Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Synthesis of poly-functionalized benzofurans via one-pot domino oxidation/[3+2] cyclization reactions of a hydroquinone ester and ynamides

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1. General

^{1}H NMR and ^{13}C NMR were recorded on a Bruker-400 MHz spectrometer. Proton chemical shifts are reported in ppm downfield from tetramethylsilane or from the residual solvent as internal standard in $CDCl_3$ (δ 7.26 ppm). Carbon chemical shifts were internally referenced to the deuterated solvent signals in $CDCl_3$ (δ 77.0 ppm). High-resolution mass spectra were recorded on a Thermo Scientific LTQ Orbitrap ESI ion trap mass spectrometer. Reagents obtained from commercial sources are used without further purification and all solvets were purified and dried according to standard methods prior to use, unless stated otherwise.

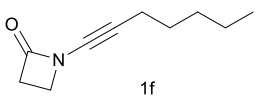
2. Preparation of ynamides

General procedure for the preparation of ynamides 1. Based on the literature procedures,[1,2] amide (3.0-4.0 mmol), $CuCl_2$ (26.9 mg, 0.20 mmol), and Na_2CO_3 (212.0 mg, 2.0 mmol) were combined in a 50 mL three-neck round-bottom flask. The flask was purged with O_2 for 10 min and connected with a ballon filled with O_2. A solution of pyridine (160.0 μL, 2.0 mmol) in 8.0 mL dry toluene was added to the reaction flask via a syringe and the flask was heated to 70 ^{o}C. A solution of alkyne (1.0 mmol) in 5.0 mL dry toluene was added to the flask slowly over a period of 3-4 h. After complete addition of the alkyne, the reaction mixture was allowed to stir at 70 ^{o}C for another 10 h. After cooling to room temperature, the crude mixture was filtered through a pad of Celite, concentrated by rotary evaporation, and purified by flash chromatography to provide the desired product, which was later stored in the freezer.

Procedure for a 6 mmol-scale reaction for the synthesis of 1h. Methyl indole-3-carboxylate (3.15 g, 18.0 mmol), $CuCl_2$ (161.4 mg, 1.2 mmol), and Na_2CO_3 (1.27 g, 12.0 mmol) were combined in a 250 mL three-neck round-bottom flask. The flask was purged with O_2 for 10 min and connected with a ballon filled with O_2. A solution of pyridine (966.7 μL, 12.0 mmol) in 45.0 mL dry toluene was added to the reaction flask via a syringe and the flask was heated to 70 ^{o}C. A solution of 1-heptyne (577.1 mg, 6.0 mmol) in 30.0 mL dry toluene was added to the flask slowly over a period of 4 h. After complete addition of the alkyne, the reaction mixture was allowed to stir at 70 ^{o}C for another 20 h. After cooling to room temperature, the crude mixture was filtered through a pad of Celite, concentrated by rotary evaporation, and purified by flash chromatography to provide **1h** (1.10 g, 68%).

Ynamides **1a**,[1] **1b**,[1] **1c**,[2] **1d**,[1] **1e**,[1] and **1g**[1] are known compounds and characterizations are the same as reported.

1-(Hept-1-yn-1-yl)azetidin-2-one (1f)

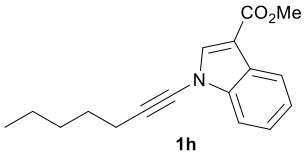


1f

Synthesized by the general procedure; 138.7 mg (84%).
Yellow oil. ^{1}H NMR (400 MHz, CDCl3): δ = 0.87 (t, *J* = 6.8 Hz, 3H, CH_3), 1.23-1.36 (m, 4H, $CH_2\times2$), 1.45-1.52 (m, 2H, CH_2), 2.25 (t, *J* = 7.2Hz, 2H, CH_2), 2.97 (t, *J* = 4.7 Hz, 2H, CH_2) , 3.55 (t, *J* = 4.8 Hz, 2H, CH_2) ppm; ^{13}C NMR (100 MHz, CDCl3): δ = 13.9, 18.3, 22.1, 28.4,

30.9, 37.4, 42.8, 69.9, 70.2, 167.2 ppm. HRMS (ESI): calcd. for $C_{10}H_{16}NO$ ($[M + H]^+$) 166.1226, found 166.1228.

Methyl 1-(hept-1-yn-1-yl)-1H-indole-3-carboxylate (1h)



1h

Synthesized by the general procedure; 177.7 mg (66%).
Colorless solid. ^{1}H NMR (400 MHz, CDCl3): δ = 0.95 (t, *J* = 7.1 Hz, 3H, CH_3), 1.34-1.51 (m, 4H, CH2×2), 1.62-1.68 (m, 2H, CH_2), 2.47 (t, *J* = 7.0 Hz, 2H, CH_2), 3.92 (s, 3H, CH_3), 7.31-7.39 (m, 2H, ArH), 7.55 (d, *J* = 8.0 Hz, 1H, ArH), 7.89 (s, 1H, ArH), 8.16 (d, *J* = 7.4Hz, 1H, ArH) ppm; ^{13}C NMR (100 MHz, CDCl3): δ = 14.0, 18.3, 22.2, 28.4, 31.0, 51.3, 70.5, 71.8, 109.8, 111.4, 121.7, 123.3, 124.1, 125.1, 135.2, 138.5, 164.5 ppm. HRMS (ESI): calcd. for $C_{17}H_{20}NO_2$ ($[M + H]^+$) 270.1489, found 270.1490.

3. Procedure for the oxidation of hydroquinone ester 5

According to literature procedure[3], silver oxide (2.09 g, 9.0 mmol) and magnesium sulfate (1.08 g, 9.0 mmol) were added to a solution of methyl 2,5-dihydroxybenzoate (**5**) (504.5 mg, 3.0 mmol) in diethyl ether (50 mL). The reaction mixture was stirred at 25 ^{o}C for 3 h. After filtration through a pad of Celite, the filtrate was concentrated in vacuo and purified by flash chromatography to furnish the desired quinone ester **2b**. Characterizations are the same as reported.

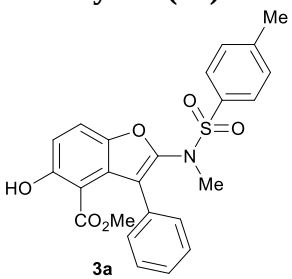
Oxidation using O_2 as the oxidant. Hydroquinone ester **5** (20.2 mg, 0.12 mmol), $MgSO_4$ (28.9 mg, 0.24 mmol) were mixed in CH_2Cl_2 (2.0 mL) under oxygen atmosphere (use of O_2 balloon). The reaction was stirred at room temperature (25 ^{o}C) for 8 h. TLC indicated that only small amount of **5** was oxidized, which indicated that O_2 is not a good oxidant for the oxidation of **5**.

4. Procedures for the one-pot domino oxidation/[3+2] cyclization

General procedure for the one-pot domino oxidation/[3+2] cyclization. Hydroquinone ester **5** (20.2 mg, 0.12 mmol), Ag_2O (55.6 mg, 0.24 mmol) and $MgSO_4$ (28.9 mg, 0.24 mmol) were mixed in CH_2Cl_2 (2.0 mL) and stirred at room temperature (25 ^{o}C) for 2 h. Then, ynamide **1** (0.1 mmol) and $Sc(OTf)_3$ (1.0 mg, 0.002 mmol) were added to the above mixture. All the reactions finished with 5 min. The crude reaction mixture was filtered through a pad of Celite, concentrated by rotary evaporation, and purified by flash chromatography to provide the desired product **3**.

Procedure for a large scale one-pot reaction. Hydroquinone ester **5** (1.23 g, 4.90 mmol), Ag_2O (3.41 g, 14.7 mmol) and $MgSO_4$ (1.77 g, 14.7 mmol) were mixed in CH_2Cl_2 (60.0 mL) and stirred at room temperature (25 ^{o}C) for 4 h. Ynamide **1h** (1.10 g, 4.08 mmol) and $Sc(OTf)_3$ (20.2 mg, 0.041 mmol) were added to the above mixture. The reaction was allowed to stir for 30 min. Then, the crude reaction mixture was filtered through a pad of Celite, concentrated by rotary evaporation, and purified by flash chromatography to provide the desired product **3h** (1.64 g, 92%).

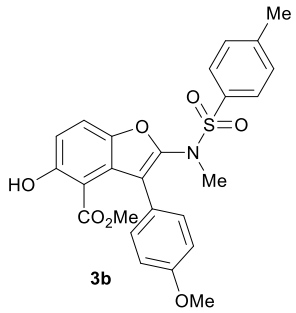
Methyl 2-((N,4-dimethylphenyl)sulfonamido)-5-hydroxy-3-phenylbenzofuran-4-carboxylate (3a)



3a

Synthesized by the general procedure; 40.9 mg (91%).
Light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (s, 3H, CH_3), 2.98 (s, 3H, CH_3), 3.02 (s, 3H, CH_3), 7.02 (d, *J* = 9.0 Hz, 1H, ArH), 7.27-7.42 (m, 7H, ArH), 7.53 (d, *J* = 9.0 Hz, 1H, ArH), 7.61 (d, *J* = 8.3 Hz, 2H, ArH), 10.8 (s, 1H, OH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.6, 37.7, 51.0, 105.1, 115.7, 118.5, 119.4, 126.0, 127.0, 127.9, 128.2, 129.1, 129.6, 133.9, 135.0, 144.1, 146.0, 148.1, 159.1, 170.0 ppm; HRMS (ESI): calcd. for $C_{24}H_{22}NO_6S$ ($[M + H]^+$) 452.1162, found 452.1163.

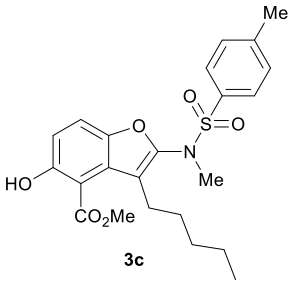
Methyl 2-((N,4-dimethylphenyl)sulfonamido)-5-hydroxy-3-(4-methoxyphenyl)benzofuran-4-carboxylate (3b)



3b

Synthesized by the general procedure; 42.3 mg (88%).
Light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.46 (s, 3H, CH_3), 2.97 (s, 3H, CH_3), 3.11 (s, 3H, CH_3), 3.87 (s, 3H, CH_3), 6.94 (d, *J* = 8.6 Hz, 2H, ArH), 7.01 (d, *J* = 9.0 Hz, 1H, ArH), 7.23-7.30 (m, 4H, ArH), 7.50 (d, *J* = 9.0 Hz, 1H, ArH), 7.64 (d, *J* = 8.2 Hz, 2H, ArH), 10.71 (s, 1H, OH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.7, 37.7, 51.2, 55.3, 105.2, 113.3, 115.6, 118.4, 119.1, 124.1, 126.2, 128.2, 129.57, 129.58, 130.1, 135.2, 144.1, 146.0, 148.2, 159.1, 170.1 ppm; HRMS (ESI): calcd. for $C_{25}H_{24}NO_7S$ ($[M + H]^+$) 482.1268, found 482.1265.

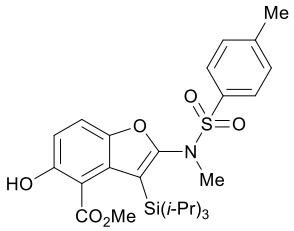
Methyl 2-((N,4-dimethylphenyl)sulfonamido)-5-hydroxy-3-pentylbenzofuran-4-carboxylate (3c)



3c

Synthesized by the general procedure; 41.8 mg (94%).
Light yellow solid. ^{1}HNMR (400 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 6.8 Hz, 3H, CH_3), 1.29-1.46 (m, 6H, CH_2×3), 2.46 (s, 3H, CH_3), 2.85 (t, *J* = 7.6 Hz, 2H, CH_2), 3.16 (s, 3H, CH_3), 4.02 (s, 3H, CH_3), 6.94 (d, *J* = 9.0 Hz, 1H, ArH), 7.32 (d, *J* = 8.0 Hz, 2H, ArH), 7.36 (d, *J* = 9.0 Hz, 1H, ArH), 7.68 (d, *J* = 8.3 Hz, 1H, ArH), 10.9 (s, 1H, OH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 21.6, 22.6, 25.9, 29.3, 32.1, 37.6, 51.9, 105.4, 115.2, 118.5, 118.6, 125.7, 128.1, 129.6, 134.7, 144.2, 146.1, 147.8, 159.2, 170.7 ppm; HRMS (ESI): calcd. for $C_{23}H_{28}NO_6S$ ($[M + H]^+$) 446.1632, found 446.1634.

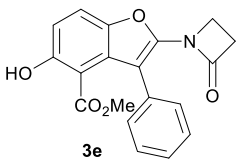
Methyl 2-((N,4-dimethylphenyl)sulfonamido)-5-hydroxy-3-(triisopropylsilyl)benzofuran-4-carboxylate (3d)



3d

Synthesized by the general procedure; 45.4 mg (85%).
Light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.20 (br, 18H, CH_3×6), 1.61-1.68 (m, 3H, CH×3), 2.48 (s, 3H, CH_3), 3.02 (s, 3H, CH_3), 3.94 (s, 3H, CH_3), 6.93 (d, *J* = 9.0 Hz, 1H, ArH), 7.29 (d, *J* =9.0 Hz, 1H, ArH), 7.34 (d, *J* = 8.2 Hz, 2H, ArH), 7.71 (d, *J* = 8.2 Hz, 2H, ArH), 9.51 (s, 1H, OH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ= 13.5, 20.0, 38.7, 52.8, 109.3, 110.8, 115.2, 116.9, 127.3, 129.2, 129.4, 130.0, 130.7, 133.6, 144.4, 146.8, 156.3, 170.0 ppm; HRMS (ESI): calcd. for $C_{27}H_{38}NO_6SSi$ ($[M + H]^+$) 532.2184, found 532.2189.

Methyl 5-hydroxy-2-(2-oxoazetidin-1-yl)-3-phenylbenzofuran-4-carboxylate (3e)

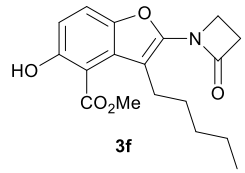


3e

Synthesized by the general procedure; 28.8 mg (85%).
Light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.99 (s, 3H, CH_3), 3.03 (t, *J* = 4.8 Hz, 2H, CH_2), 3.20 (t, *J* = 4.7 Hz, 2H, CH_2), 6.91 (d, *J* = 9.0 Hz, 1H, ArH), 7.29-7.31 (m, 2H, ArH), 7.36-7.43 (m, 3H, ArH), 7.59 (d, *J* = 8.9 Hz, 1H, ArH), 10.9 (s, 1H, OH) ppm; ^{13}C

NMR (100 MHz, $CDCl_3$): δ = 38.0, 41.0, 50.8, 104.4, 108.0, 113.8, 118.2, 126.8, 127.3, 127.7, 130.2, 133.6, 145.1, 145.9, 159.5, 164.1, 170.3 ppm; HRMS (ESI): calcd. for $C_{19}H_{16}NO_5$ ($[M + H]^+$) 338.1023, found 338.1022.

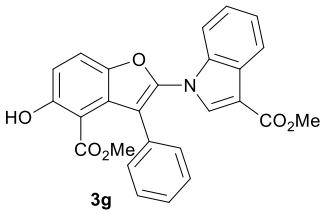
Methyl 5-hydroxy-2-(2-oxoazetidin-1-yl)-3-pentylbenzofuran-4-carboxylate (3f)



3f

Synthesized by the general procedure; 30.1 mg (91%).
Light yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ = 0.87 (t, *J* = 6.9 Hz, 3H, CH_3), 1.25-1.45 (m, 6H, CH_2 ×3), 2.80 (t, *J* = 7.6 Hz, 2H, CH_2), 3.21 (t, *J* = 4.7 Hz , 2H, CH_2), 3.81 (t, *J* = 4.6 Hz, 2H, CH_2), 3.99 (s, 3H, CH_3), 6.89 (d, *J* = 9.0 Hz, 1H, ArH), 7.45 (d, *J* = 9.0 Hz, 1H, ArH), 10.9 (s, 1H, OH) ppm; ^{13} C NMR (100 MHz, $CDCl_3$): δ = 14.1, 22.6, 25.1, 29.7, 31.9, 37.3, 40.9, 51.8, 105.1, 110.8, 114.1, 118.2, 126.6, 144.6, 145.6, 159.2, 164.5, 170.8 ppm; HRMS (ESI): calcd. for $C_{18}H_{22}NO_5$ ($[M + H]^+$) 332.1492, found 332.1494.

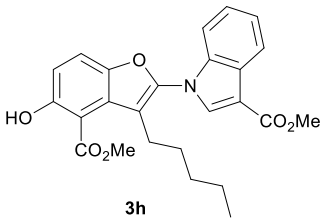
Methyl 1-(5-hydroxy-4-(methoxycarbonyl)-3-phenylbenzofuran-2-yl)-1H-indole-3-carboxylate (3g)



3g

Synthesized by the general procedure; 39.6 mg (90%).
Light yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.05 (s, 3H, CH_3), 3.86 (s, 3H, CH_3), 7.09 (d, *J* = 9.0 Hz, 1H, ArH), 7.16-7.19 (m, 2H, ArH), 7.28-7.34 (m, 5H, ArH), 7.42-7.44 (m, 1H, ArH), 7.60 (s, 1H, ArH), 7.67 (d, *J* = 9.0 Hz, 1H, ArH), 8.14-8.16 (m, 1H, ArH), 10.8 (s, 1H, OH) ppm; ^{13}C NMR(100 MHz, $CDCl_3$): δ = 51.12, 51.26, 105.1, 110.9, 111.7, 115.7, 118.6, 121.7, 123.2, 124.2, 126.0, 126.1, 127.5, 128.4, 128.9, 132.8, 133.9, 137.5, 145.8, 146.0, 159.5, 164.7, 170.0 ppm; HRMS (ESI): calcd. for $C_{26}H_{20}NO_6$ ($[M + H]^+$) 442.1285, found 442.1284.

Methyl 1-(5-hydroxy-4-(methoxycarbonyl)-3-pentylbenzofuran-2-yl)-1H-indole-3-carboxylate (3h)



3h

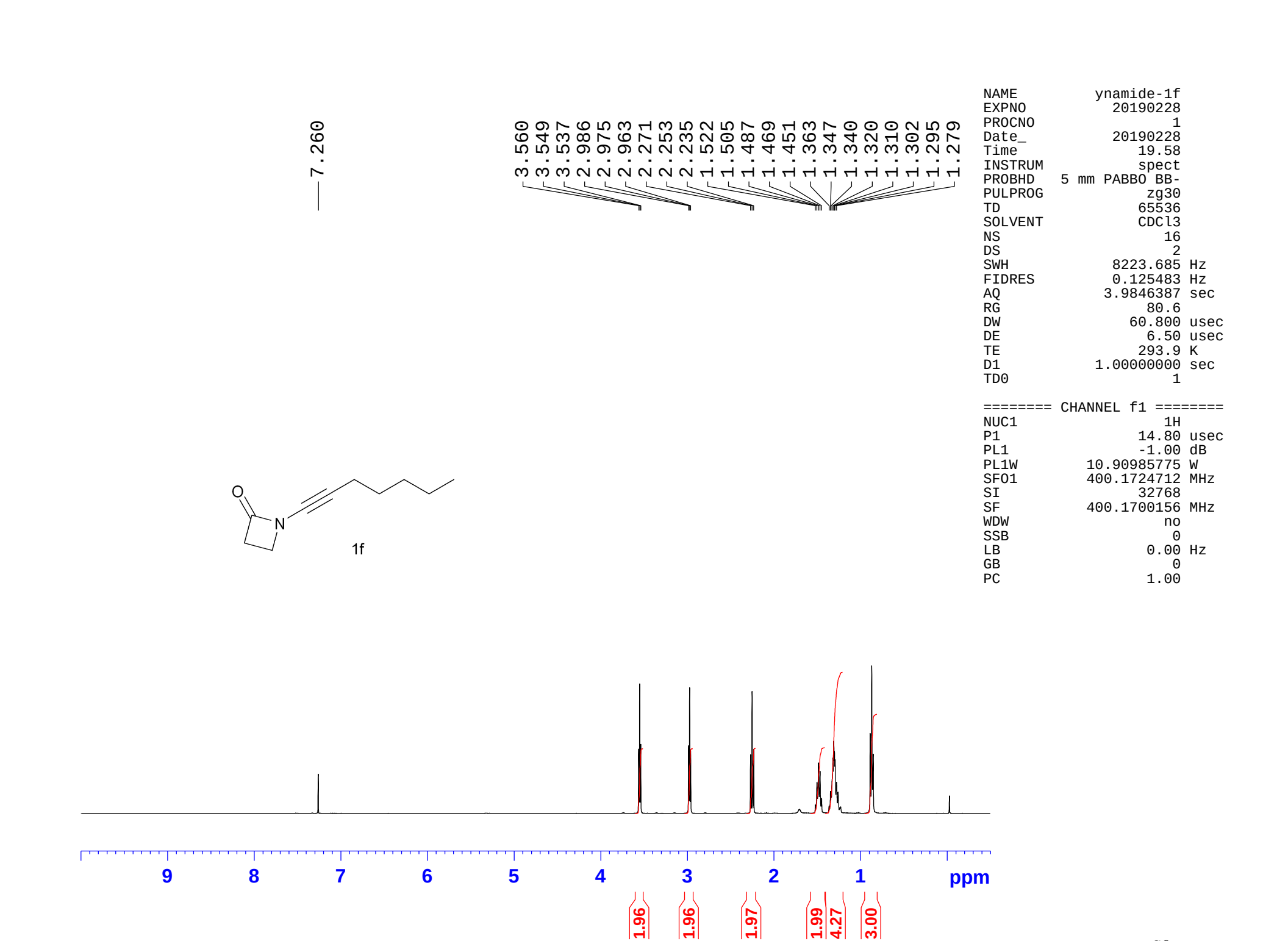
Synthesized by the general procedure; 41.2 mg (90%); for a large scale reaction 1.64 g (92%). Light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.72 (t, *J* = 6.7 Hz, 3H, CH_3), 1.05-1.12 (m, 3H, CH_2 ×1.5), 1.35-1.45 (m, 3H, CH_2 ×1.5), 2.64 (t, *J* = 7.6 Hz, 2H, CH_2), 3.96 (s, 3H, CH_3), 4.04 (s, 3H, CH_3), 7.05 (d, *J* = 9.0 Hz, 1H, ArH), 7.28-7.39 (m, 3H, ArH), 7.59 (d, *J* = 9.0 Hz, 1H, ArH), 7.96 (s, 1H, ArH), 8.24-8.27 (m, 1H, ArH), 11.0 (s, 1H, OH) ppm; ^{13}C NMR(100 MHz, $CDCl_3$): δ = 13.8, 22.3, 25.5, 29.5, 31.5, 51.4, 52.0, 105.4, 111.0, 111.3, 115.0, 115.7, 119.0, 121.9, 123.2, 124.3, 125.7, 126.1, 134.3, 137.8, 145.1, 146.5, 159.6, 164.9, 170.5 ppm; HRMS (ESI): calcd. for $C_{25}H_{26}NO_6$ ($[M + H]^+$) 436.1755, found 436.1777.

5. References

[1] T. Hamada, X. Ye and S. S. Stahl, *J. Am. Chem. Soc.*, 2008, **130**, 833.

[2] W. D. Mackay, M. Fistikci, R. M. Carris and J. S. Johnson, *Org. Lett.*, 2014, **16**, 1626.

[3] Y. H. Chen, D. J. Cheng, J. Zhang, Y. Wang, X. Y. Liu and B. Tan, *J. Am. Chem. Soc.*, 2015, **137**, 15062.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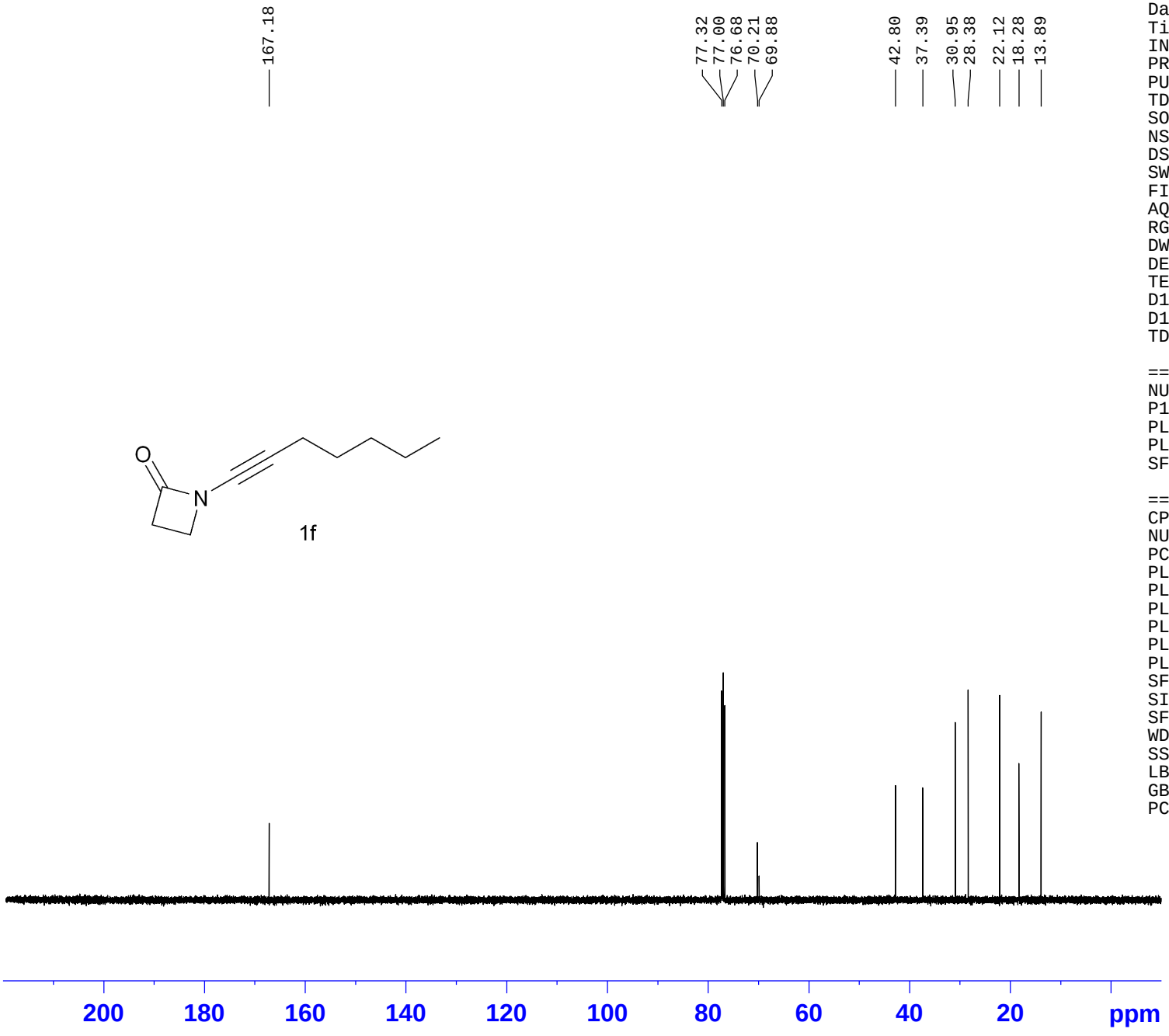


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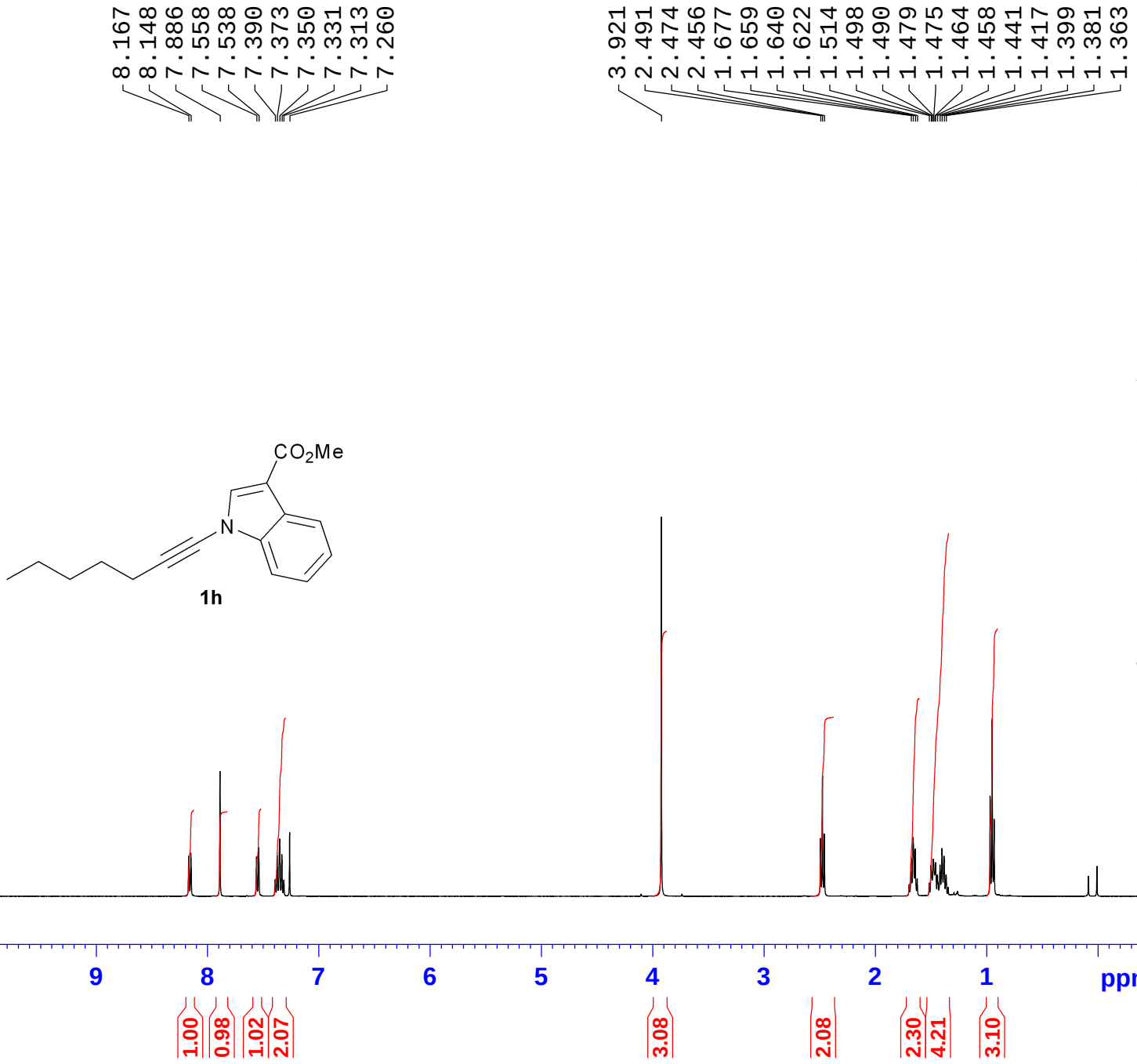

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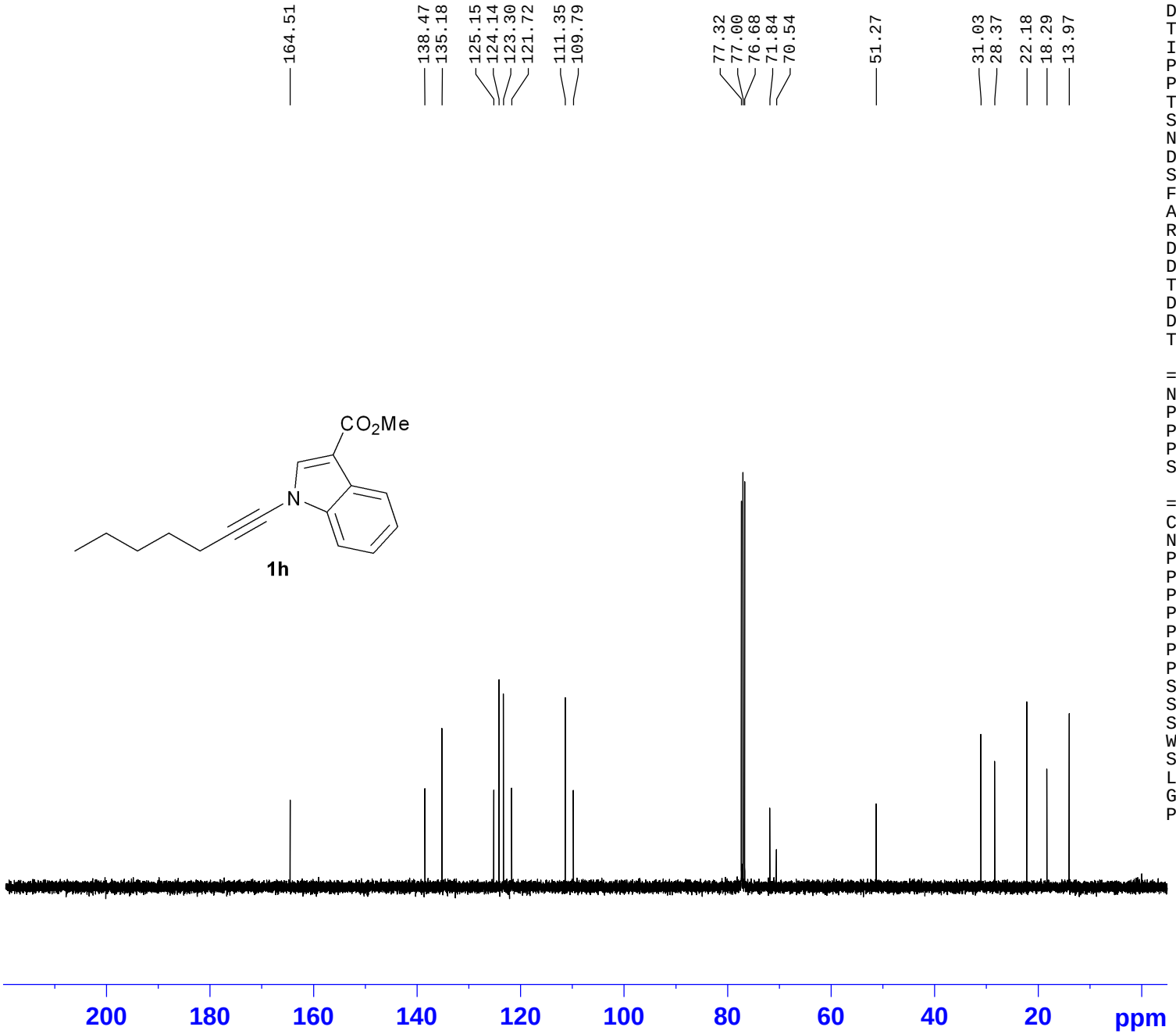
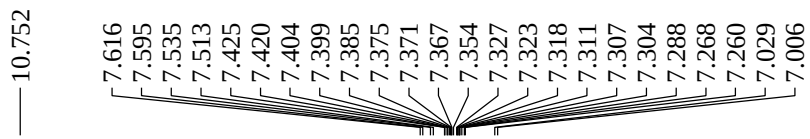
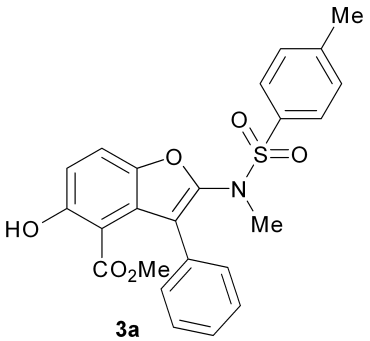
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	90.5
DW	60.800 usec
DE	6.50 usec
TE	294.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.80 usec
PL1	-1.00 dB
PL1W	10.90985775 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700153 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



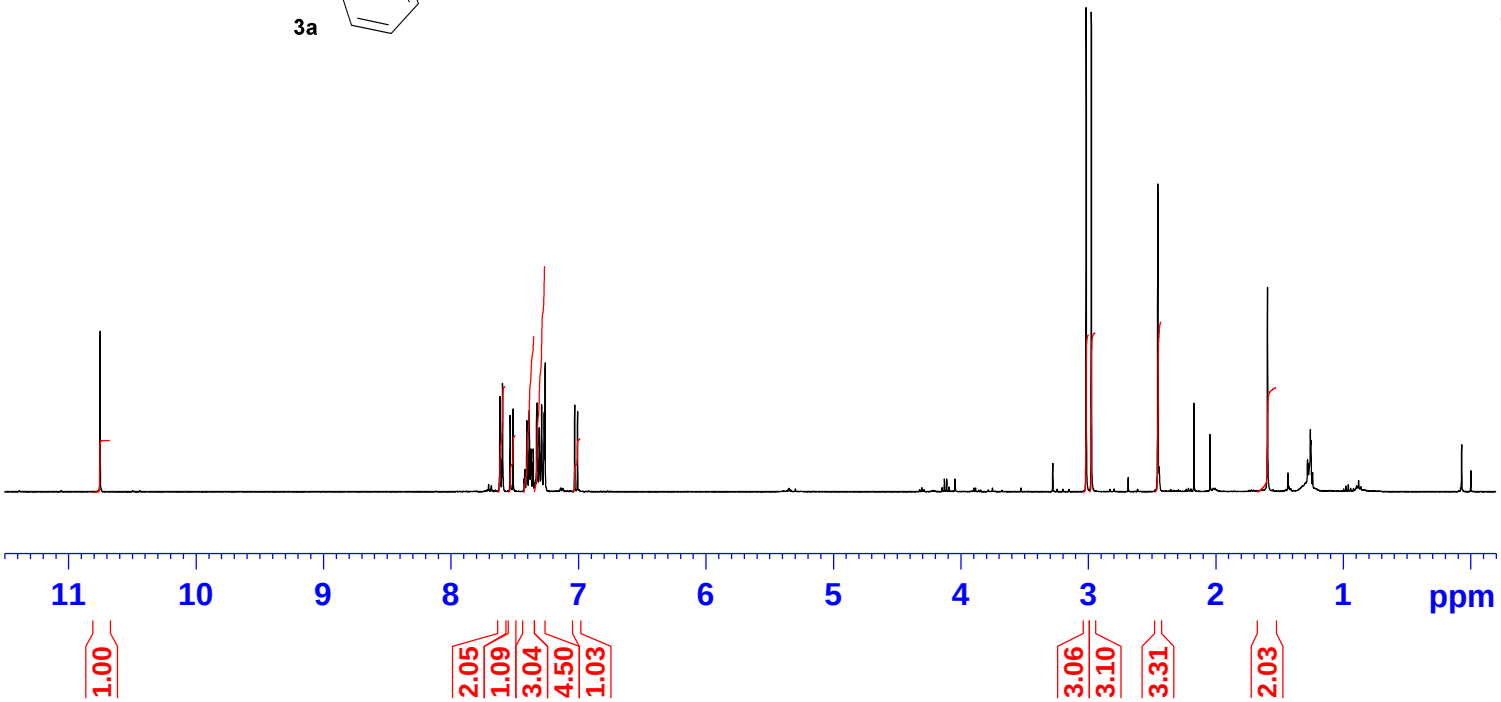

```
NAME          ynamide-1h
EXPNO         2019022802
PROCNO                1
Date_          20190228
Time              20.19
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  179
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 2050
DW                20.800 usec
DE                 6.50 usec
TE                294.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -1.10 dB
PL1W        40.29647064 W
SFO1        100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               14.68 dB
PL13               17.68 dB
PL2W        10.90985775 W
PL12W        0.29499799 W
PL13W        0.14784923 W
SFO2        400.1716007 MHz
SI                 32768
SF          100.6228341 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.40
```

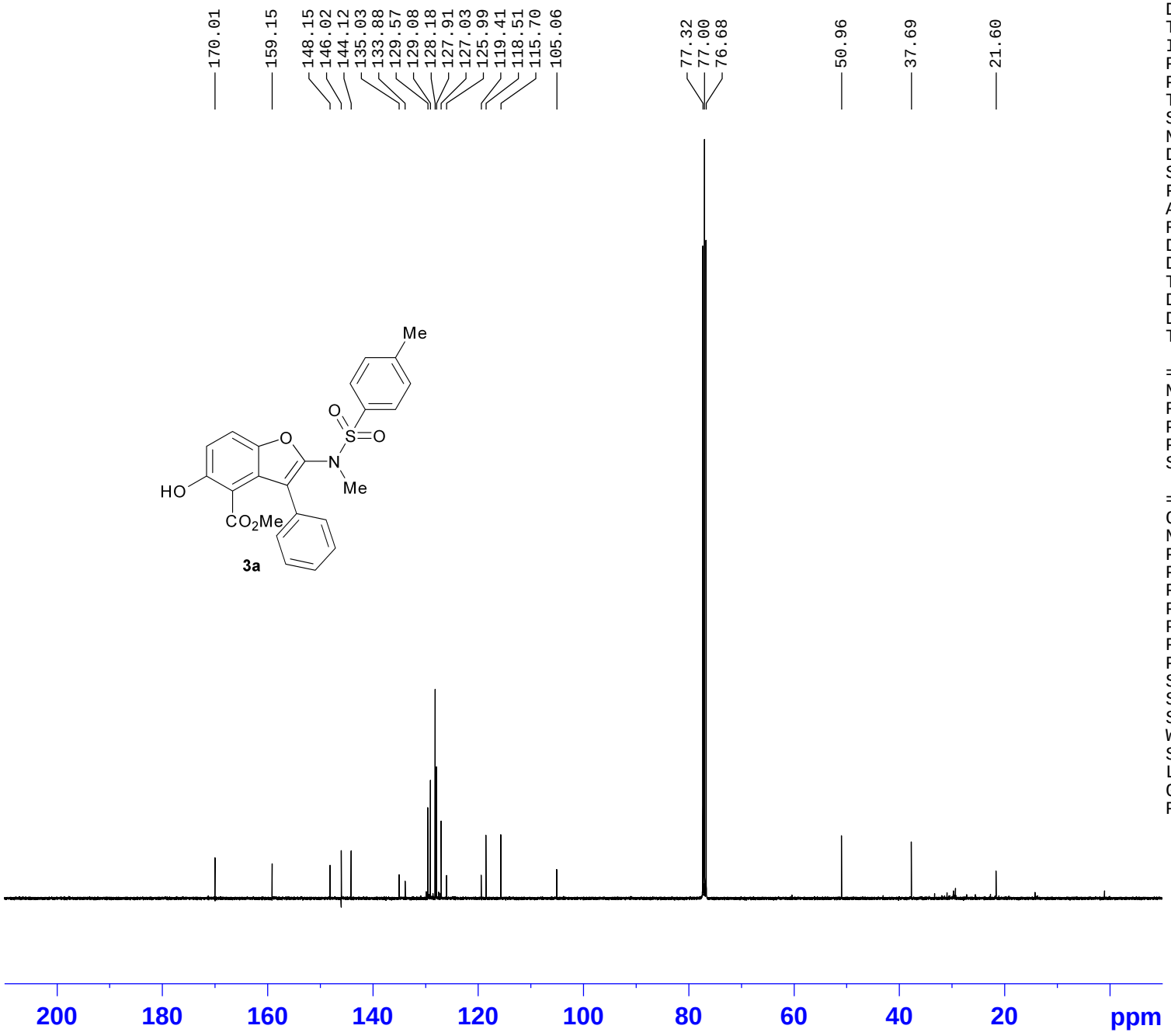




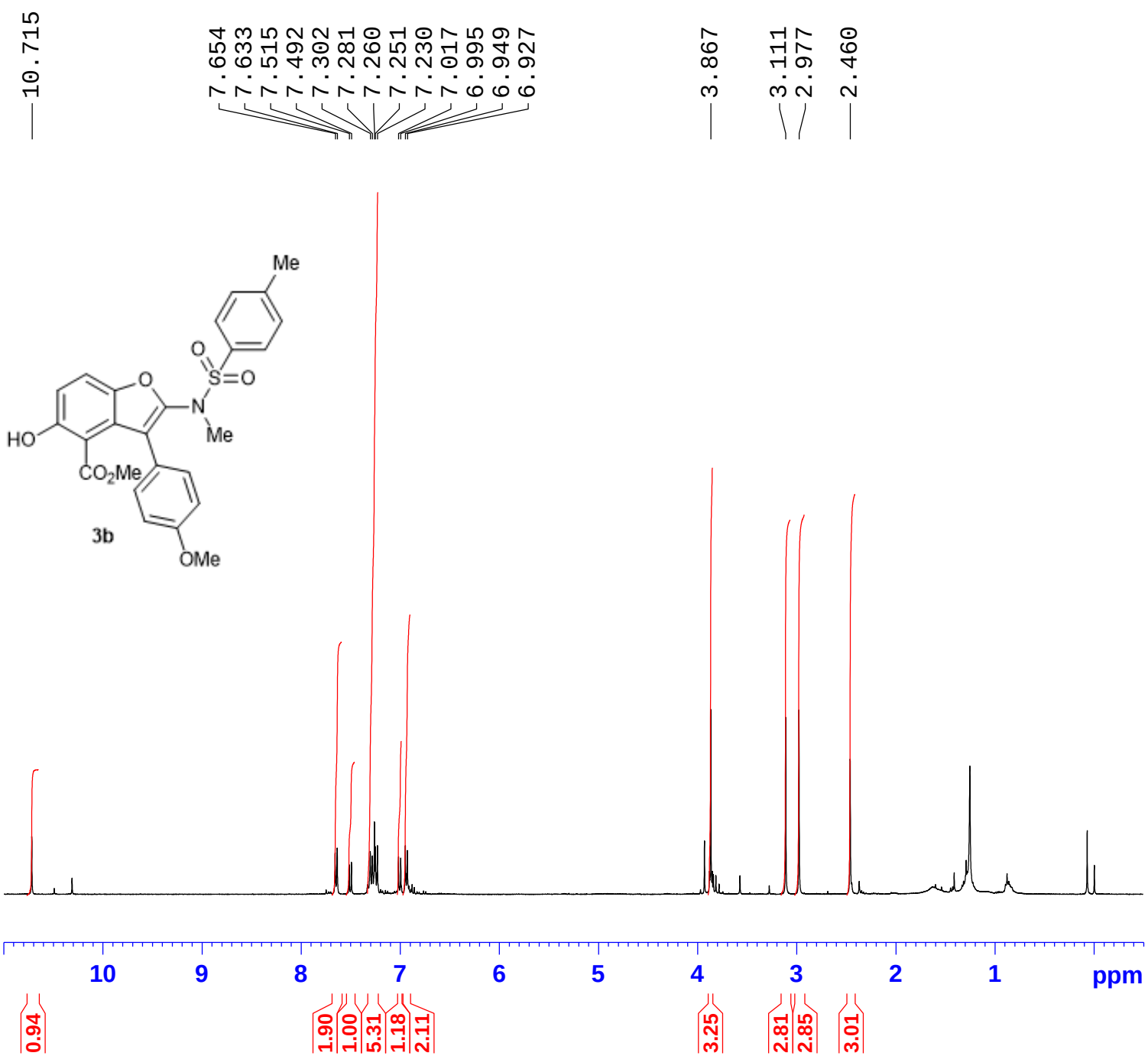
3a

```
NAME            fanyin 5
EXPNO          20181127
PROCNO                1
Date_          20181127
Time              15.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  228
DW                60.800 usec
DE                 6.50 usec
TE                296.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.80 usec
PL1               -1.00 dB
PL1W        10.90985775 W
SFO1        400.1724712 MHz
SI                32768
SF          400.1700153 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

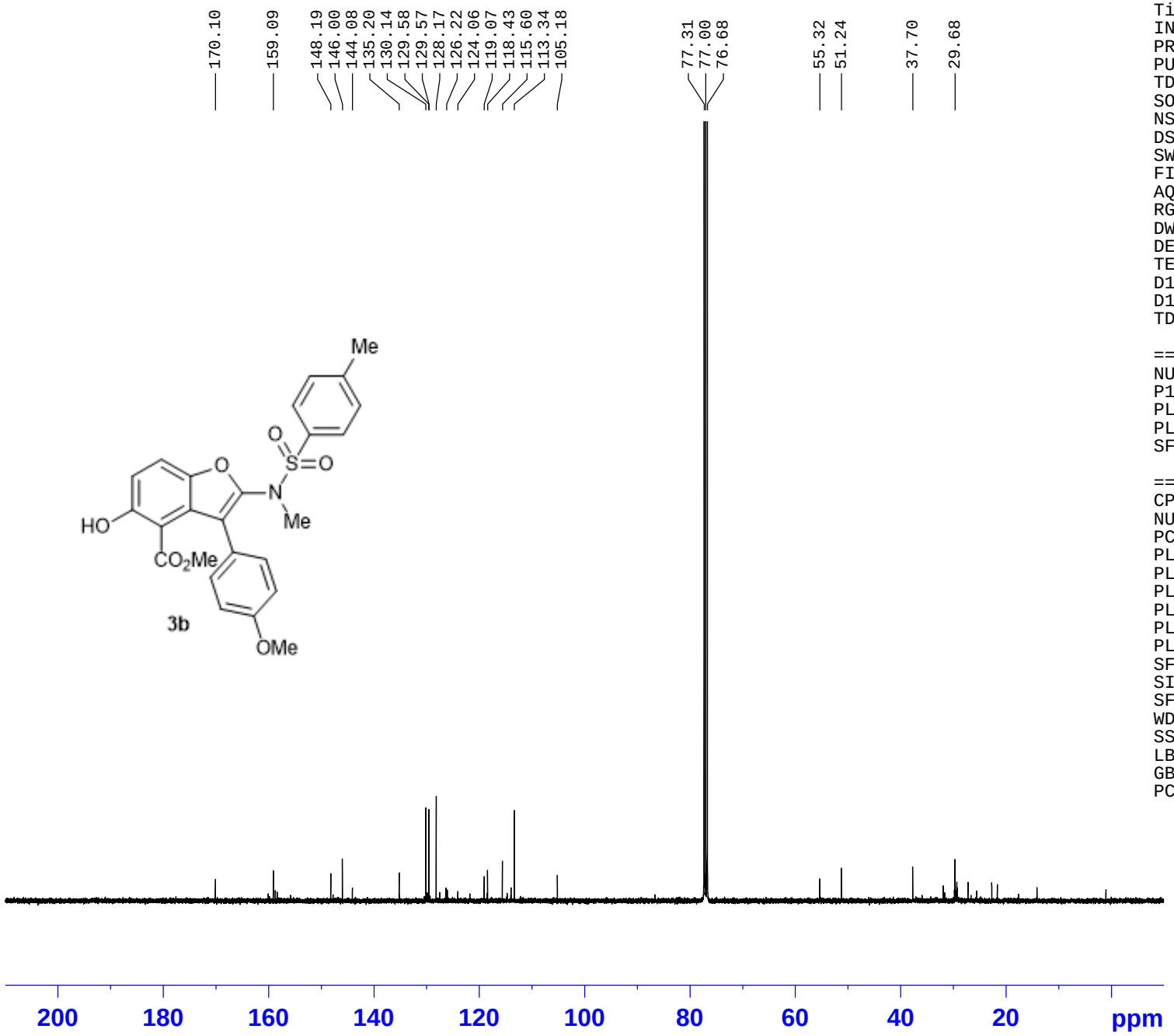




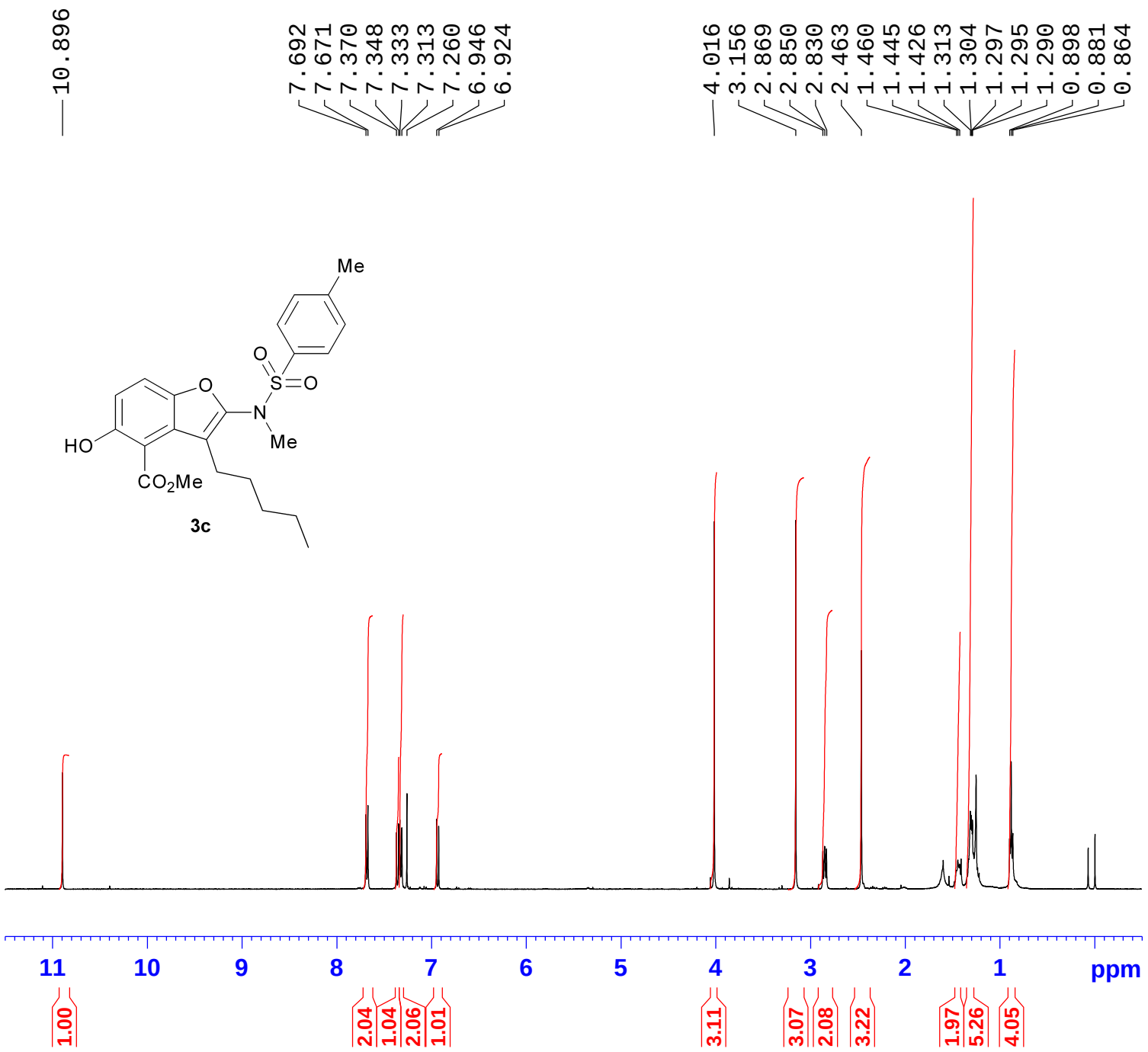

```
NAME            fanyin 5
EXPNO        2018112701
PROCNO              1
Date_           20181127
Time               15.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                10000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 2050
DW                20.800 usec
DE                 6.50 usec
TE                296.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -1.10 dB
PL1W        40.29647064 W
SFO1        100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               14.68 dB
PL13               17.68 dB
PL2W        10.90985775 W
PL12W        0.29499799 W
PL13W        0.14784923 W
SFO2        400.1716007 MHz
SI                 32768
SF          100.6228319 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.40
```



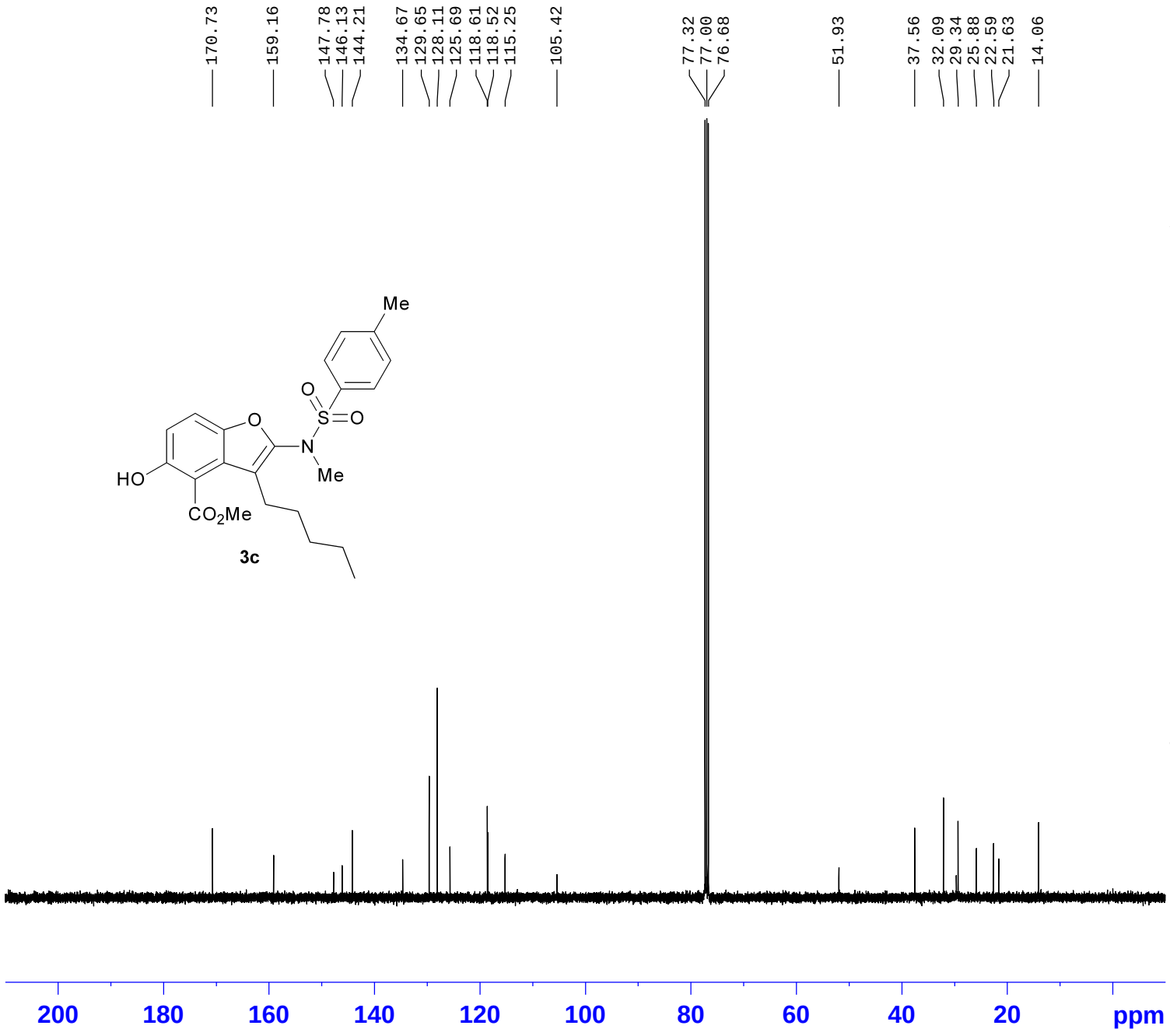

```
NAME                dz01
EXPNO           20181130
PROCNO                 1
Date_           20181130
Time                17.07
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    203
DW                 60.800 usec
DE                  6.50 usec
TE                 295.6 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.80 usec
PL1                -1.00 dB
PL1W         10.90985775 W
SFO1         400.1724712 MHz
SI                 32768
SF           400.1700158 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



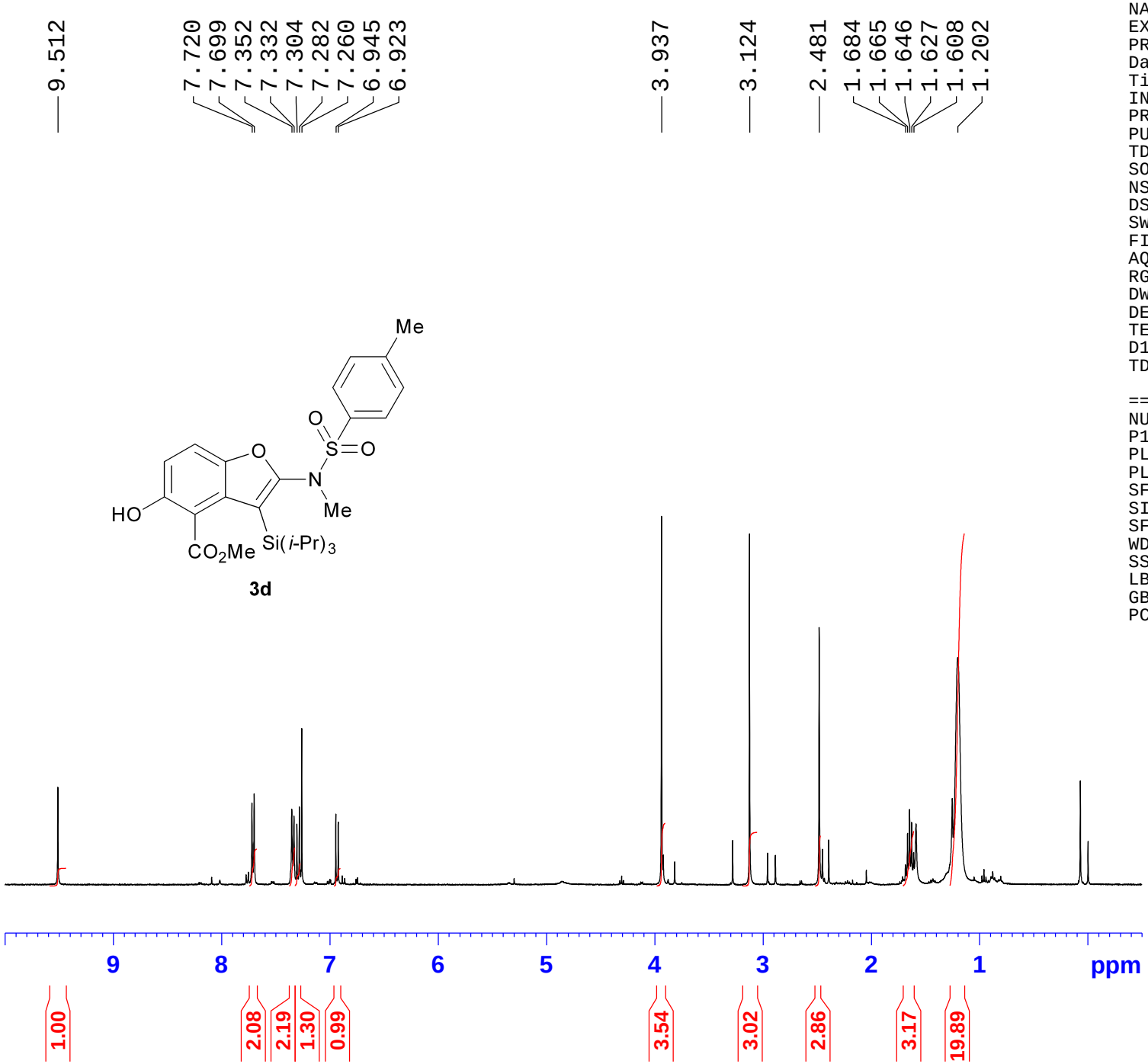

```
NAME                dz01
EXPNO         2018113004
PROCNO                 1
Date_           20181130
Time               22.38
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                12000
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE               295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.90 usec
PL1              -1.10 dB
PL1W        40.29647064 W
SFO1       100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -1.00 dB
PL12              14.68 dB
PL13              17.68 dB
PL2W        10.90985775 W
PL12W        0.29499799 W
PL13W        0.14784923 W
SFO2       400.1716007 MHz
SI                32768
SF         100.6228303 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                1.40
```



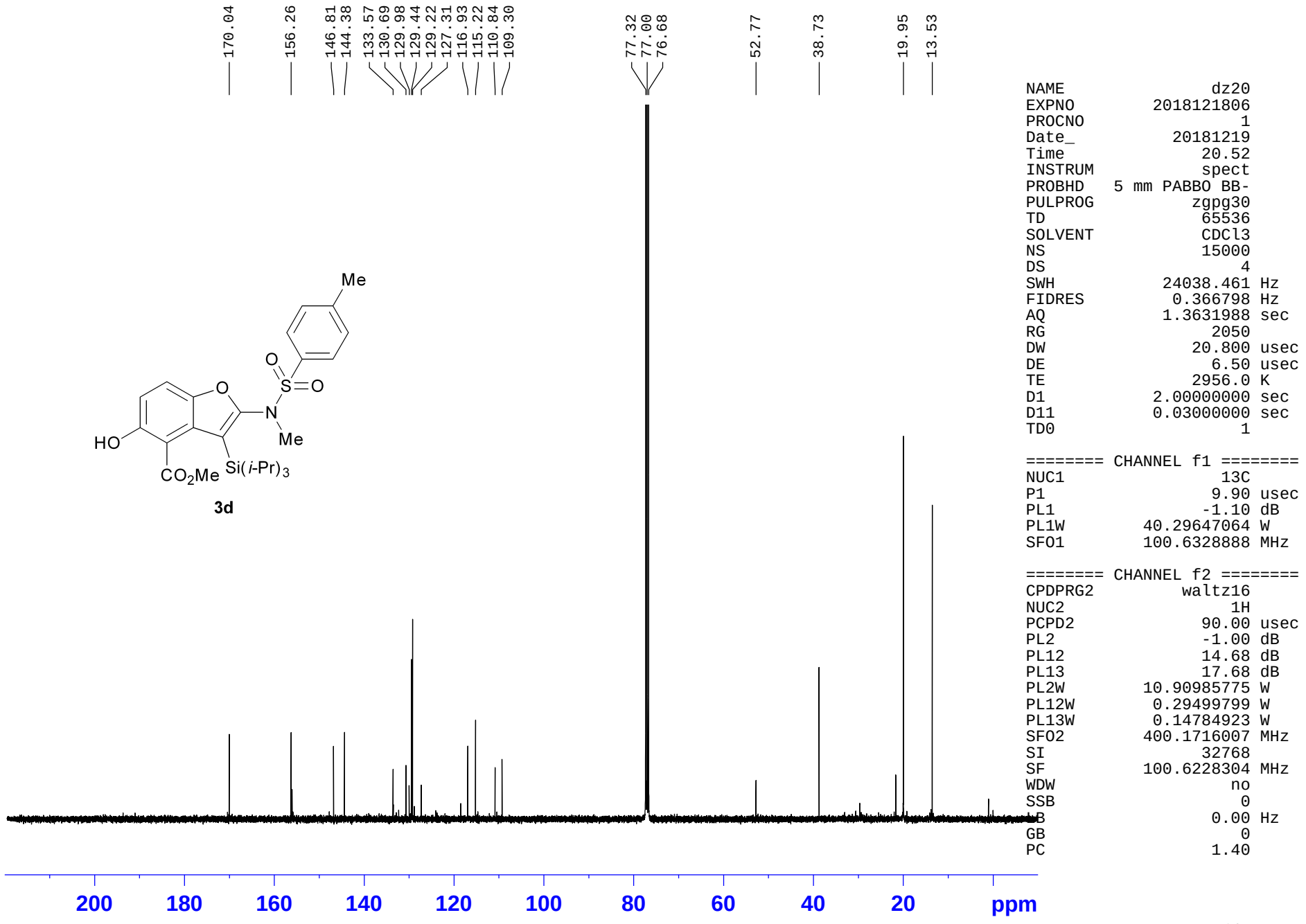
NAME	dz02
EXPNO	2018113001
PROCNO	1
Date_	20181130
Time	21.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	144
DW	60.800 usec
DE	6.50 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.80 usec
PL1	-1.00 dB
PL1W	10.90985775 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700153 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



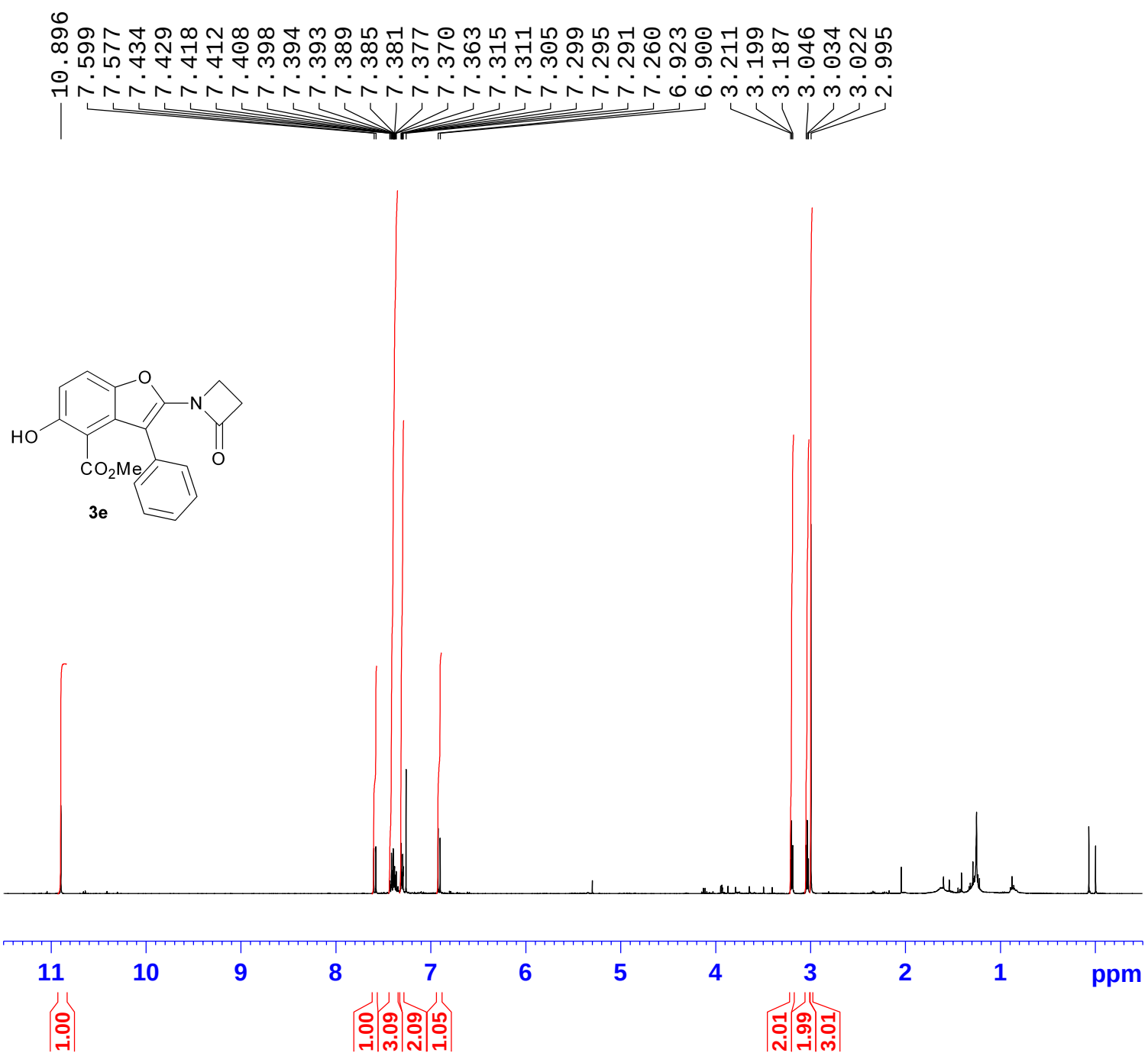
NAME	dz02
EXPNO	2018113002
PROCNO	1
Date_	20181130
Time	21.25
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1093
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	295.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-1.10 dB
PL1W	40.29647064 W
SFO1	100.6328888 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	14.68 dB
PL13	17.68 dB
PL2W	10.90985775 W
PL12W	0.29499799 W
PL13W	0.14784923 W
SFO2	400.1716007 MHz
SI	32768
SF	100.6228319 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40



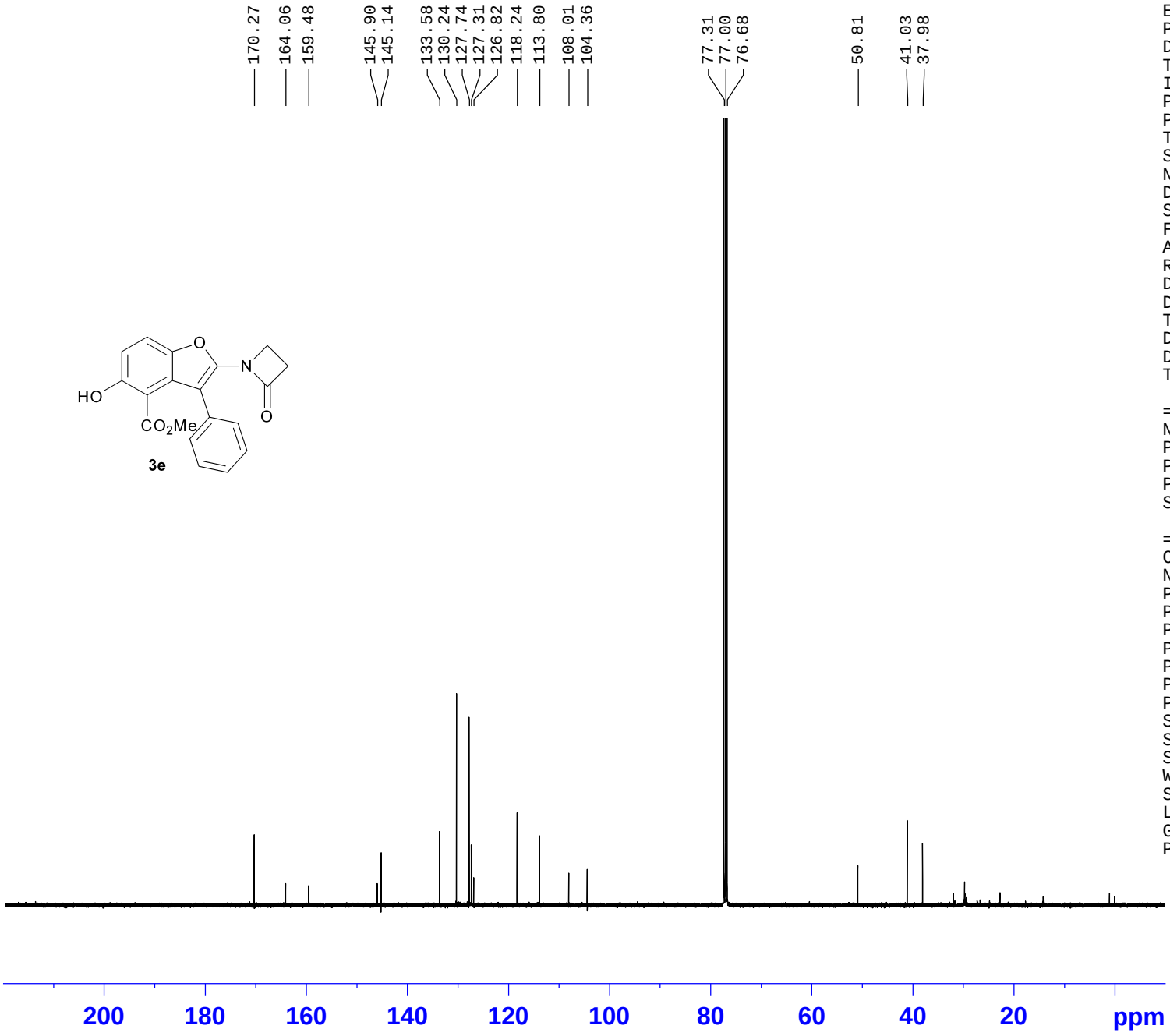

```
NAME                dz20
EXPNO         2018121801
PROCNO                 1
Date_           20181219
Time               14.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                   228
DW                60.800 usec
DE                 6.50 usec
TE               2952.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.80 usec
PL1                -1.00 dB
PL1W        10.90985775 W
SFO1        400.1724712 MHz
SI                 32768
SF          400.1700158 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 1.00
```



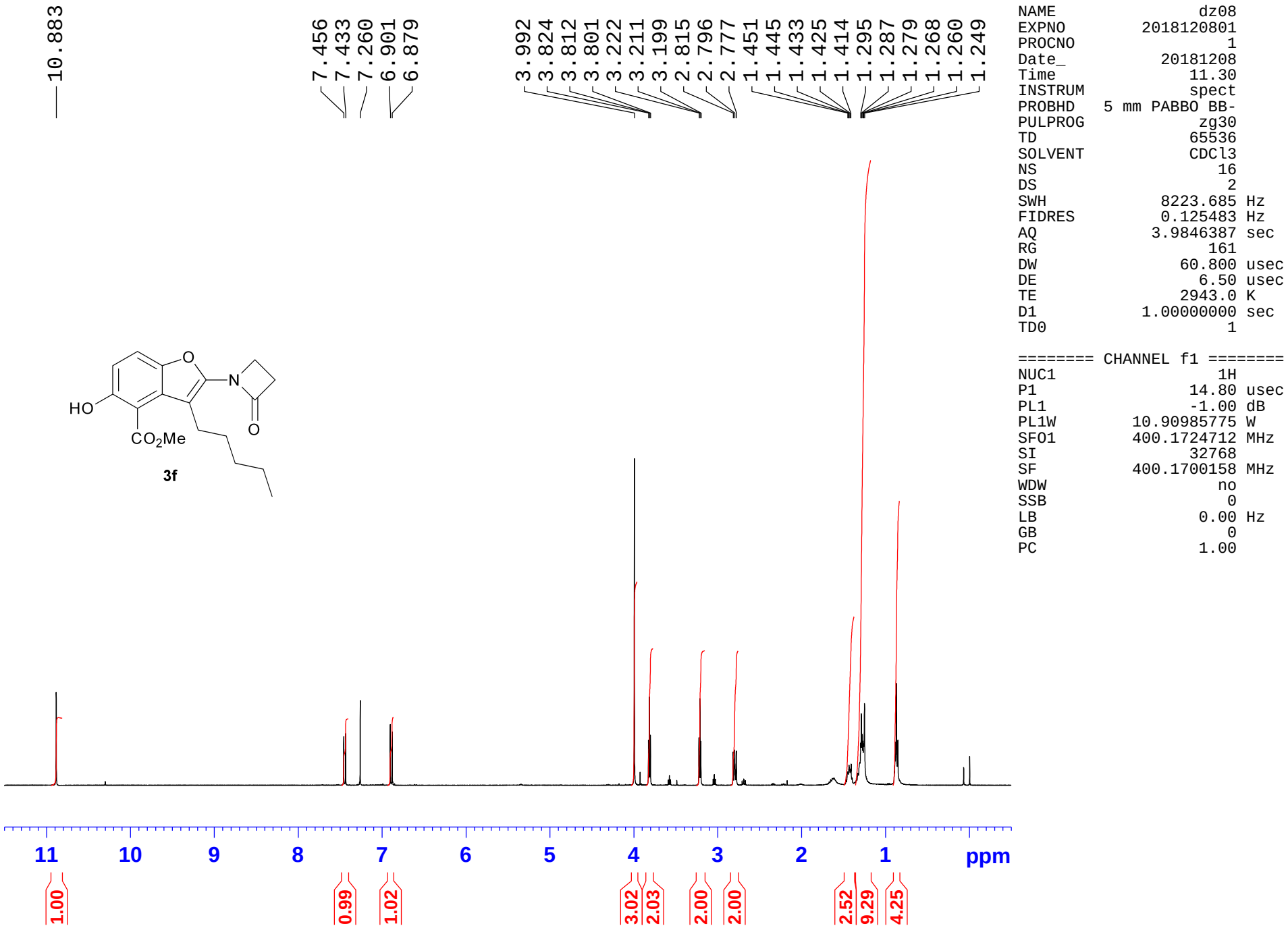

```
NAME                dz20
EXPNO         2018121806
PROCNO                 1
Date_           20181219
Time               20.52
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 15000
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                2956.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -1.10 dB
PL1W        40.29647064 W
SFO1        100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               14.68 dB
PL13               17.68 dB
PL2W        10.90985775 W
PL12W        0.29499799 W
PL13W        0.14784923 W
SFO2        400.1716007 MHz
SI                 32768
SF          100.6228304 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```



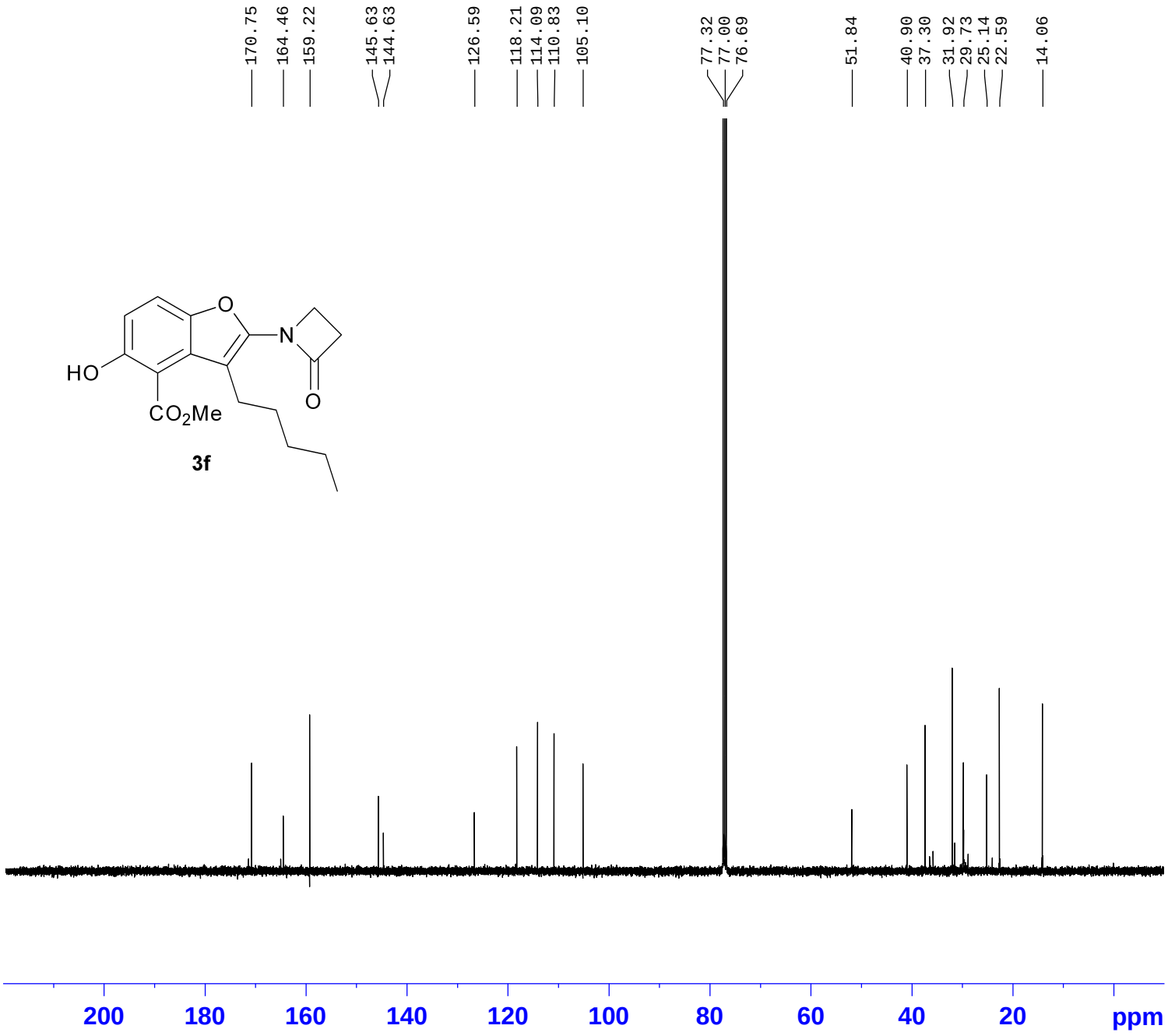
NAME	dz05
EXPNO	2018120501
PROCNO	1
Date_	20181205
Time	17.06
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	228
DW	60.800 usec
DE	6.50 usec
TE	2958.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.80 usec
PL1	-1.00 dB
PL1W	10.90985775 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700158 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



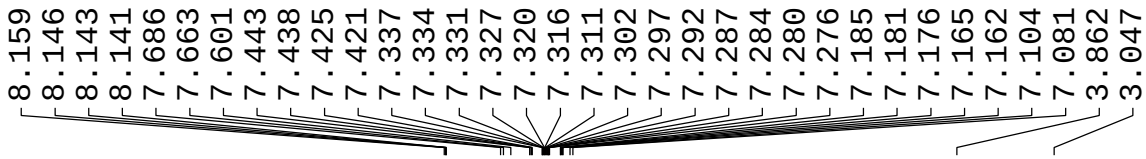

```
NAME                dz05
EXPNO         2018120502
PROCNO                 1
Date_           20181205
Time               17.13
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 10000
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                2959.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                 -1.10 dB
PL1W         40.29647064 W
SFO1         100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                14.68 dB
PL13                17.68 dB
PL2W         10.90985775 W
PL12W         0.29499799 W
PL13W         0.14784923 W
SFO2         400.1716007 MHz
SI                 32768
SF           100.6228318 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```

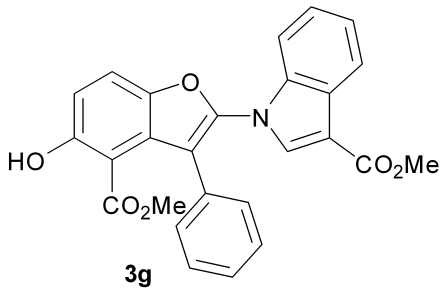


NAME	dz08
EXPNO	2018120801
PROCNO	1
Date_	20181208
Time	11.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	161
DW	60.800 usec
DE	6.50 usec
TE	2943.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.80 usec
PL1	-1.00 dB
PL1W	10.90985775 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700158 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



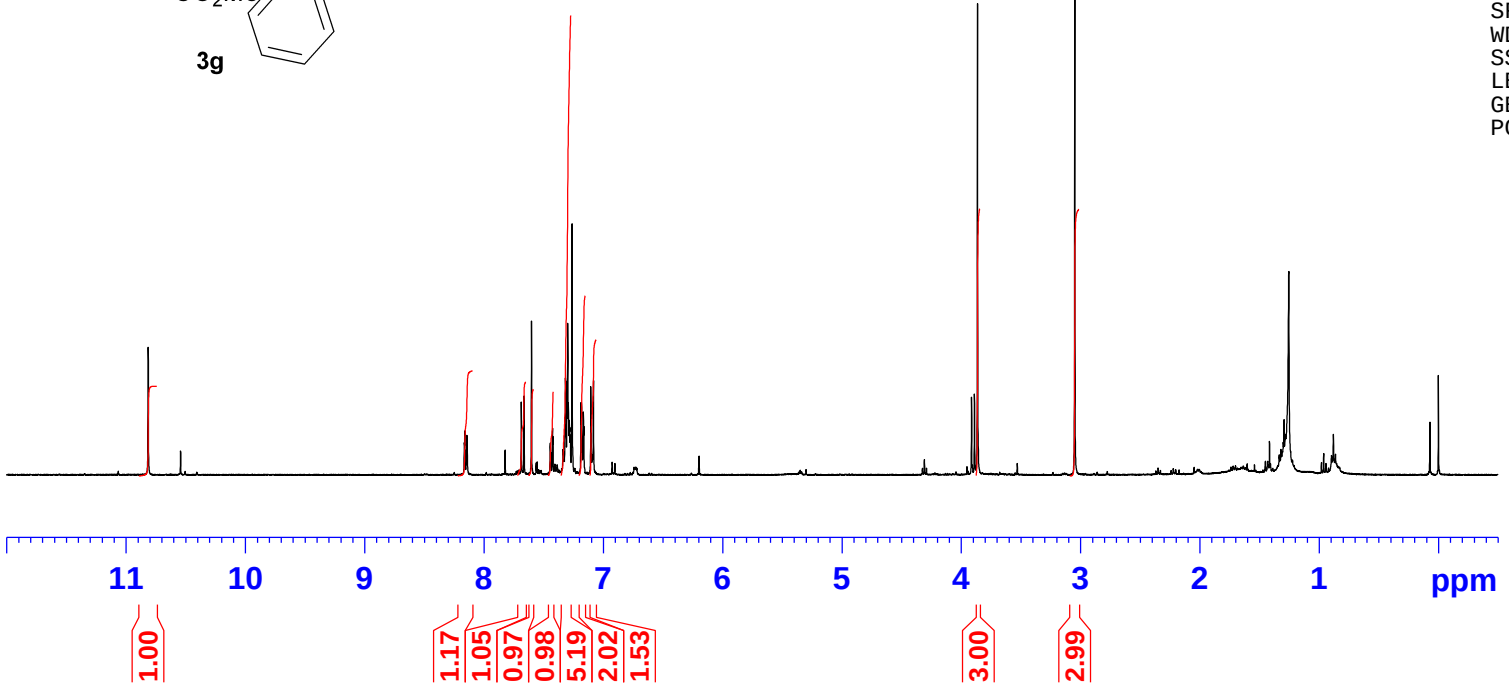

```
NAME                dz08
EXPNO         2018120802
PROCNO                 1
Date_           20181208
Time                12.00
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3794
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                2943.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.90 usec
PL1               -1.10 dB
PL1W        40.29647064 W
SFO1       100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               14.68 dB
PL13               17.68 dB
PL2W        10.90985775 W
PL12W        0.29499799 W
PL13W        0.14784923 W
SFO2       400.1716007 MHz
SI                 32768
SF         100.6228319 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.40
```

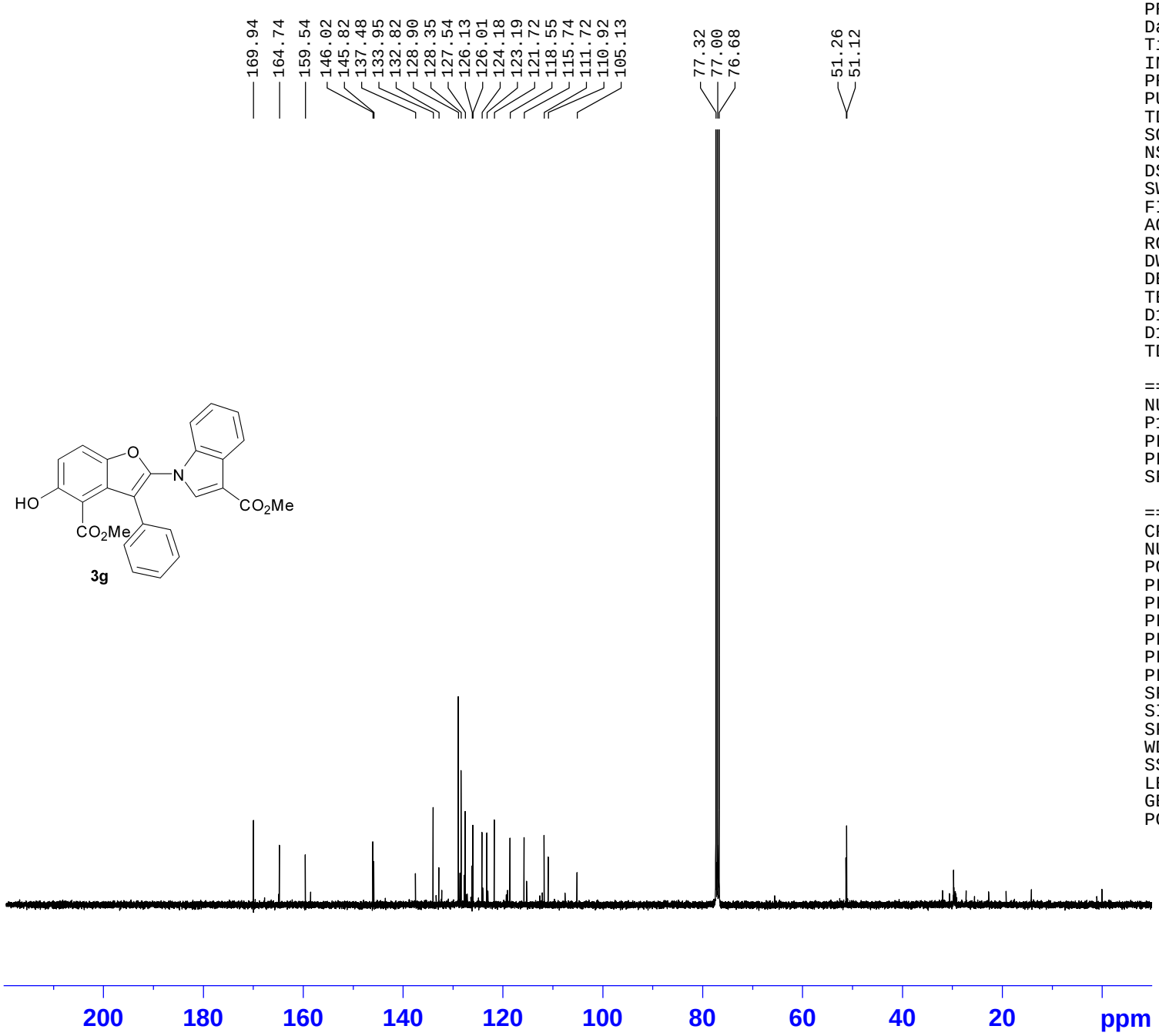




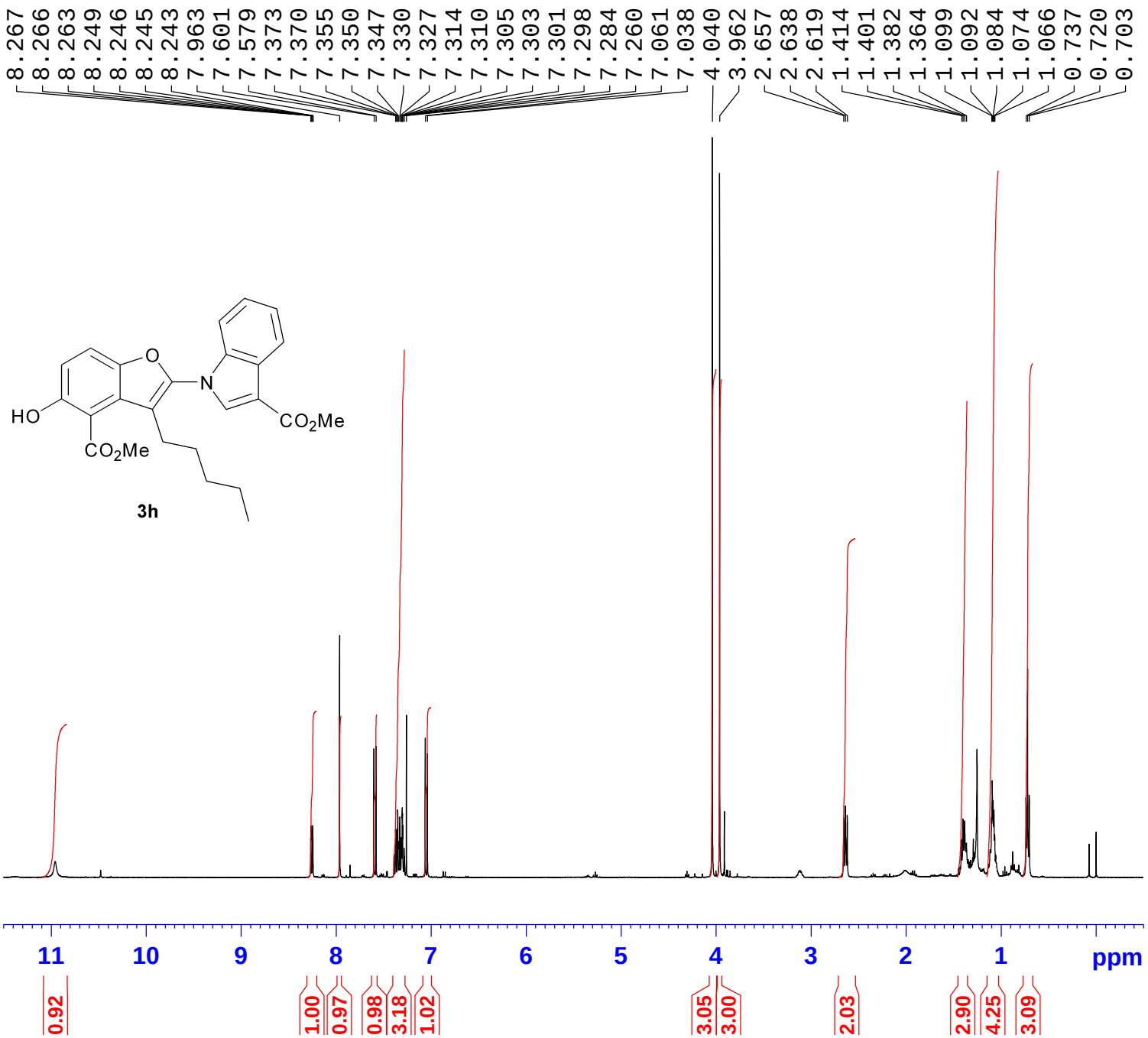
3g

```
NAME                dz10
EXPNO         2018121301
PROCNO                 1
Date_           20181213
Time               11.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   228
DW                60.800 usec
DE                 6.50 usec
TE               2944.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.80 usec
PL1                -1.00 dB
PL1W         10.90985775 W
SFO1         400.1724712 MHz
SI                 32768
SF           400.1700156 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                  1.00
```

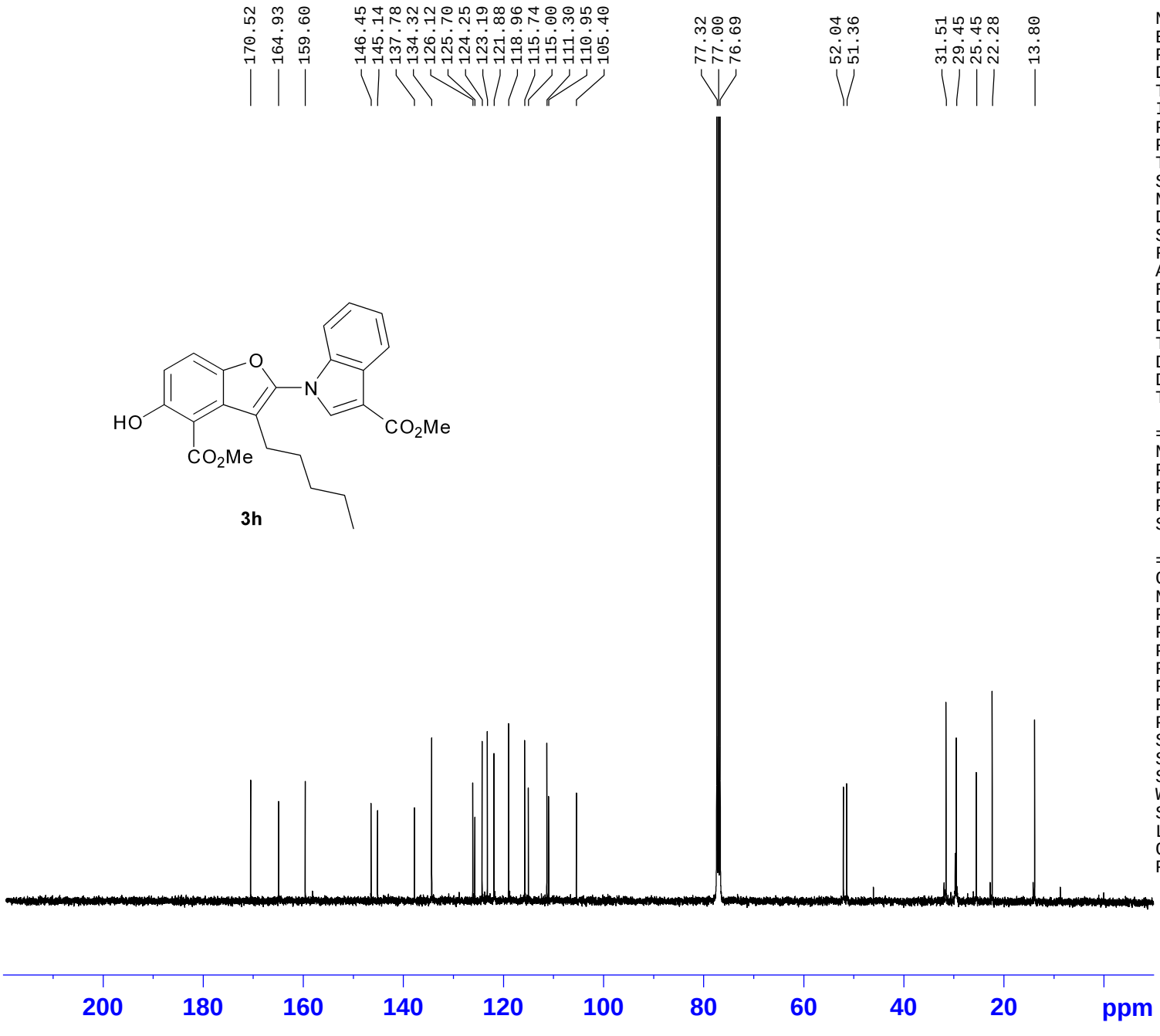





```
NAME                dz10
EXPNO         2018121302
PROCNO                 1
Date_           20181213
Time               15.25
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10000
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   2050
DW                 20.800 usec
DE                  6.50 usec
TE                2956.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.90 usec
PL1                 -1.10 dB
PL1W         40.29647064 W
SFO1         100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                14.68 dB
PL13                17.68 dB
PL2W         10.90985775 W
PL12W         0.29499799 W
PL13W         0.14784923 W
SFO2         400.1716007 MHz
SI                  32768
SF           100.6228311 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40
```



NAME	dz12
EXPNO	2018121301
PROCNO	1
Date_	20181213
Time	11.43
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	181
DW	60.800 usec
DE	6.50 usec
TE	2946.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.80 usec
PL1	-1.00 dB
PL1W	10.90985775 W
SFO1	400.1724712 MHz
SI	32768
SF	400.1700158 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00




```
NAME                dz12
EXPNO         2018121302
PROCNO                 1
Date_           20181213
Time               11.56
INSTRUM            spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3266
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW               20.800 usec
DE                6.50 usec
TE              2952.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.90 usec
PL1                -1.10 dB
PL1W        40.29647064 W
SFO1       100.6328888 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               14.68 dB
PL13               17.68 dB
PL2W        10.90985775 W
PL12W        0.29499799 W
PL13W        0.14784923 W
SFO2       400.1716007 MHz
SI                 32768
SF         100.6228319 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```