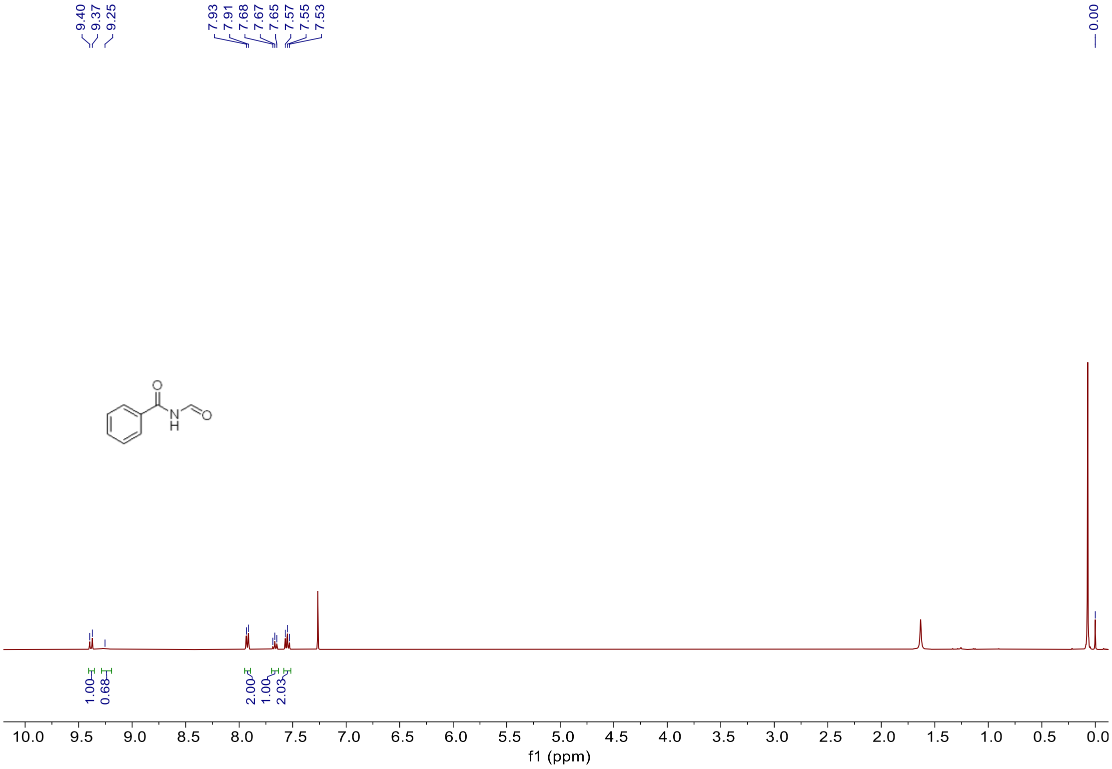
^{1}H NMR spectrum (400 MHz) of **3g** (see procedure)



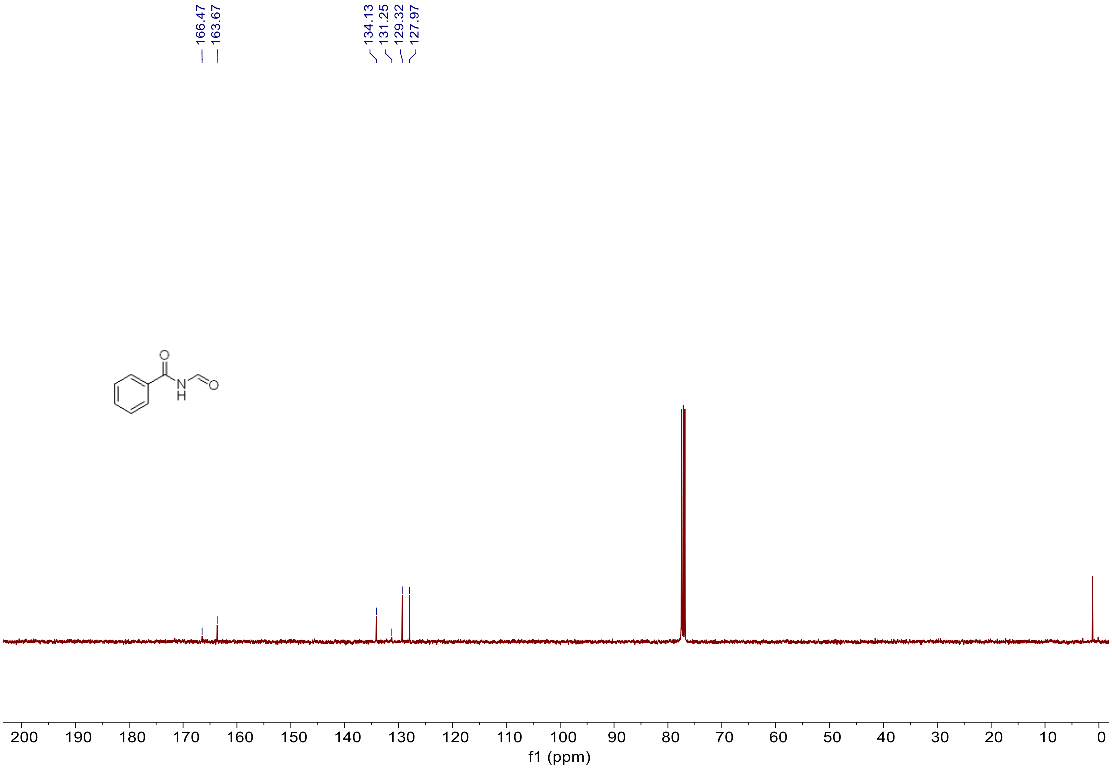
^{13}C NMR spectrum (101 MHz) of **3g** (see procedure)



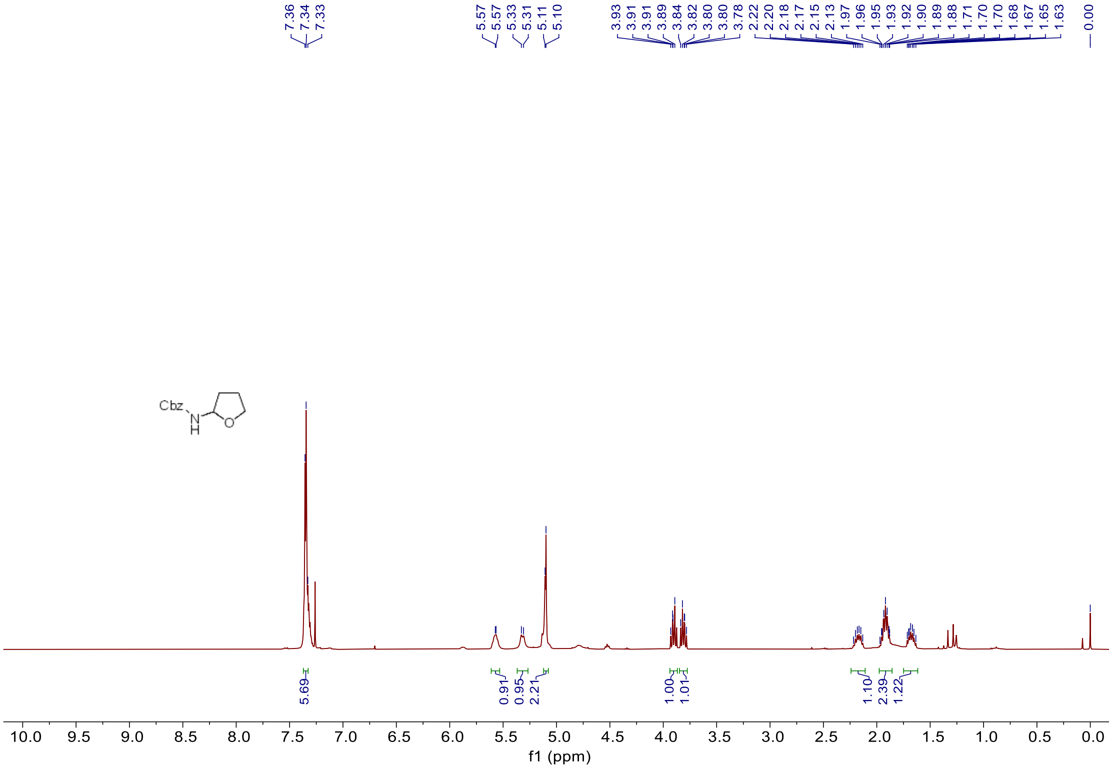
^{1}H NMR spectrum (400 MHz) of **8g** (see procedure)



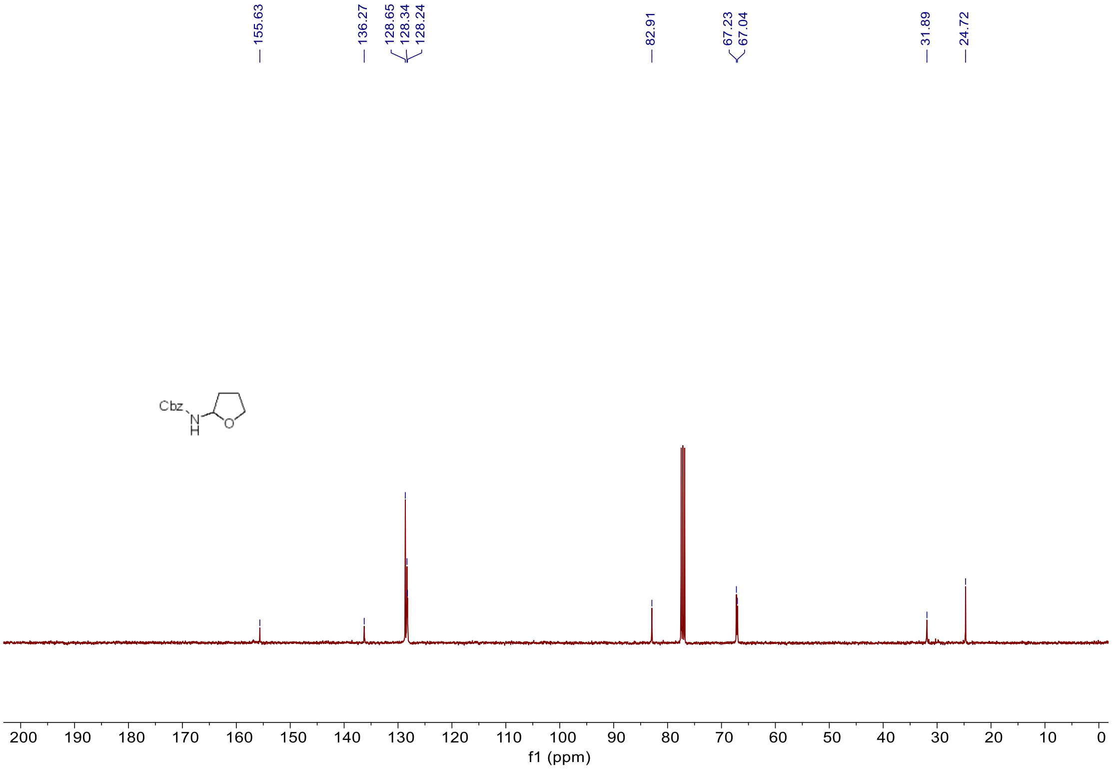
^{13}C NMR spectrum (101 MHz) of **8g** (see procedure)



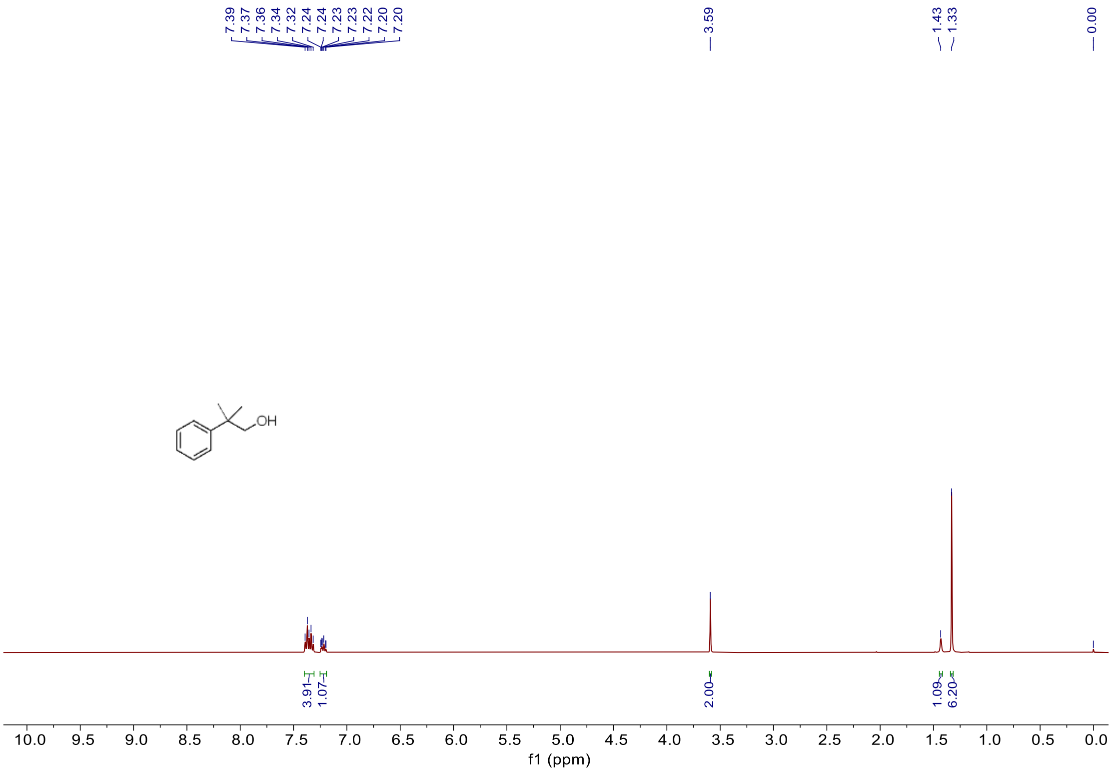
^{1}H NMR spectrum (400 MHz) of **1h** (see procedure)



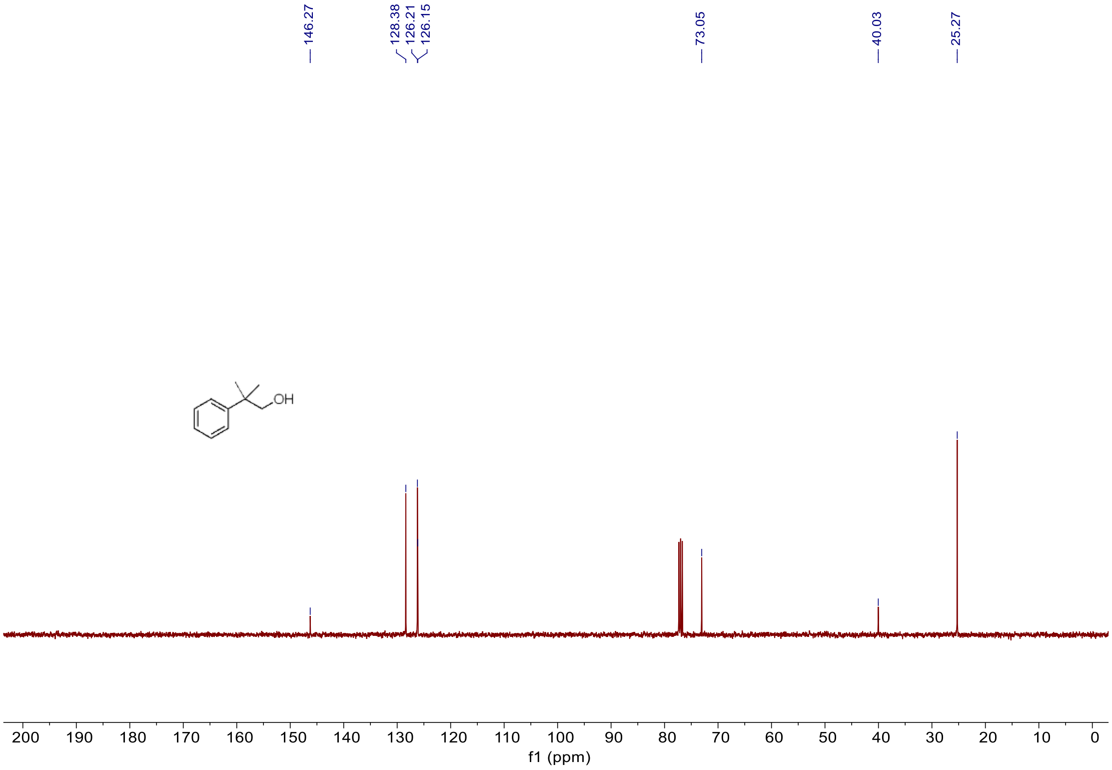
^{13}C NMR spectrum (101 MHz) of **1h** (see procedure)



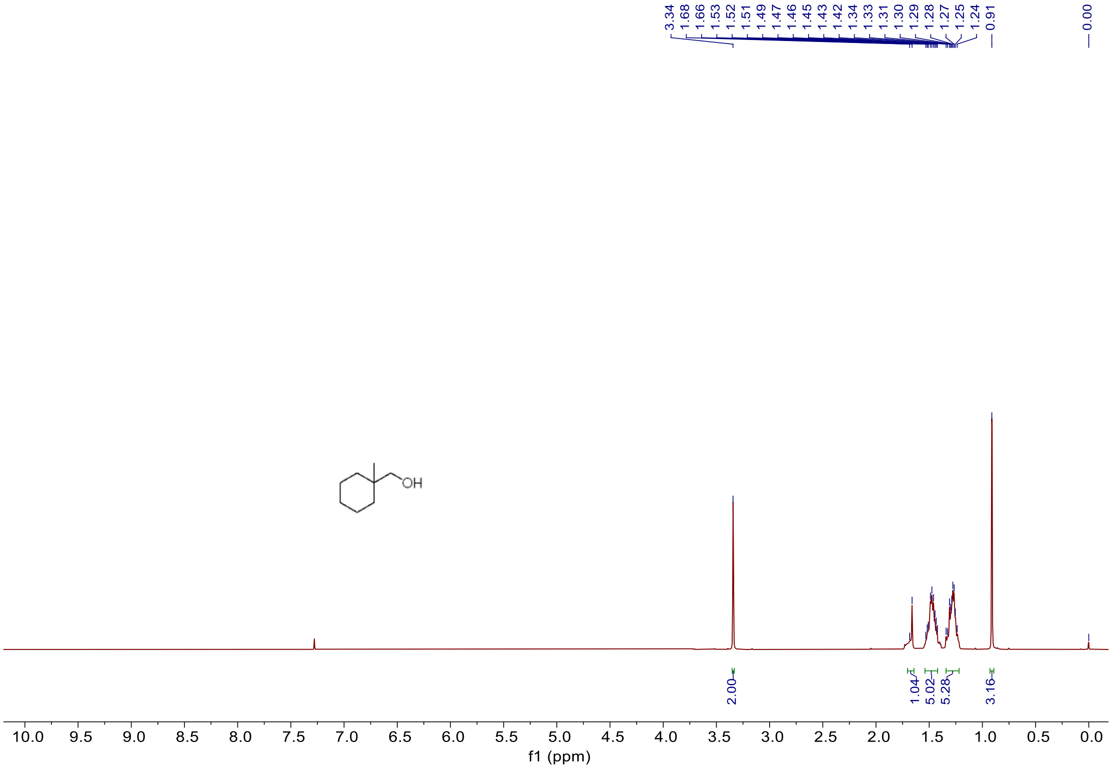
^{1}H NMR spectrum (400 MHz) of **26a** (see procedure)



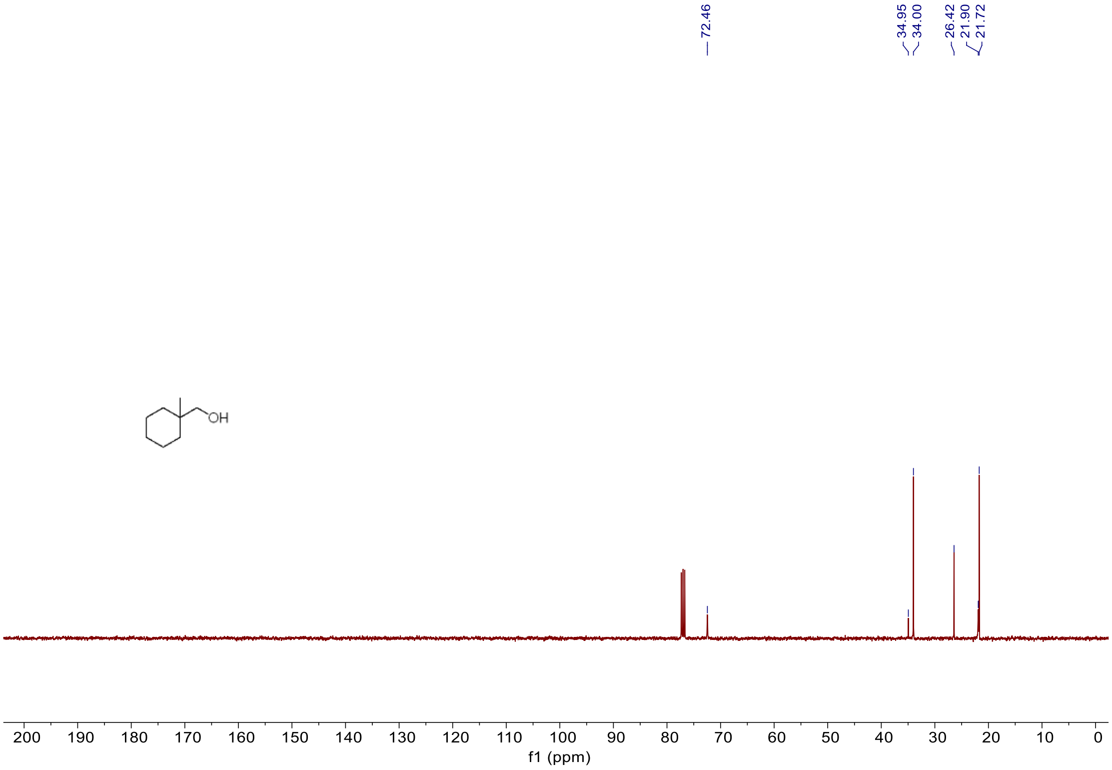
^{13}C NMR spectrum (101 MHz) of **26a** (see procedure)



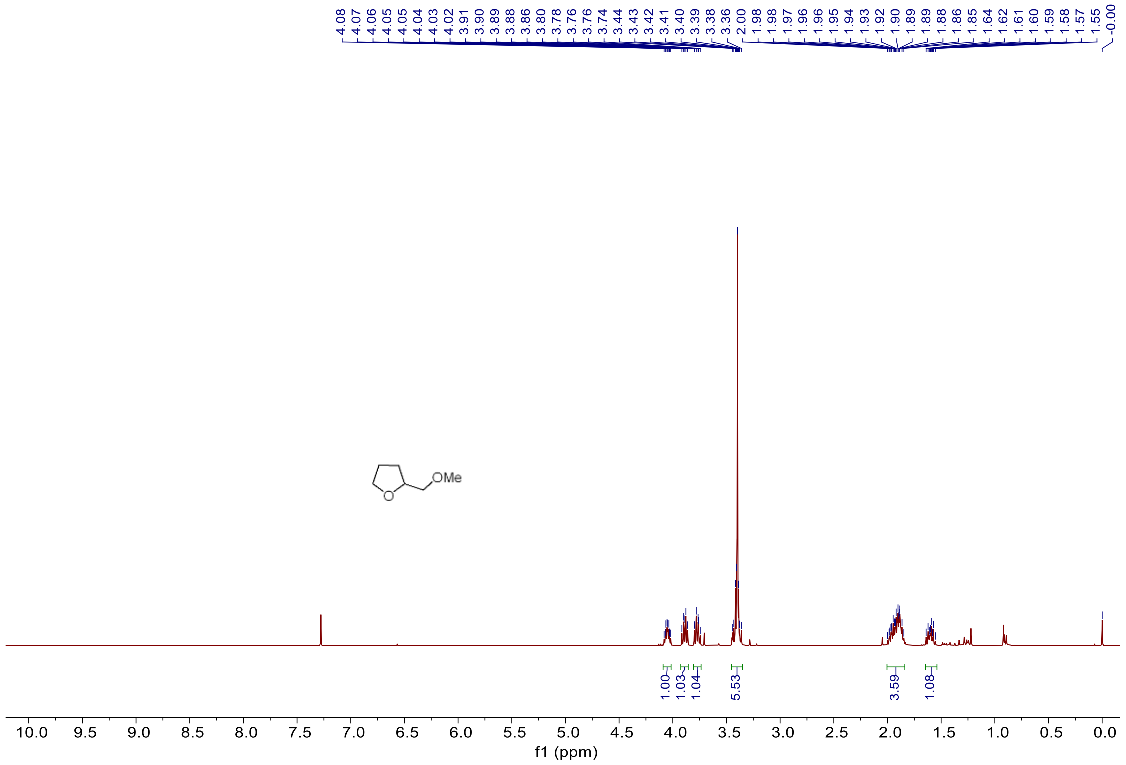
^{1}H NMR spectrum (400 MHz) of **30a** (see procedure)



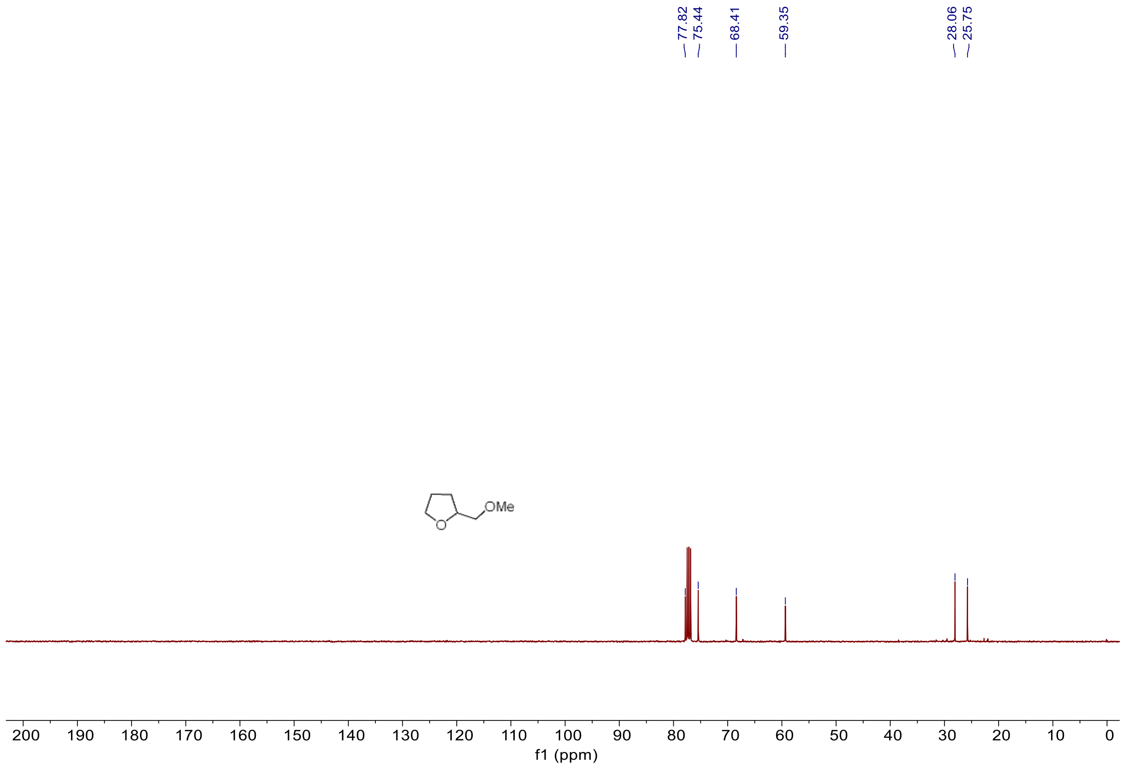
^{13}C NMR spectrum (101 MHz) of **30a** (see procedure)



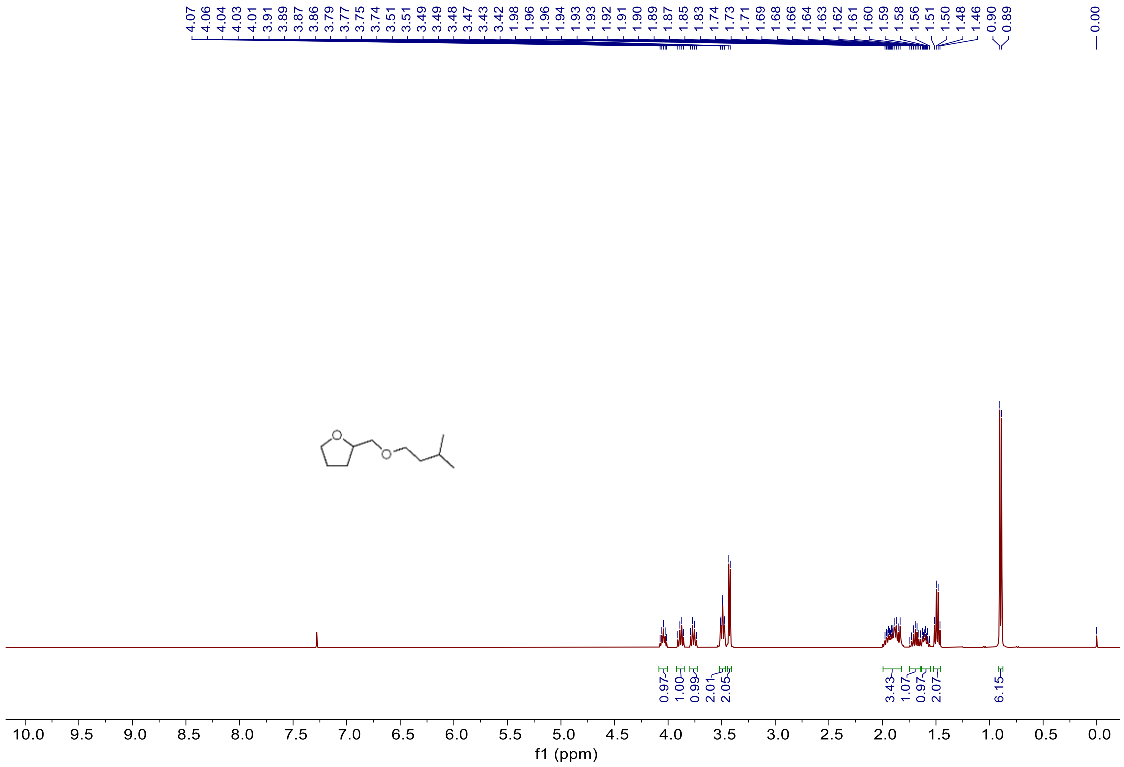
^{1}H NMR spectrum (400 MHz) of **2f** (see procedure)



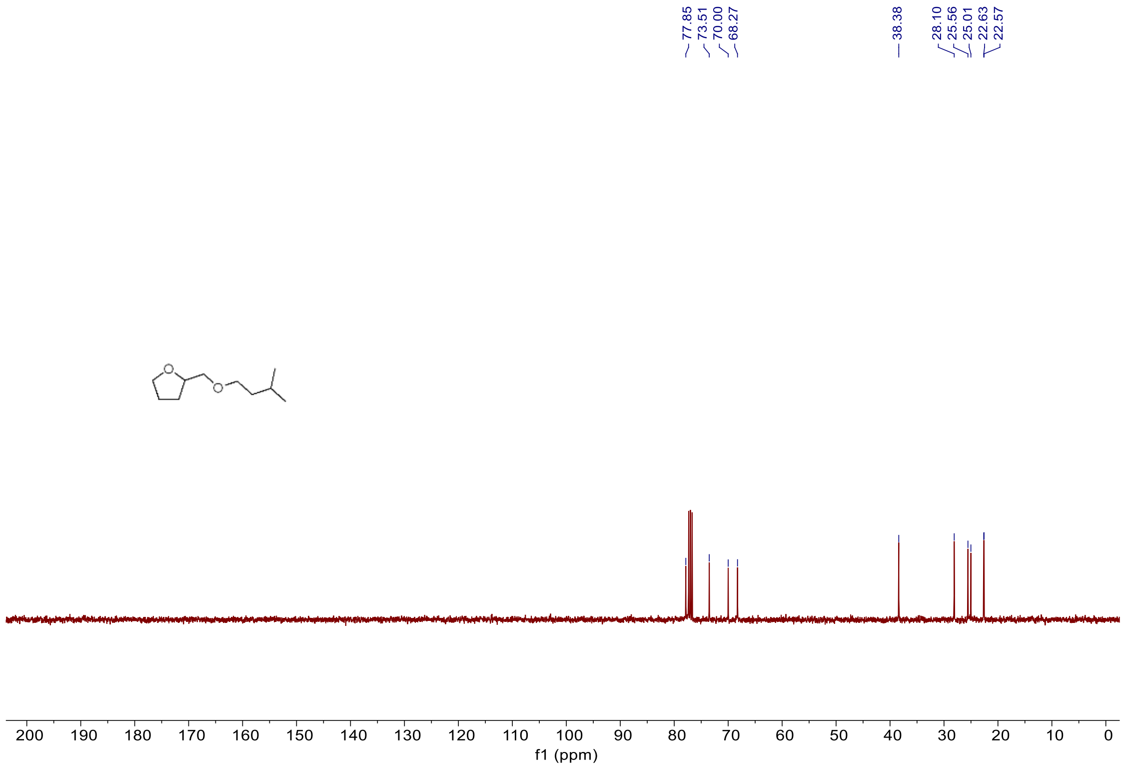
^{13}C NMR spectrum (101 MHz) of **2f** (see procedure)



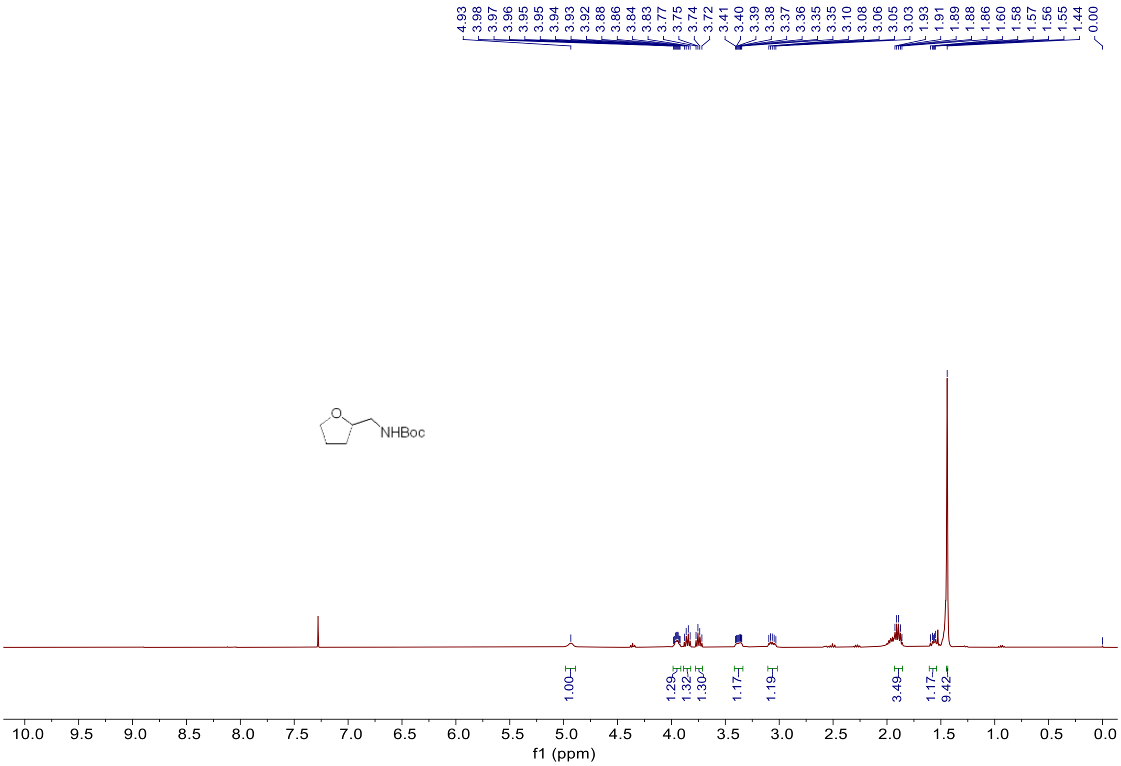
^{1}H NMR spectrum (400 MHz) of **3f** (see procedure)



^{13}C NMR spectrum (101 MHz) of **3f** (see procedure)



^{1}H NMR spectrum (400 MHz) of **4f** (see procedure)



^{13}C NMR spectrum (101 MHz) of **4f** (see procedure)