Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

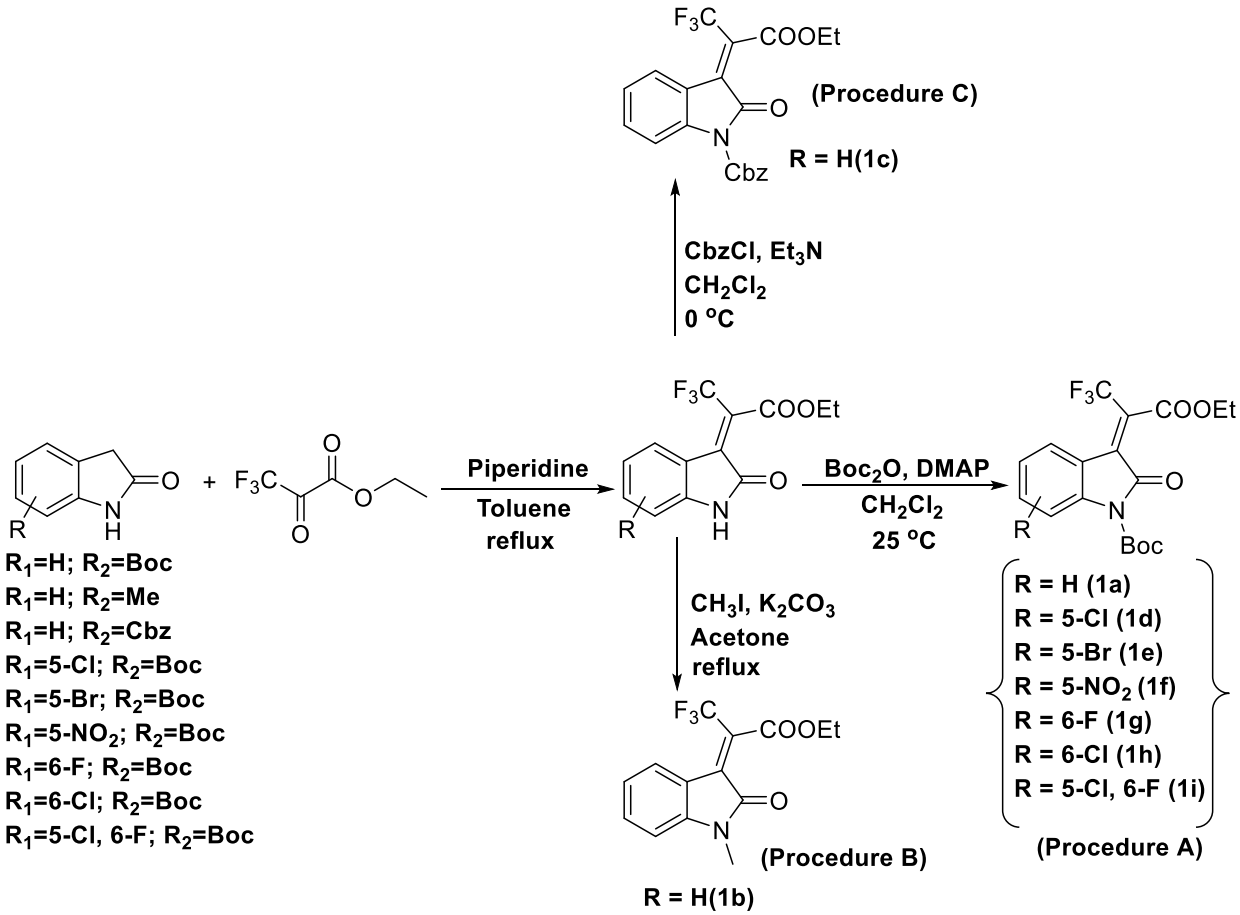
Iodine assisted synthesis of CF_3 appended spirodihydrofuryl/cyclopropyloxindoles by changing the active methylene sources

Thirupathi Reddy Penjarla,[a,b] Maheshwar Kundarapu,[b] R. Krishnan[a] and Anupam Bhattacharya[a]*

[a]Department of Chemistry, Birla Institute of Technology and Science-Pilani (Hyderabad Campus), Hyderabad-500078, India.
[b]Department of Medicinal Chemistry, GVK Biosciences Pvt. Ltd., Survey Nos: 125 (part) & 126**,** IDA Mallapur, Hyderabad 500076, India
E-mail: anupam@hyderabad.bits-pilani.ac.in; Fax: +91-40-66303998; Tel: +91-40-66303522.

Contents:

Scheme for the synthesis of oxindole derivatives



General procedure (A): Ethyl trifluoropyruvate (11.27 mmol) and piperidine (22.55 mmol) were added to a suspension of appropriate oxindole (7.5 mmol) in toluene (10 ml), and the solution was refluxed at 110°C for 4 h. It was then cooled to room temperature, and volatiles were evaporated. The resulting solid was dissolved in CH_2Cl_2(15 ml) and $(Boc)_2O$ (9.02 mmol) and DMAP (0.037 mmol) were added to it. The resulting mixture was left to stir at the room temperature for 2 hours. On completion of the reaction, it was quenched with water(15 ml) and extracted with CH_2Cl_2 (2 x 20 ml). Organic layers were then combined, washed with brine (10 ml) and dried over Na_2SO_4. It was subsequently evaporated to

afford the crude compound. The purified product was finally obtained using silica gel column chromatography by eluting with 10-20% EtOAc/hexanes to give the desired compounds.

Characterisation data for compounds 1a, 1d-1i

(Z)-tert-butyl 3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-2-oxoindoline-1-carboxylate (1a).(51%)**;** ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.92-7.9 (d, J = 8.4 Hz, 1H), 7.8-7.78 (d, J = 8 Hz, 1H), 7.94-7.45 (m, 1H), 7.22-7.18 (m, 1H), 4.49 – 4.43 (q, J = 7.2 Hz, 2H), 1.6 (s, 9H), 1.41 – 1.39 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.1, 162.7, 148.2, 133.42, 131.1, 127.1, 126.1, 125 (q, J = 273 Hz), 124.8, 117.3, 115.4, 85.25, 62.9, 27.9, 13.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -57.8 (s); MS-ESI (+) m / z: 386.13 $[M + H]^+$.

(Z)-tert-butyl 5-chloro-3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-2-oxoindoline-1-carboxylate(1d). (75%) ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91-7.89 (d, J = 8.8 Hz, 1H), 7.74 (s, 1H), 7.46-7.43 (dd, J = 8.8, 2 Hz, 1H), 4.49 – 4.44 (q, J = 7.2 Hz, 2H), 1.61 (s, 9H), 1.33 – 1.29 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.4, 162.2 (d, J = 3 Hz), 148, 140.3, 133.2, 130.2 (t, J = 4Hz), 128 (t, J = 37 Hz), 126 (m), 122.2 (q, J = 274 Hz), 118.6, 116.7, 85.6, 63, 27.9, 13.69; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.2 (s);MS-ESI (+) m / z: 420.1 $[M + H]^+$.

(Z)-tert-butyl 5-bromo-3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-2-oxoindoline-1-carboxylate (1e). (73%) ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.88-7.83 (m, 2H), 7.6-7.58 (dd, J = 8.8, 2 Hz, 1H), 4.49 – 4.44 (q, J = 6.8 Hz, 2H), 1.61 (s, 9H), 1.4 – 1.37 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.4, 162.2 (d, J = 3 Hz), 148, 140.8, 136, 130.2 (t, J = 4Hz), 128.8 (q, J = 6 Hz), 128.4, 119.5 (q, J = 274 Hz), 119, 117.9, 117, 85.6, 63.1, 27.9, 13.69; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.2 (s);MS-ESI (+) m / z: 464.1 $[M + H]^+$.

(Z)-tert-butyl 3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-5-nitro-2-oxoindoline-1-carboxylate (1f). (68%) ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.68-8.67 (d, J = 2 Hz, 1H), 8.4-8.38 (dd, J = 8.8, 2 Hz, 1H), 8.17-8.14 (d, J = 8.8 Hz, 1H), 4.52 – 4.46 (q, J = 7.2 Hz, 2H), 1.61 (s, 9H), 1.4 – 1.37 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.1, 161.7, 147.7, 146.1, 144.7, 130 (m), 128.6, 119.3 (q, J = 273 Hz), 117.8, 115.7, 86.6, 63.3, 27.9, 13.69; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.2 (s);MS-ESI (+) m / z: 431.2 $[M + H]^+$.

(Z)-tert-butyl 3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-6-fluoro-2-oxoindoline-1-carboxylate (1g). (71%)^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.69-7.66 (q, J = 5.6 Hz, 1H), 6.77-6.72 (m, 1H), 6.63-6.6 (q, J = 6Hz, 1H), 4.45 – 4.43 (q, J = 7.2 Hz, 2H), 1.5 (s, 9H), 1.4 – 1.36 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.96, 164.4, 162.67, 145, 131.49, 128.9, 126.4, 122.57 (q, J = 273 Hz), 113.9, 110.3, 110.1, 99.4, 99.2, 85.25, 62.9, 27.9, 13.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.4 (s), -102 (m); MS-ESI (+) m/z: 404.1 $[M + H]^+$.

(Z)-tert-butyl 6-chloro-3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-2-oxoindoline-1-carboxylate(1h). (68%) 8.01 (d, J = 2 Hz, 1H), 7.71-7.69 (d, J = 8.4 Hz, 1H), 7.2-7.18 (dd, J = 8.4 Hz, 1H), 4.49 – 4.43 (q, J = 7.2 Hz, 2H), 1.62 (s, 9H), 1.4 – 1.37 (t, J = 7.2 Hz, 3H); ^{13}C

NMR (100 MHz, $CDCl_3$) δ: 162.7, 162.4 (d, J = 3 Hz), 148, 142.6, 139.6, 130 (d, J = 4.1 Hz), 127 (m), 125.1, 122.4 (q, J = 274 Hz), 116.2, 115.7 (d, J = 1 Hz), 85.7, 63, 27.9, 13.67; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.3 (s);MS-ESI (+) m / z: 420.1 $[M + H]^+$.

(Z)-tert-butyl 5-chloro-3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-6-fluoro-2-oxoindoline-1-carboxylate(1i).(70%) ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.12-8.1 (d, J = 6.5 Hz, 1H), 7.58-7.56 (d, J = 8.5 Hz, 1H), 4.49 – 4.43 (m, 2H), 1.62 (s, 9H), 1.4 – 1.37 (t, J = 7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.4, 162.2 (d, J = 3 Hz), 148, 140.8, 136, 130.2 (t, J = 4Hz), 128.8 (q, J = 6 Hz), 128.4, 119.5 (q, J = 274 Hz), 119, 117.9, 117, 85.6, 63.1, 27.9, 13.69; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.2 (s);MS-ESI (+) m / z: 438.09 $[M + H]^+$.

General procedure (B): To a stirred solution of (Z)-ethyl 3,3,3-trifluoro-2-(2-oxoindolin-3-ylidene)propanoate (0.7 mmol) in acetone (5 ml) was added K_2CO_3 (2.1 mmol) followed by MeI (2.1 mmol) and the reaction was heated under reflux for 6 hours. The reaction mixture was then cooled to room temperature, and volatiles evaporated under reduced pressure. The residue obtained was stirred in water (10 ml) and solids thus obtained were filtered and dried to obtain compound (Z)-ethyl 3,3,3-trifluoro-2-(1-methyl-2-oxoindolin-3-ylidene)propanoate (1b).

Yield:(185 mg, 70%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71-7.69 (d, J = 8 Hz, 1H), 7.4-7.38 (m, 1H), 7.08-7.04 (m, 1H), 6.82-6.8 (d, J = 7.6 Hz, 1H), 4.49 – 4.42 (m, 2H), 3.19 (s, 3H), 1.4 – 1.3 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.9, 162.7 (m), 145.7, 133.1, 132, 127.1(m), 126.1(m), 123, 122 (q, J = 273 Hz), 117.2, 109.02, 62.7, 26, 13.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.2 (s); MS-ESI (+) m / z: 300.1 $[M + H]^+$.

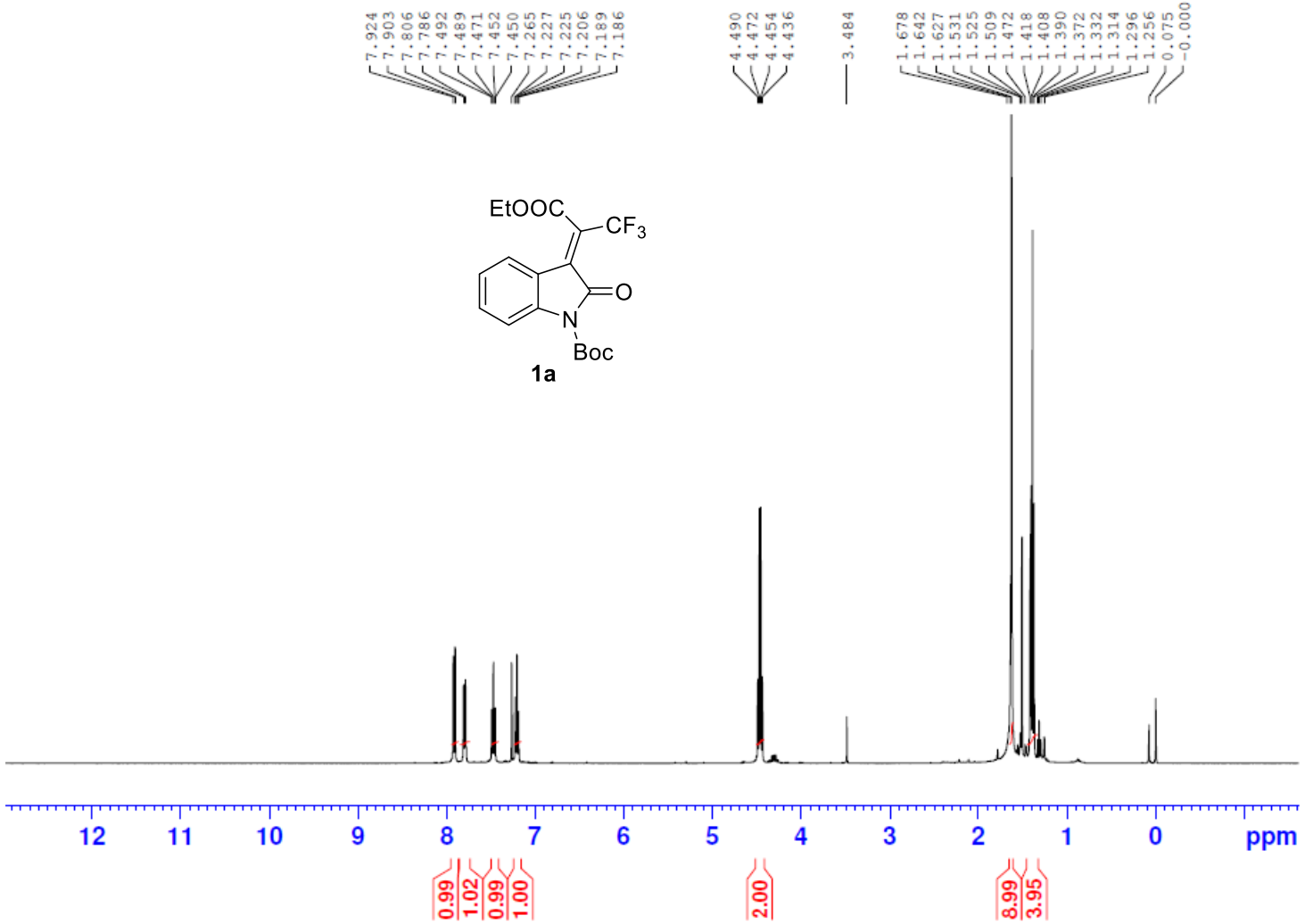
General procedure (C): CbzCl (1.05 mmol) was added to a stirred solution of (Z)-ethyl 3,3,3-trifluoro-2-(2-oxoindolin-3-ylidene)propanoate (0.7 mmol) and Et_2N (2.1 mmol) in CH_2Cl_2 (5 ml) at 0°C and the reaction was allowed to stir in the same temperature for 2 hours. On completion, the reaction mixture was warmed to room temperature and diluted with water (10 ml). The combined layer was subsequently extracted with CH_2Cl_2 (2 x 10 ml), and the pooled organic layers were washed with brine, dried over Na_2SO_4 and evaporated to afford the crude compound. It was further purified by column chromatography using silica gel as the solid phase and (15%EtOAc/hexanes as the mobile phase to give the desired compound(Z)-benzyl 3-(3-ethoxy-1,1,1-trifluoro-3-oxopropan-2-ylidene)-2-oxoindoline-1-carboxylate (1c).

Yield:(205 mg, 69%); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8-7.98 (d, J = 8 Hz, 1H), 8.2-8 (d, J = 7.6 Hz, 1H), 7.49-7.21 (m, 6H), 5.45 (s, 2H), 4.49 – 4.4 (q, J = 7.2 Hz, 2H), 1.41 – 1.37 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163, 162.5, 149.9, 141.4, 134.6, 133.6, 130.8, 128, 127, 126.2, 125.3, 119.7 (q, J = 273 Hz), 117.5, 115.6, 69, 63, 13.73; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -60.38 (s); MS-ESI (+) m / z: 420.2 $[M + H]^+$.

Entry	2 (eq)	base (eq)	Halogen source (eq)	Solvent	t	Yield (%)	dr
1	1.5	$NaHCO_3$ (2.5)	I_2 (2)	DMSO	1h	20	4:1
2	1.5	Na_2CO_3(2)	I_2 (2)	DMSO	1h	75	8:1
3	1.5	Na_2CO_3 (1.2)	I_2 (2)	DMSO	45 min	78	8:1
4	1	Na_2CO_3 (1.2)	I_2 (2)	DMSO	45 min	89	9:1
5	1	Na_2CO_3 (1.2)	I_2 (2)	EtOH	45 min	75	8:1
6	1.1	Na_2CO_3 (1.2)	I_2 (1.5)	THF	1 h	55	6:1
7	1	K_2CO_3(1.2)	I_2 (1.2)	Toluene	1 h	30	4:1
8	1.2	K_2CO_3(1.5)	I_2 (1.5)	DCM	1.5 h	40	6:1
9	1	Et_3N (2)	I_2 (1.5)	DMSO	2 h	0	0
10	1.1	DIPEA (2.5)	I_2 (1.5)	DMSO	2 h	0	0
11	1	Na_2CO_3 (1.2)	DAIB (1.5)	DMSO	2 h	0	0
12	1.2	DBU (1.2)	DAIB (2)	CH_3CN	2 h	0	0
13	1.2	K_2CO_3(1.2)	NIS (1.5)	DMSO	1 h	60	6:1
14	1	K_2CO_3(1.5)	NBS (2)	EtOH	45 min	50	7:1
15	1	Na_2CO_3 (1.2)	N-bromosaccharin (1.5)	EtOH	1.5 h	45	6:1
16	1.2	Na_2CO_3 (1.2)	NBS (1.5)	Et_2O	1.5	47	5:1
17	1	Na_2CO_3 (1.1)	-	DMSO	1 h	0	0

Screening table

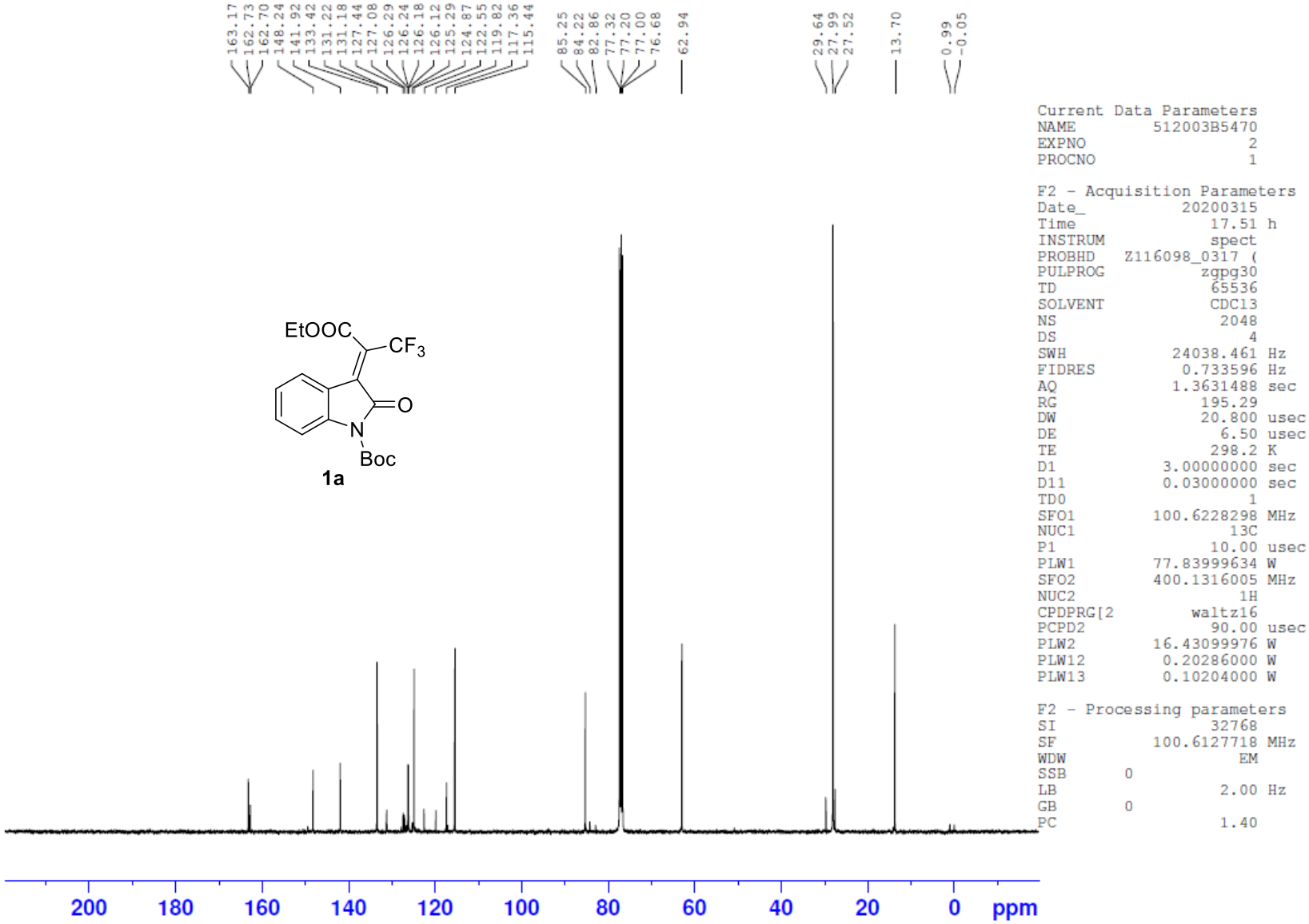
NOTE: All the equivalent calculations are based substrate **1**; reactions were carried out at room temperature; DAIB-diacetoxyiodo benzene; DIPEA-diisopropylethyl amine; DBU-1,8-diazabicyclo[5.4.0]undec-7-ene; NBS-*N*-bromosuccinamide; Y-isolated yield.



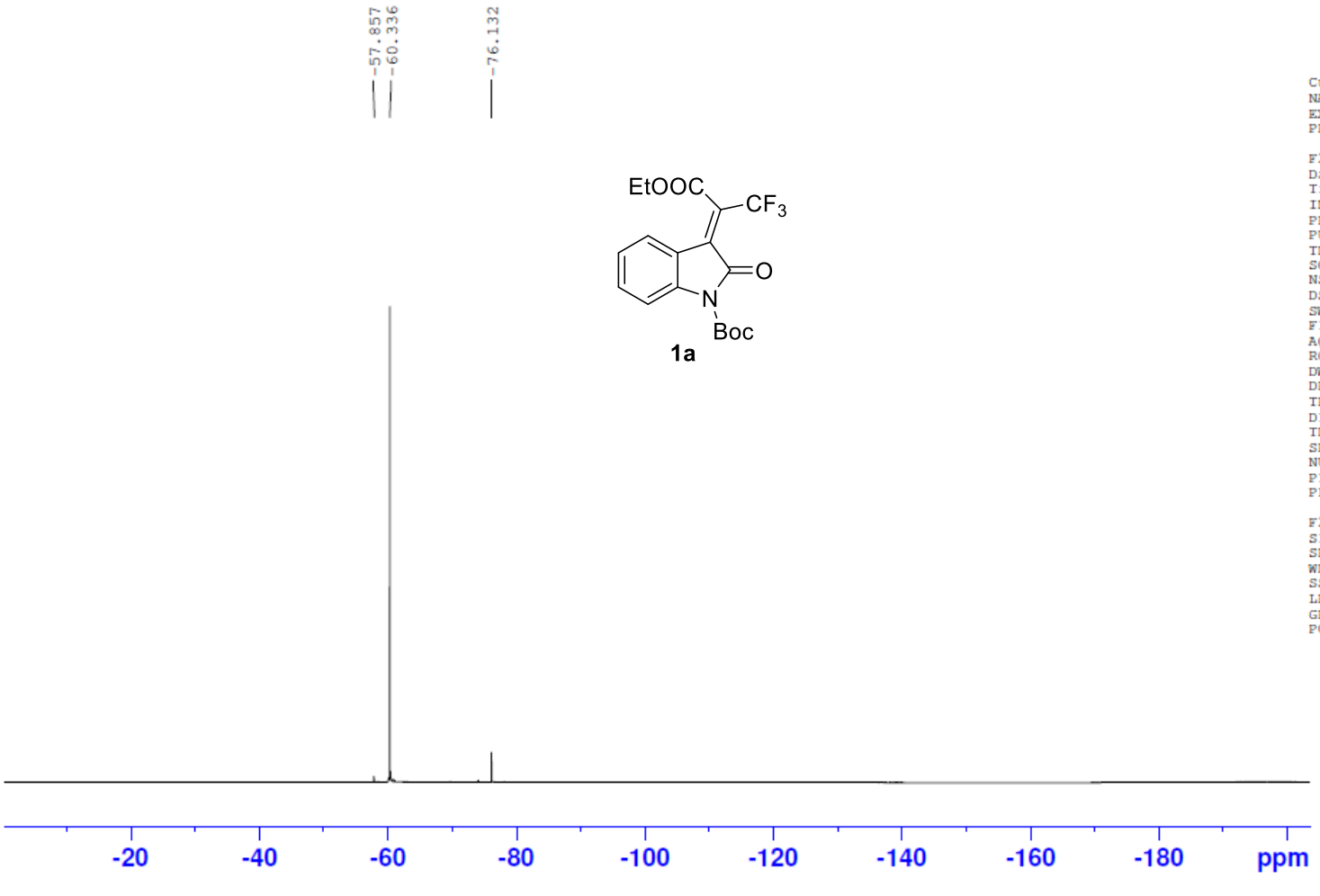

```
Current Data Parameters
NAME          512003B5470
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200315
Time               15.18 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 39.79
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324710 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300084 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-001

TP-11580-01-001

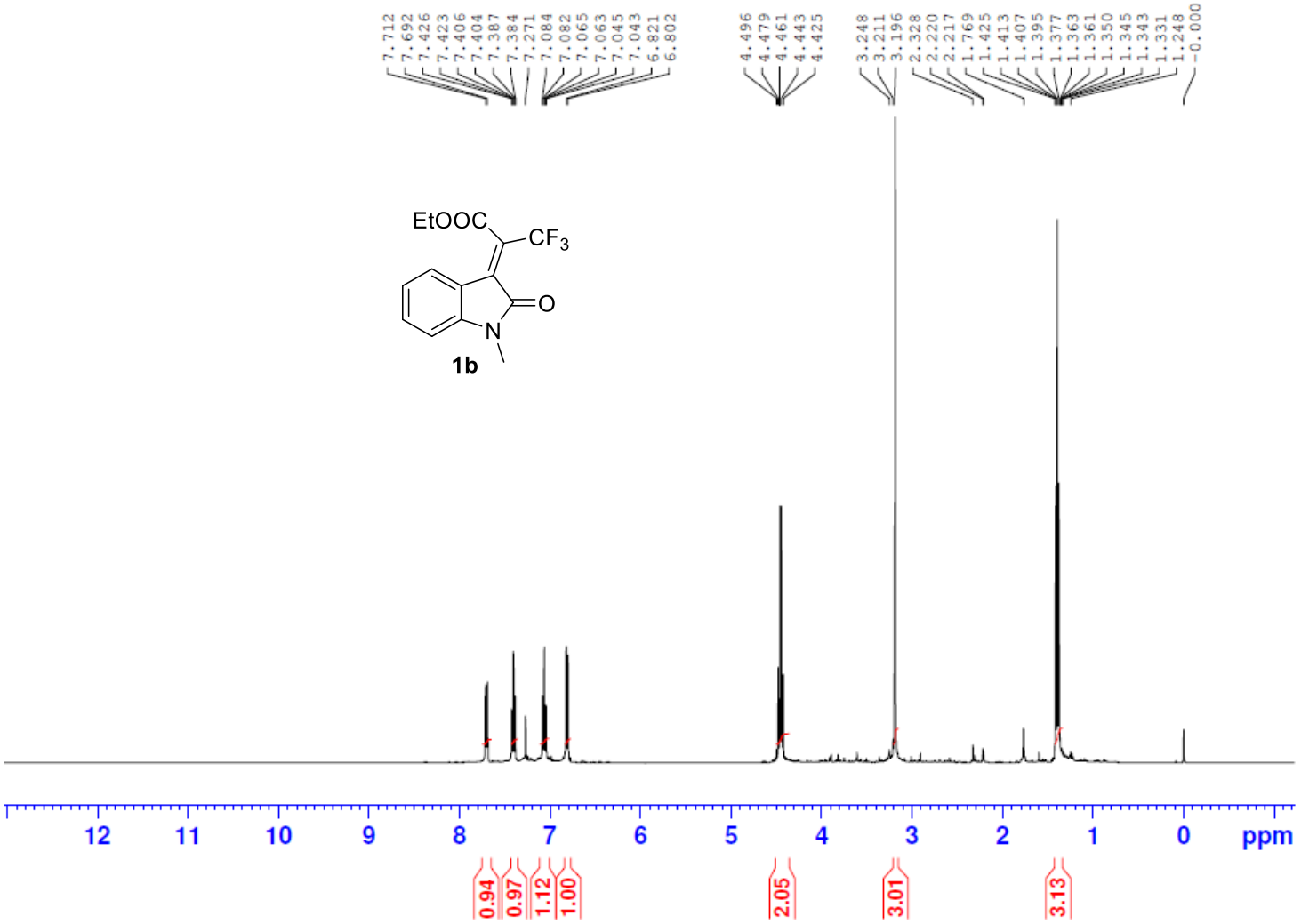



```
Current Data Parameters
NAME         512003B5470
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time               17.51 h
INSTRUM             spect
PROBHD   Z116098_0317 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                  2048
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                195.29
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                  13C
P1                10.00 usec
PLW1          77.83999634 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                 32768
SF            100.6127718 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME         512003B5470
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200316
Time                9.20 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          1.362392 Hz
AQ              0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          376.4607164 MHz
NUC1                  19F
P1                 18.00 usec
PLW1          19.85499954 W

F2 - Processing parameters
SI                 65536
SF            376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

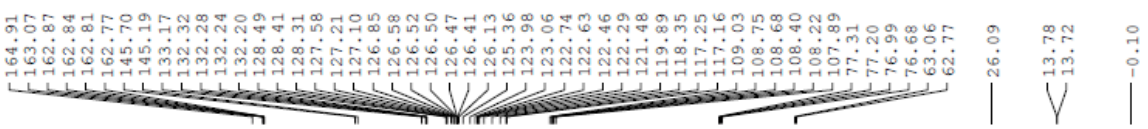
ANL-MCL5-NMR-001

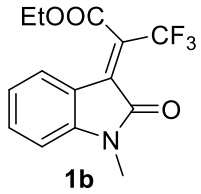



```
Current Data Parameters
NAME         512003B5467
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time              20.31 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                 31.51
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324710 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300057 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```

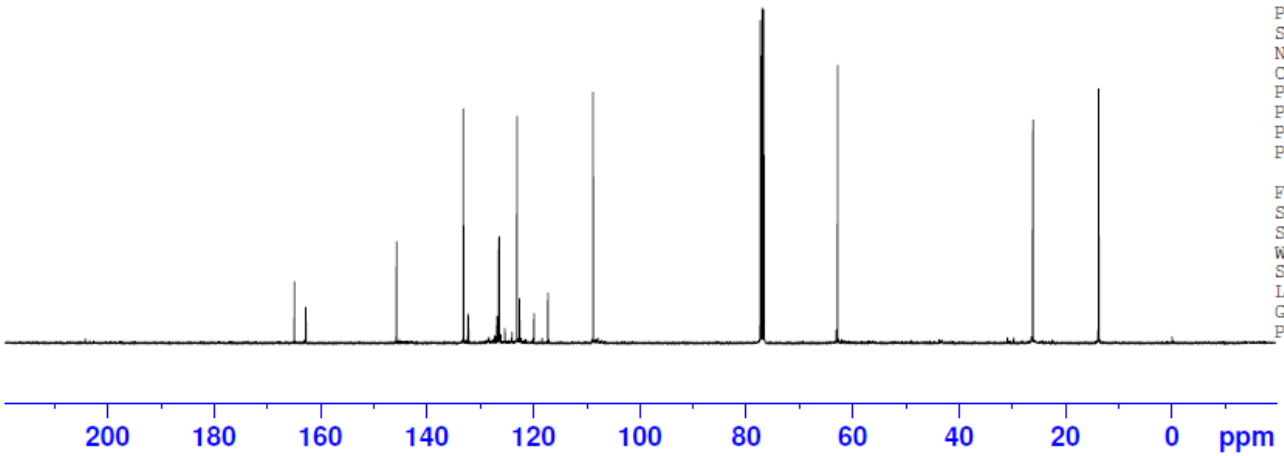
ANL-MCL5-NMR-001

TP-11580-01-NMe

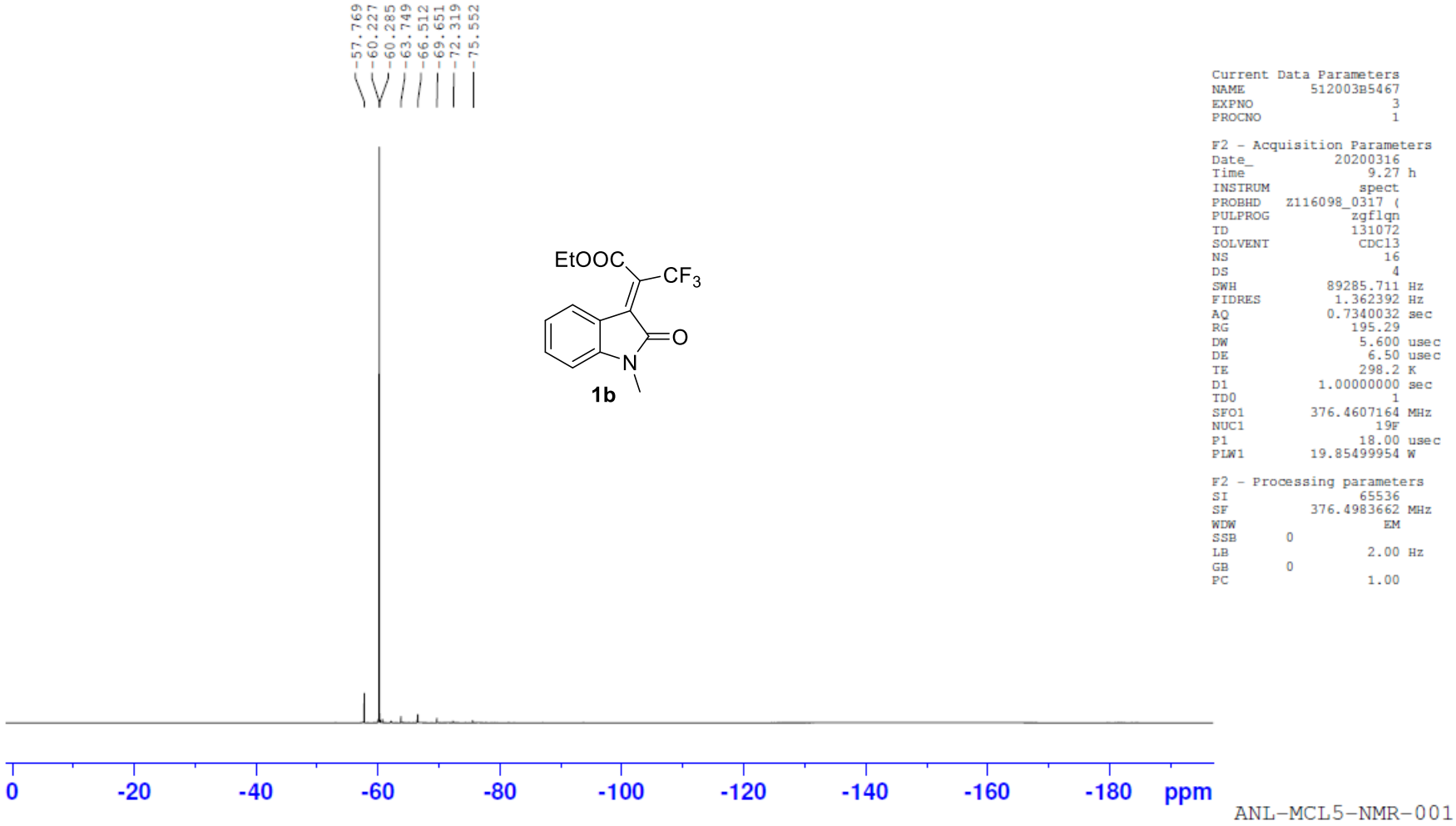




1b

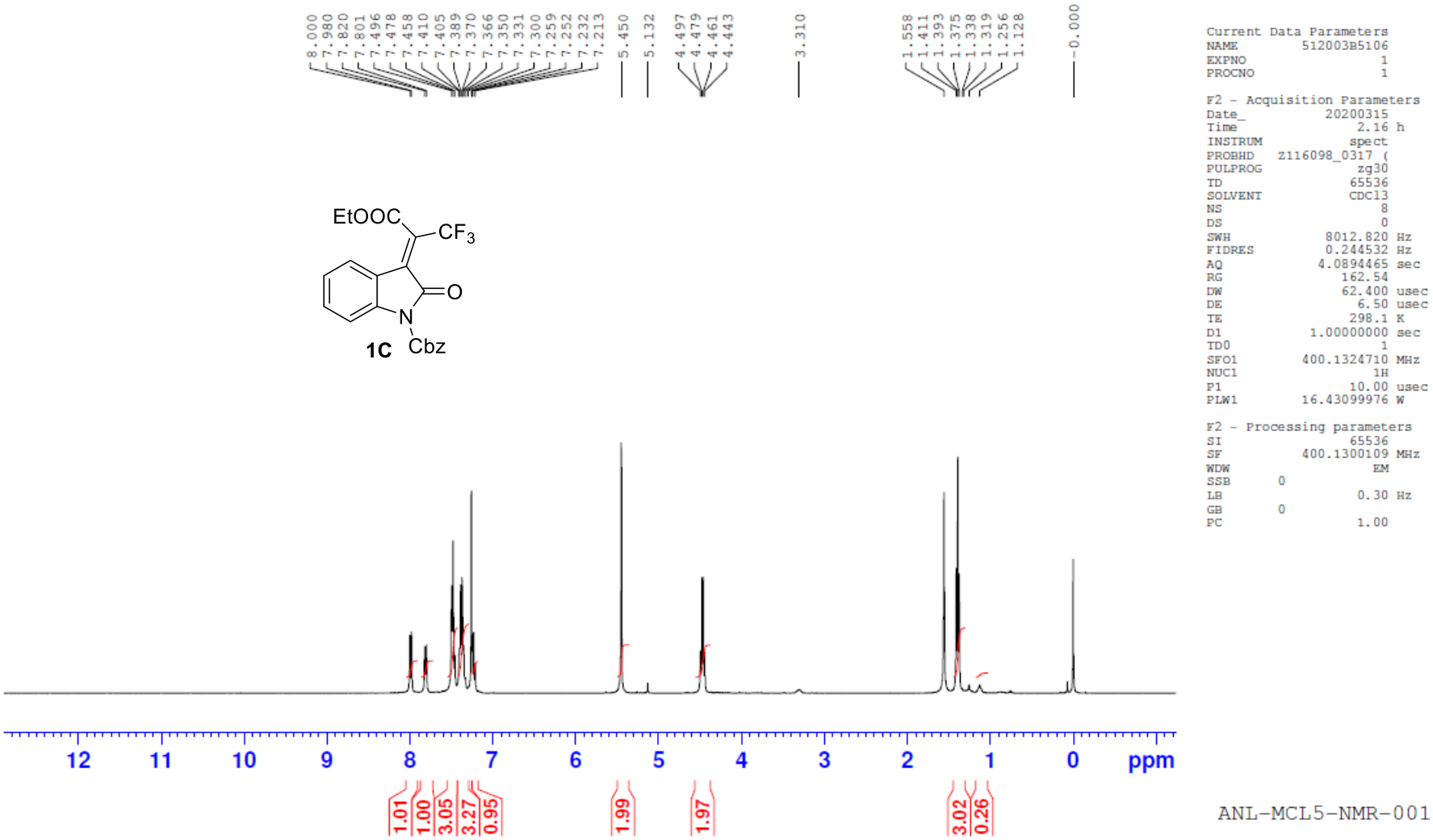



```
Current Data Parameters
NAME         512003B5467
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time              23.04 h
INSTRUM             spect
PROBHD   Z116098_0317 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               195.29
DW               20.800 usec
DE                6.50 usec
TE                298.2 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6228298 MHz
NUC1                13C
P1                10.00 usec
PLW1        77.83999634 W
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        16.43099976 W
PLW12        0.20286000 W
PLW13        0.10204000 W

F2 - Processing parameters
SI                32768
SF          100.6127762 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```




```
Current Data Parameters
NAME         512003B5467
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200316
Time                9.27 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG          zgflqn
TD               131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          1.362392 Hz
AQ              0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          376.4607164 MHz
NUC1                  19F
P1                 18.00 usec
PLW1          19.85499954 W

F2 - Processing parameters
SI                 65536
SF            376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

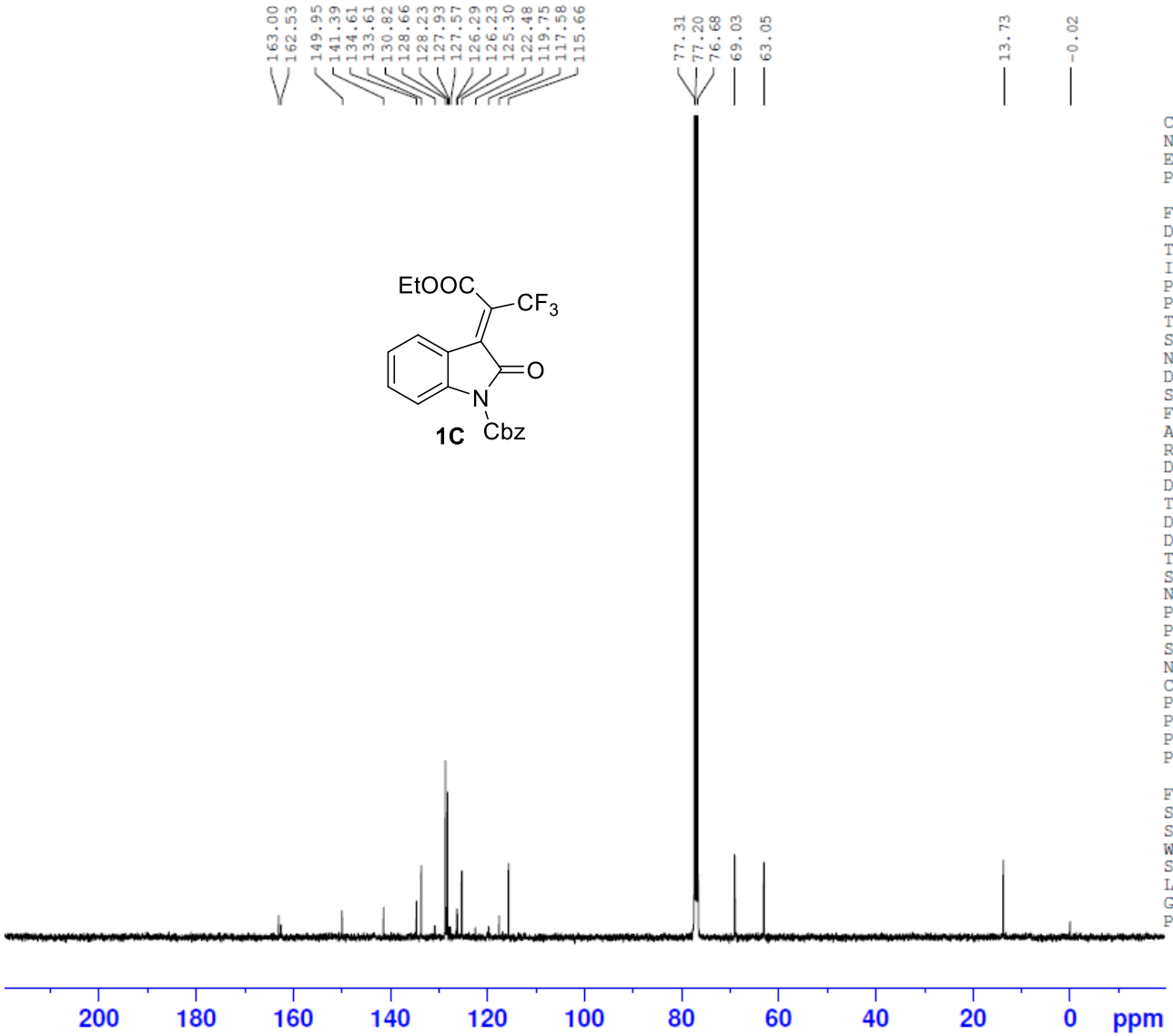
ANL-MCL5-NMR-001



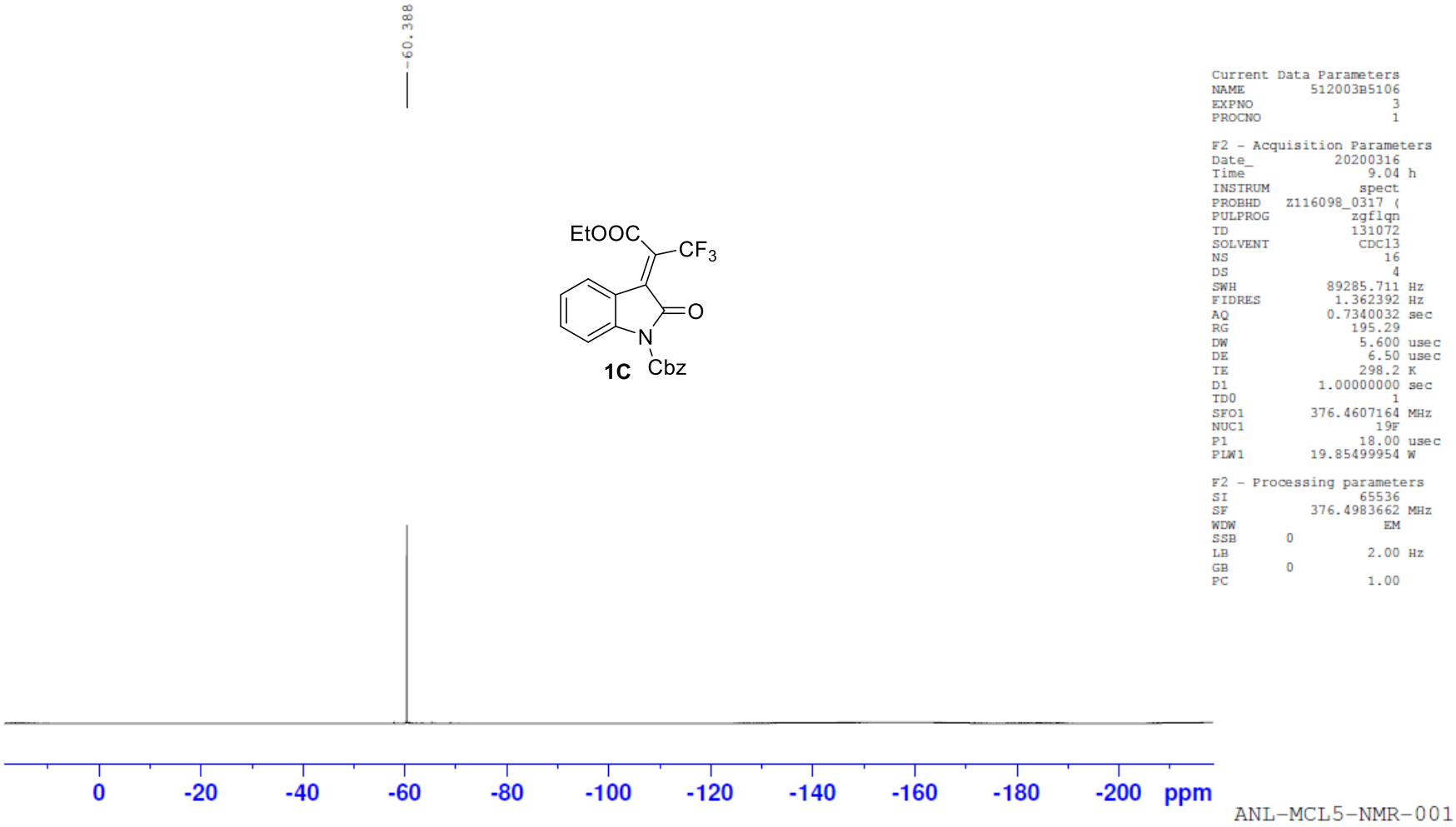

```
Current Data Parameters
NAME         512003B5106
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time                2.16 h
INSTRUM            spect
PROBHD    Z116098_0317 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ              4.0894465 sec
RG                162.54
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1324710 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300109 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-001

TP-11580-001-CBZ

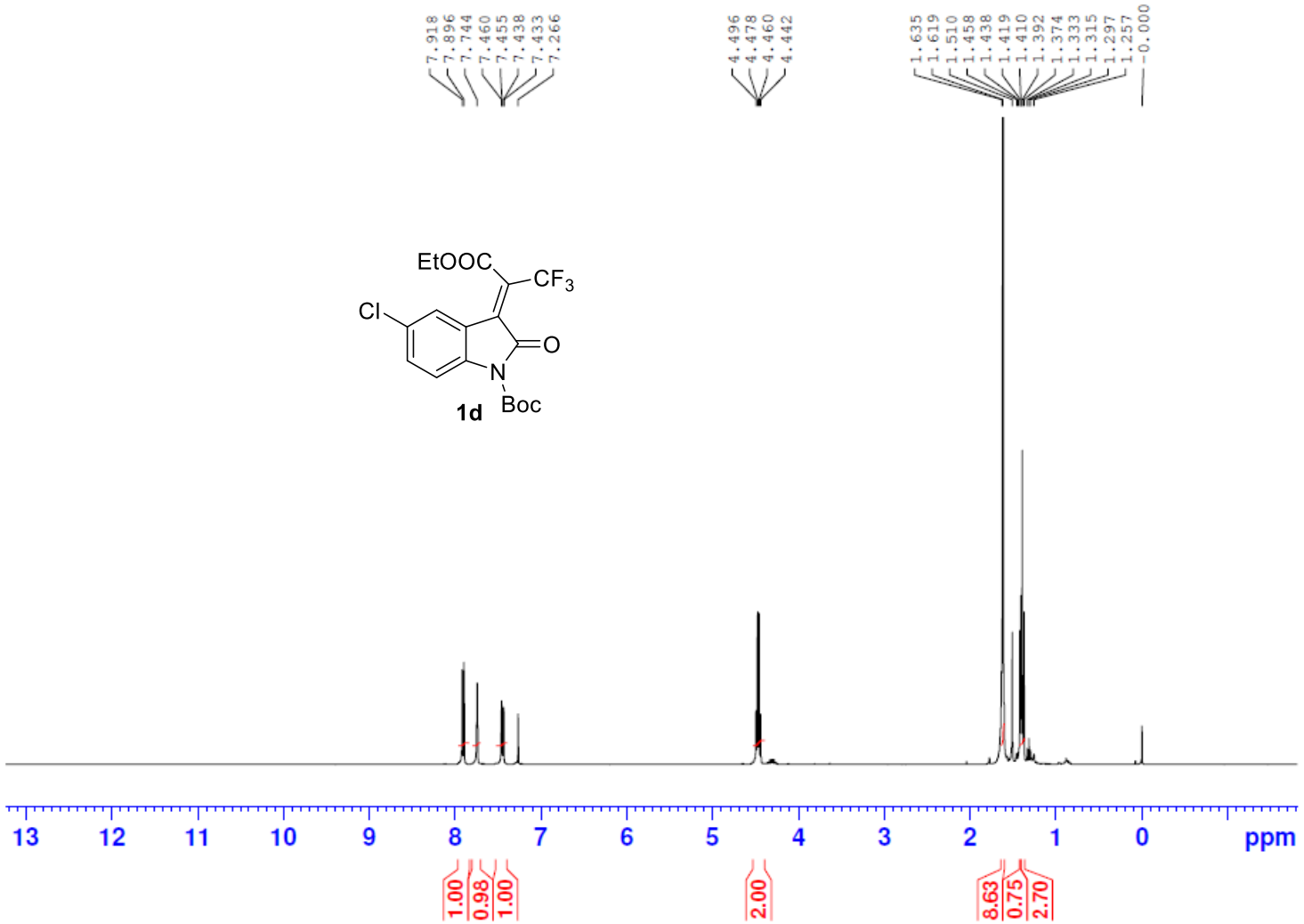



```
Current Data Parameters
NAME         512003B5106
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time                4.49 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                  2048
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 195.29
DW                 20.800 usec
DE                  6.50 usec
TE                 298.2 K
D1              3.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1           100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1           77.83999634 W
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           16.43099976 W
PLW12           0.20286000 W
PLW13           0.10204000 W

F2 - Processing parameters
SI                 32768
SF            100.6127711 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME         512003B5106
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200316
Time                9.04 h
INSTRUM            spect
PROBHD    Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT             CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          1.362392 Hz
AQ              0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          376.4607164 MHz
NUC1                  19F
P1                 18.00 usec
PLW1          19.85499954 W

F2 - Processing parameters
SI                 65536
SF            376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

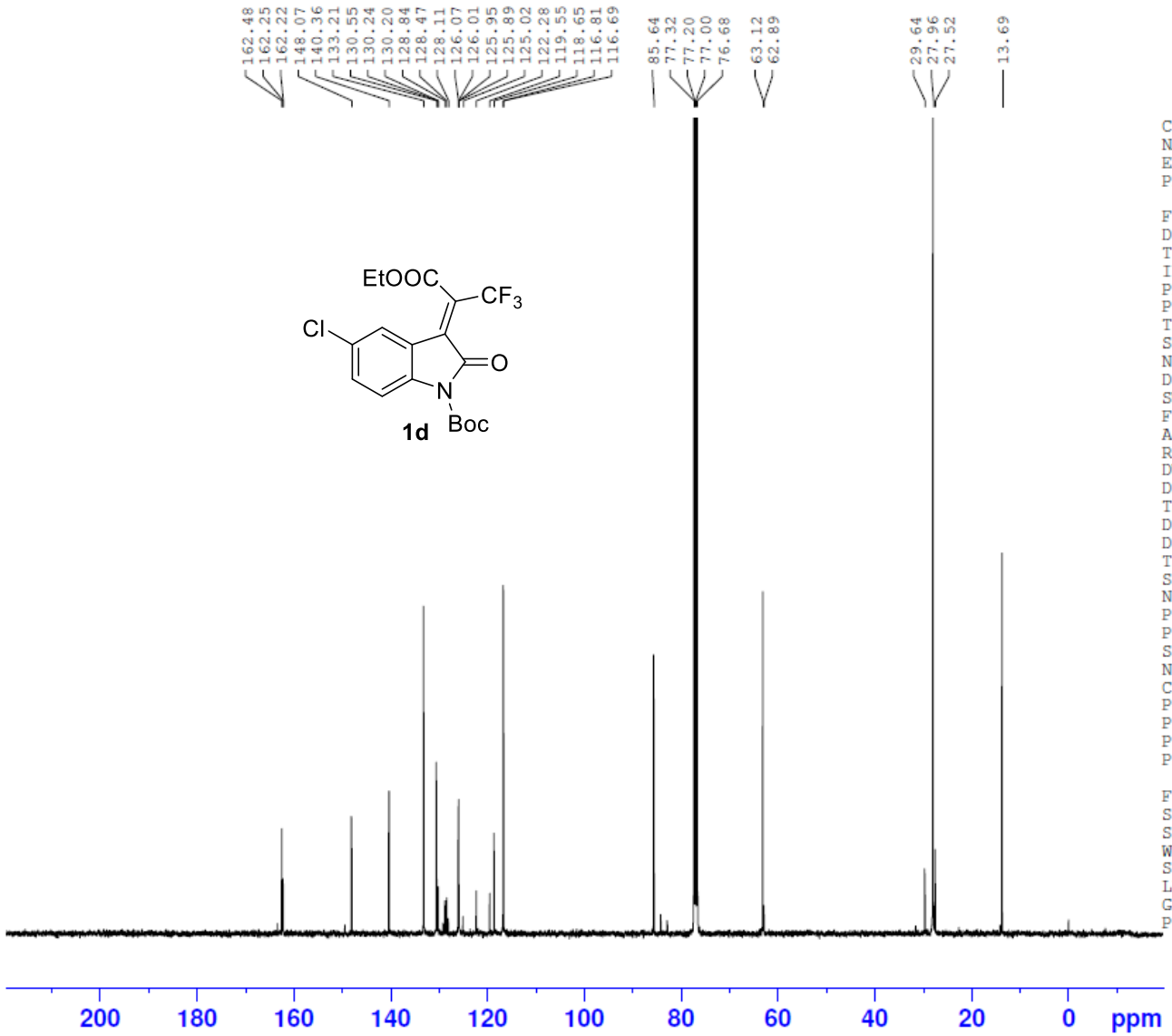
ANL-MCL5-NMR-001



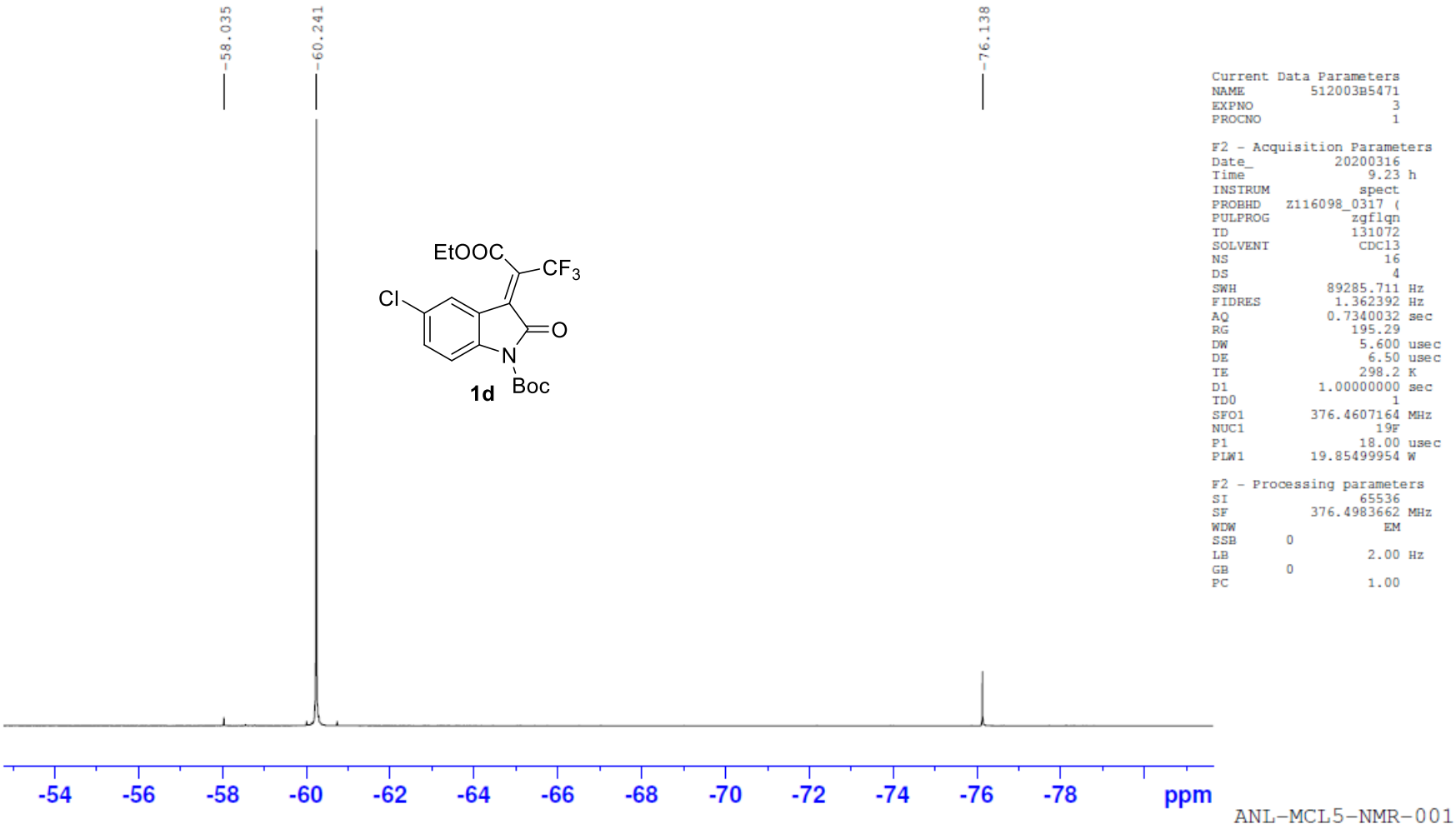

```
Current Data Parameters
NAME          512003B5471
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200315
Time                17.55 h
INSTRUM              spect
PROBHD    Z116098_0317 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  70.07
DW                  62.400 usec
DE                   6.50 usec
TE                  298.1 K
D1             1.00000000 sec
TD0                     1
SFO1           400.1324710 MHz
NUC1                   1H
P1                  10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                  65536
SF             400.1300078 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

ANL-MCL5-NMR-001

TP-11580-01-005

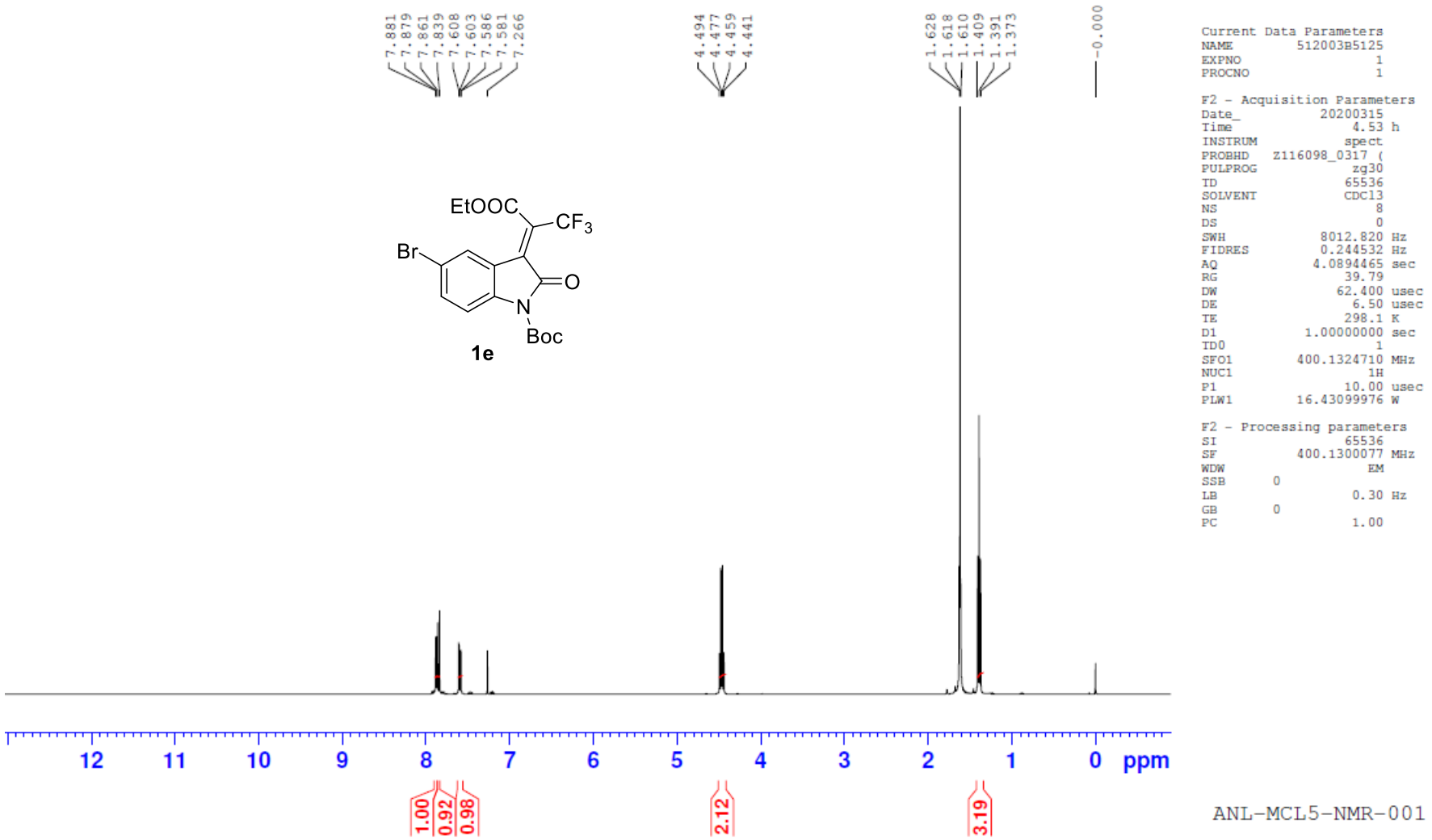



```
Current Data Parameters
NAME         512003B5471
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time               20.27 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  2048
DS                     4
SWH            24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 195.29
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          77.83999634 W
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                 32768
SF            100.6127715 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME         512003B5471
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200316
Time               9.23 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG          zgflqn
TD               131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES         1.362392 Hz
AQ             0.7340032 sec
RG                195.29
DW                5.600 usec
DE                6.50 usec
TE                298.2 K
D1            1.00000000 sec
TD0                    1
SFO1          376.4607164 MHz
NUC1                  19F
P1                18.00 usec
PLW1         19.85499954 W

F2 - Processing parameters
SI                 65536
SF            376.4983662 MHz
WDW                   EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.00
```

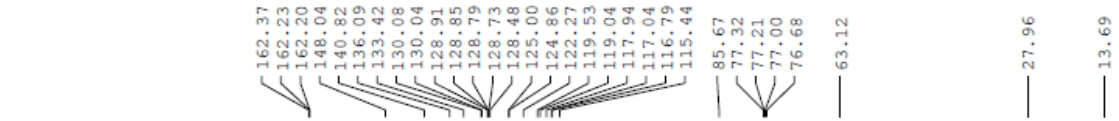
ANL-MCL5-NMR-001

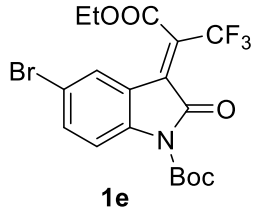



```
Current Data Parameters
NAME         512003B5125
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time                4.53 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                 39.79
DW                 62.400 usec
DE                  6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1
SFO1           400.1324710 MHz
NUC1                  1H
P1                 10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                 65536
SF           400.1300077 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

ANL-MCL5-NMR-001

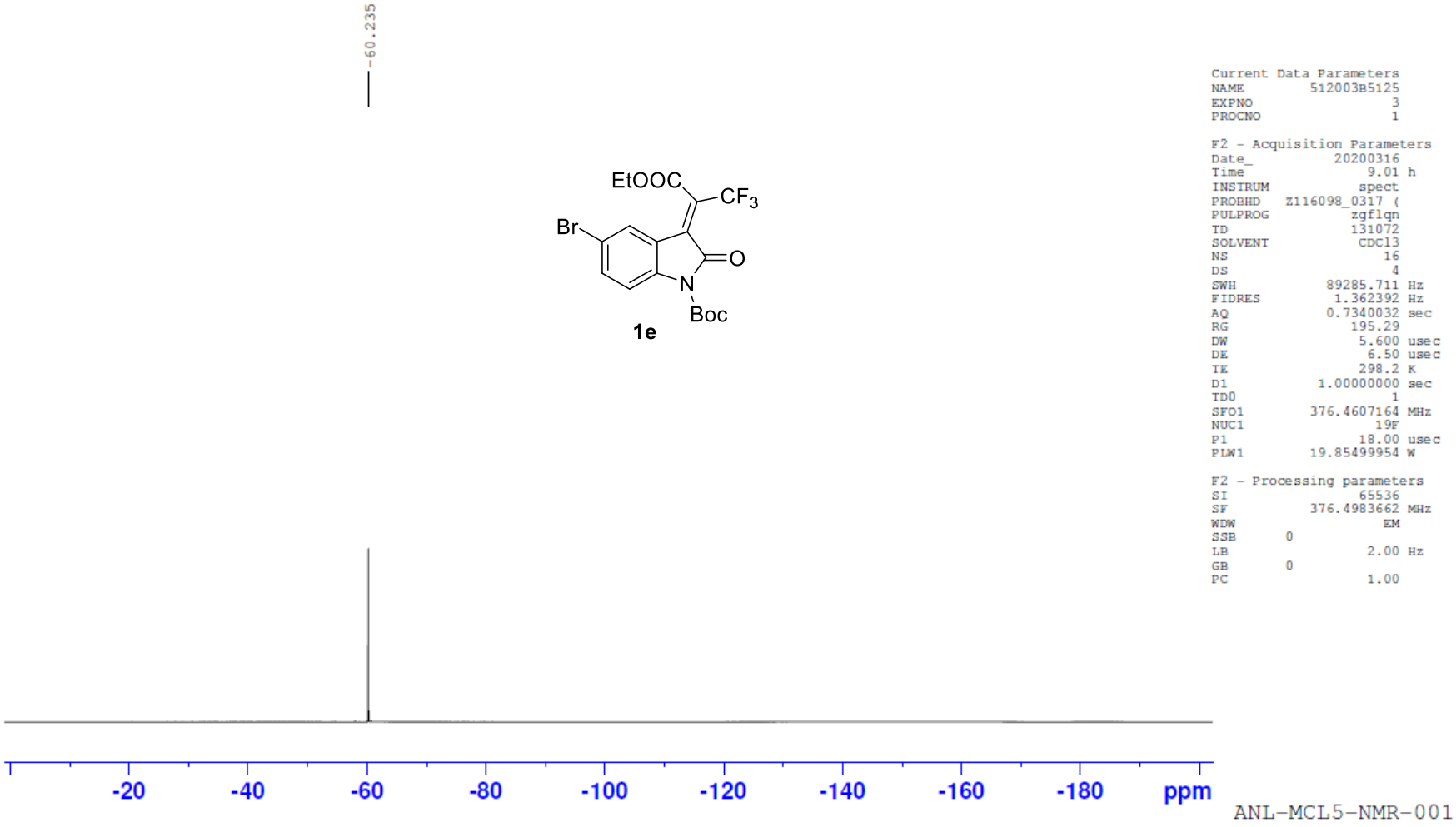
TP-11580-001-Br



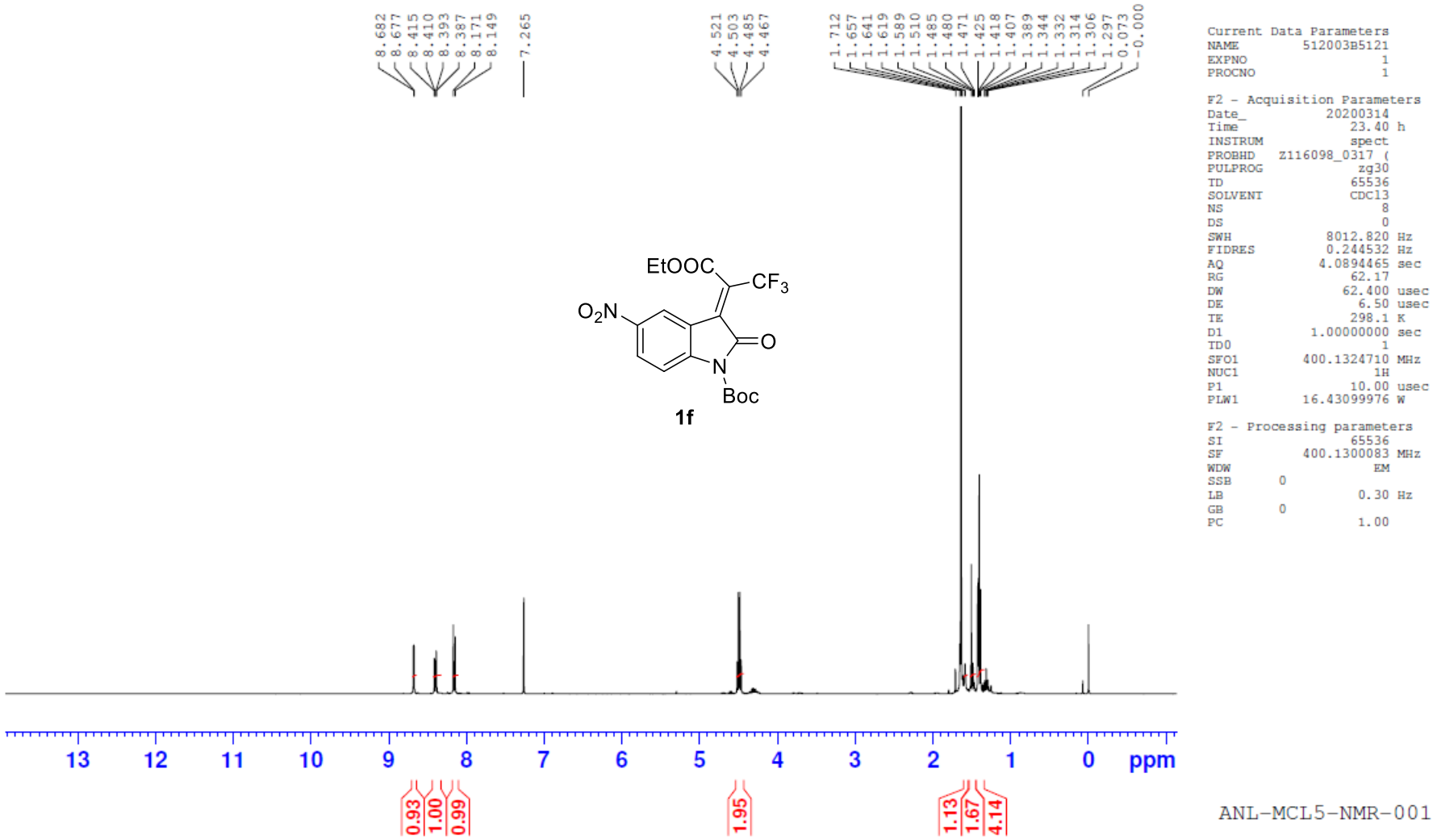


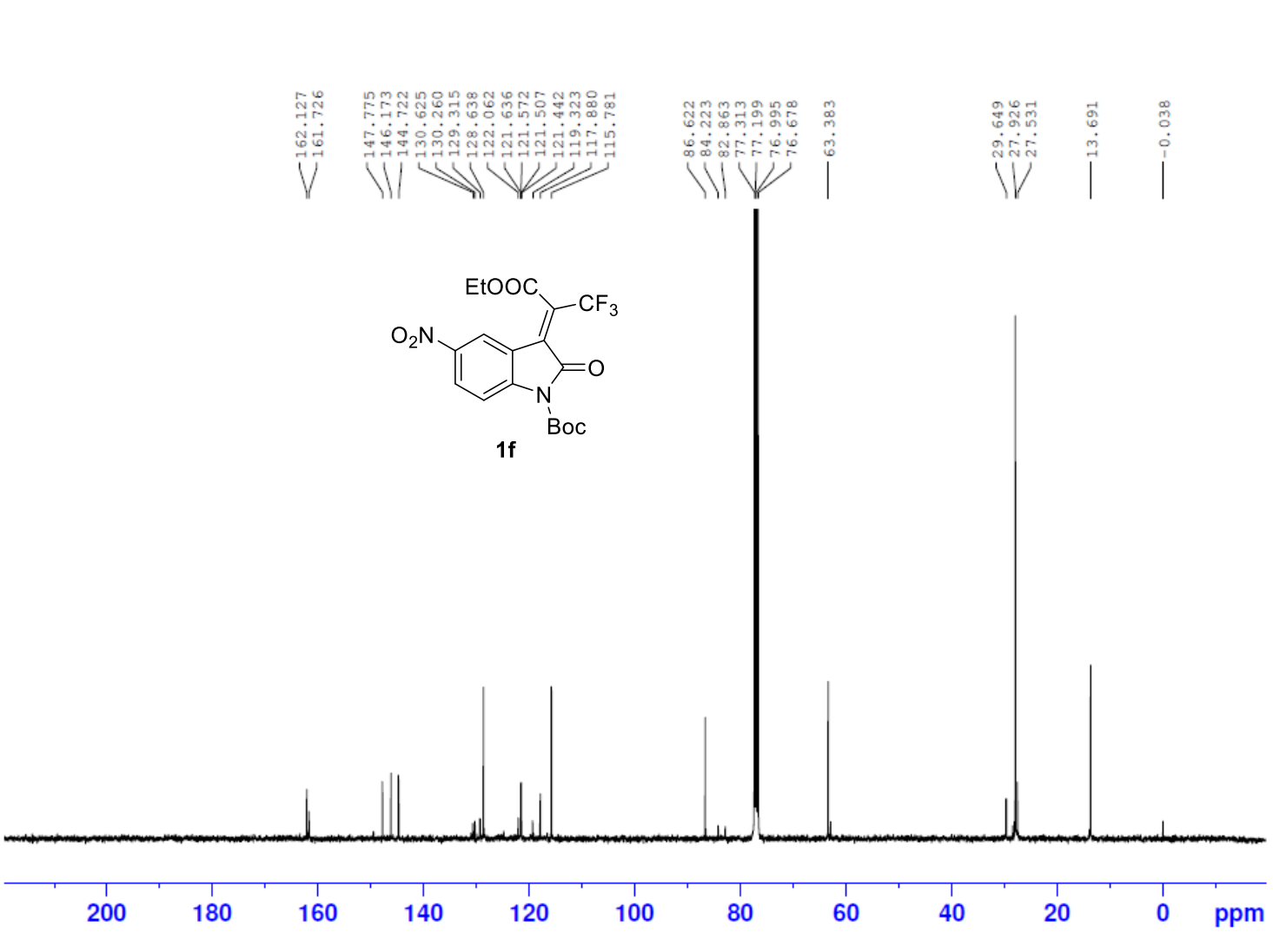
1e


```
Current Data Parameters
NAME         512003B5125
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time                7.25 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                195.29
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1         77.83999634 W
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                 32768
SF           100.6127717 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```



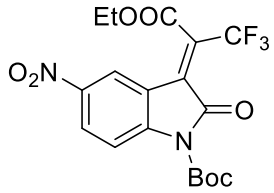

```
Current Data Parameters
NAME         512003B5125
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200316
Time                9.01 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           89285.711 Hz
FIDRES         1.362392 Hz
AQ             0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1         376.4607164 MHz
NUC1                19F
P1                 18.00 usec
PLW1         19.85499954 W

F2 - Processing parameters
SI                 65536
SF           376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```


8.682
8.677
8.415
8.410
8.393
8.387
8.171
8.149
7.265
4.521
4.503
4.485
4.467
1.712
1.657
1.641
1.619
1.589
1.510
1.485
1.480
1.471
1.425
1.418
1.407
1.389
1.344
1.332
1.314
1.306
1.297
0.073
-0.000
EtOOC
CF3
O2N
O
N
Boc
1f
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
1.00
0.99
1.95
1.13
1.67
4.14
Current Data Parameters
NAME 512003B5121
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200314
Time 23.40 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 62.17
DW 62.400 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ANL-MCL5-NMR-001



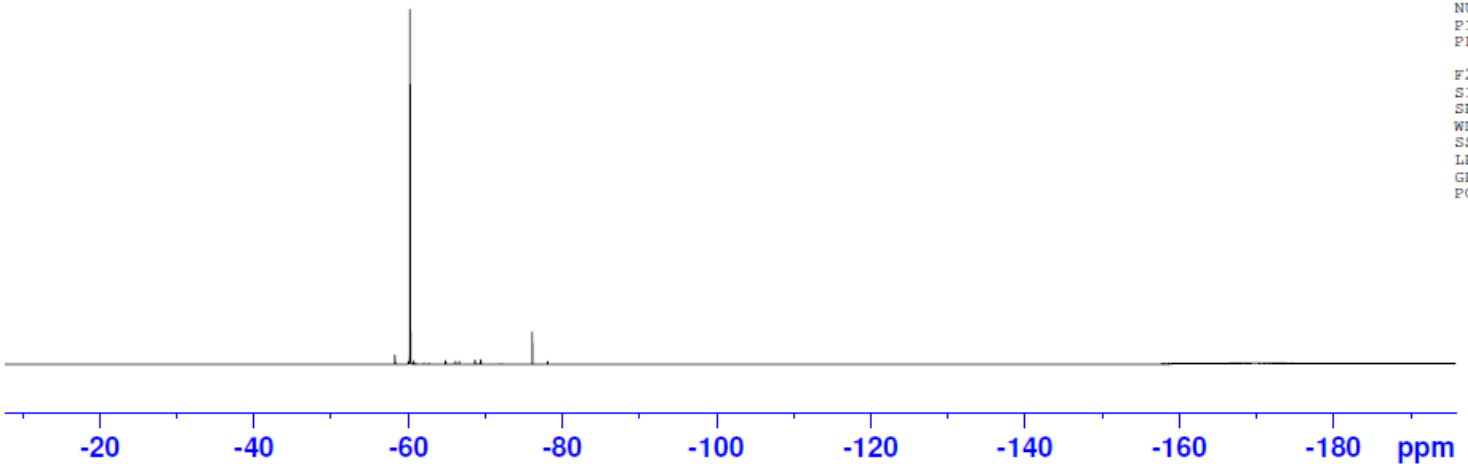

```
Current Data Parameters
NAME         512003B5121
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time                2.13 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                 2048
DS                    4
SWH           24038.461 Hz
FIDRES        0.733596 Hz
AQ            1.3631488 sec
RG               195.29
DW               20.800 usec
DE                6.50 usec
TE               298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1         77.83999634 W
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                32768
SF           100.6127713 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.40
```

ANL-MCL5-NMR-001

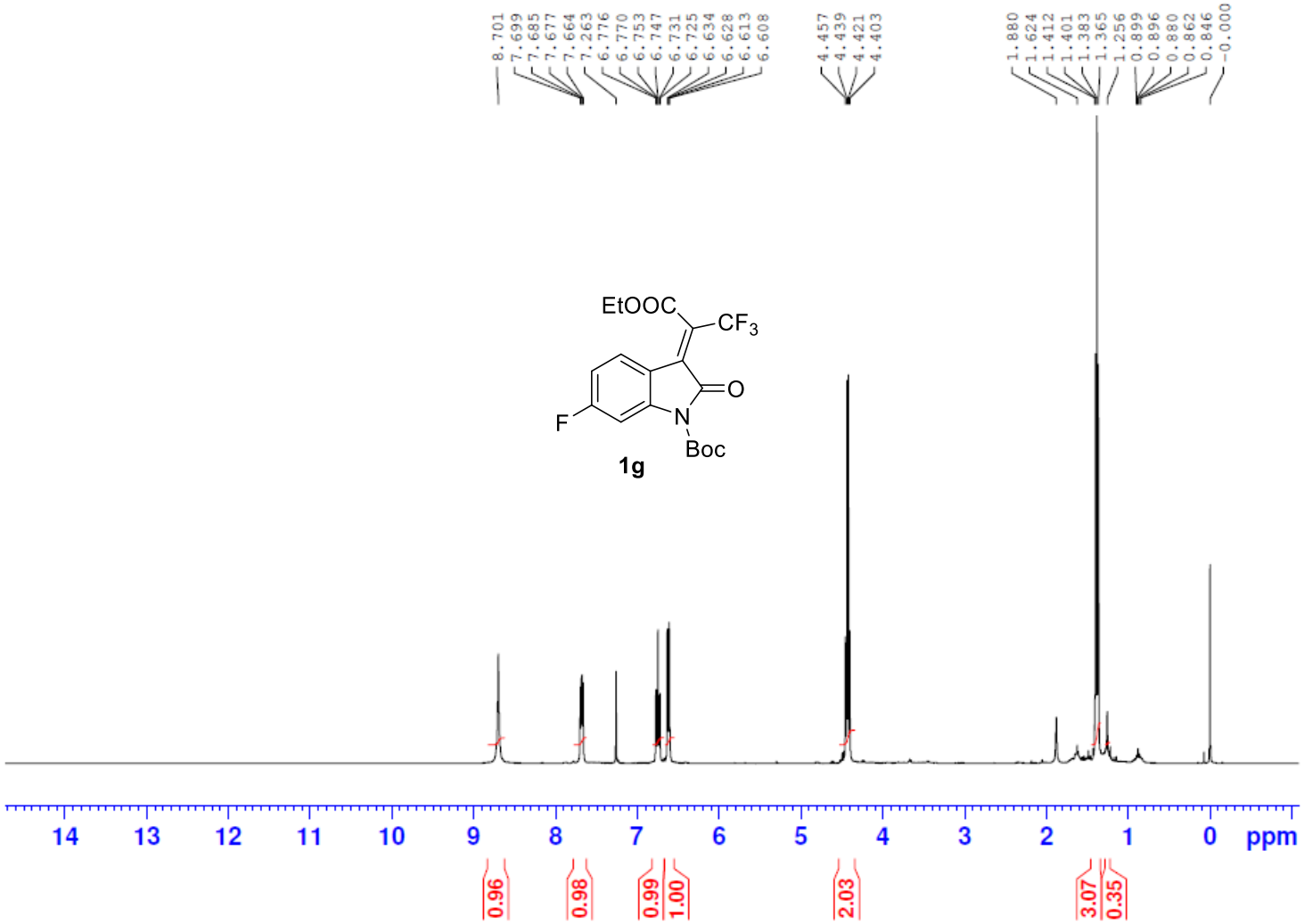


1f

```
Current Data Parameters
NAME          512003B5121
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200316
Time                8.57 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           89285.711 Hz
FIDRES         1.362392 Hz
AQ             0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1
SFO1         376.4607164 MHz
NUC1                  19F
P1                18.00 usec
PLW1         19.85499954 W

F2 - Processing parameters
SI                 65536
SF           376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

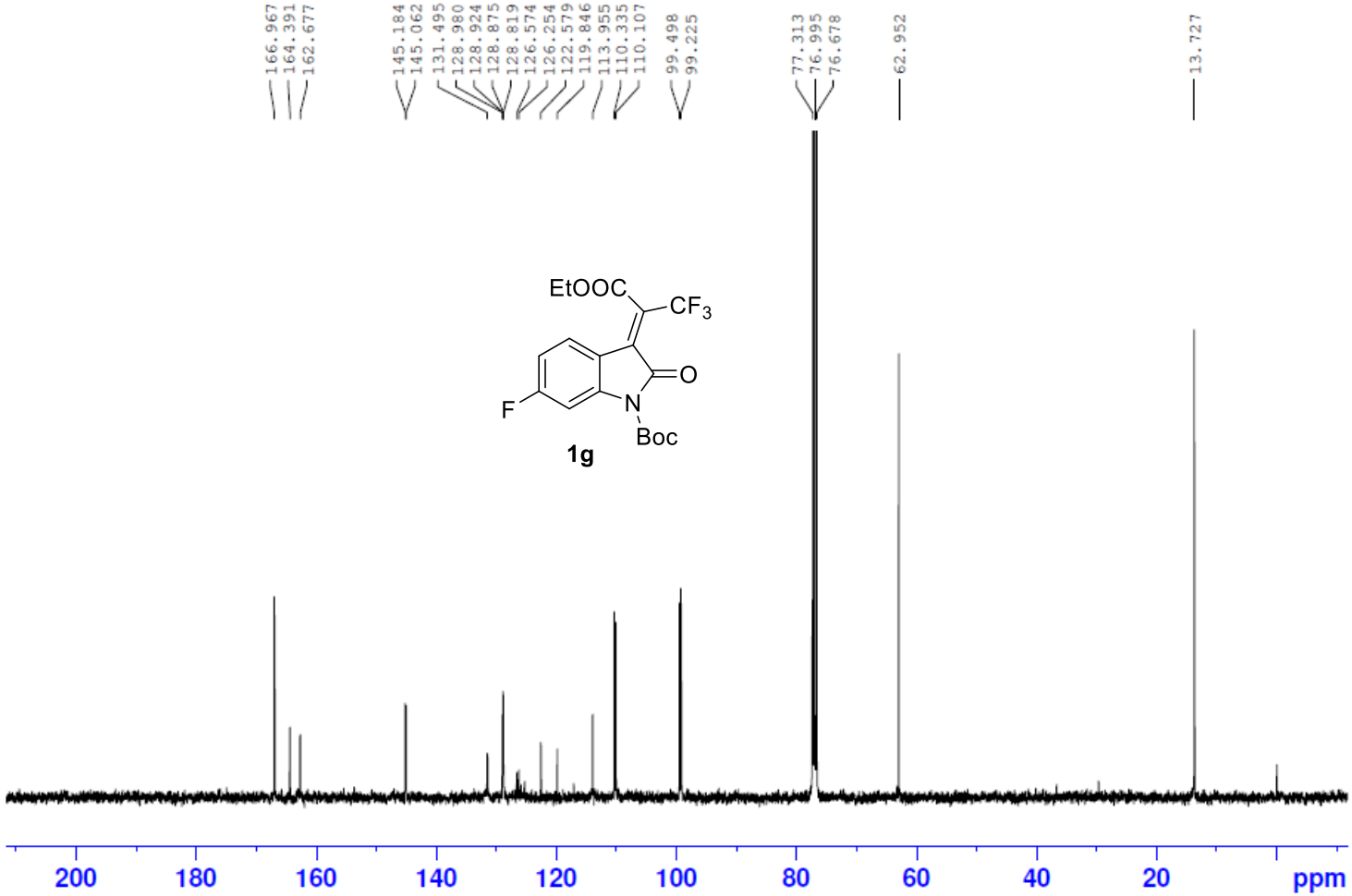


ANL-MCL5-NMR-001



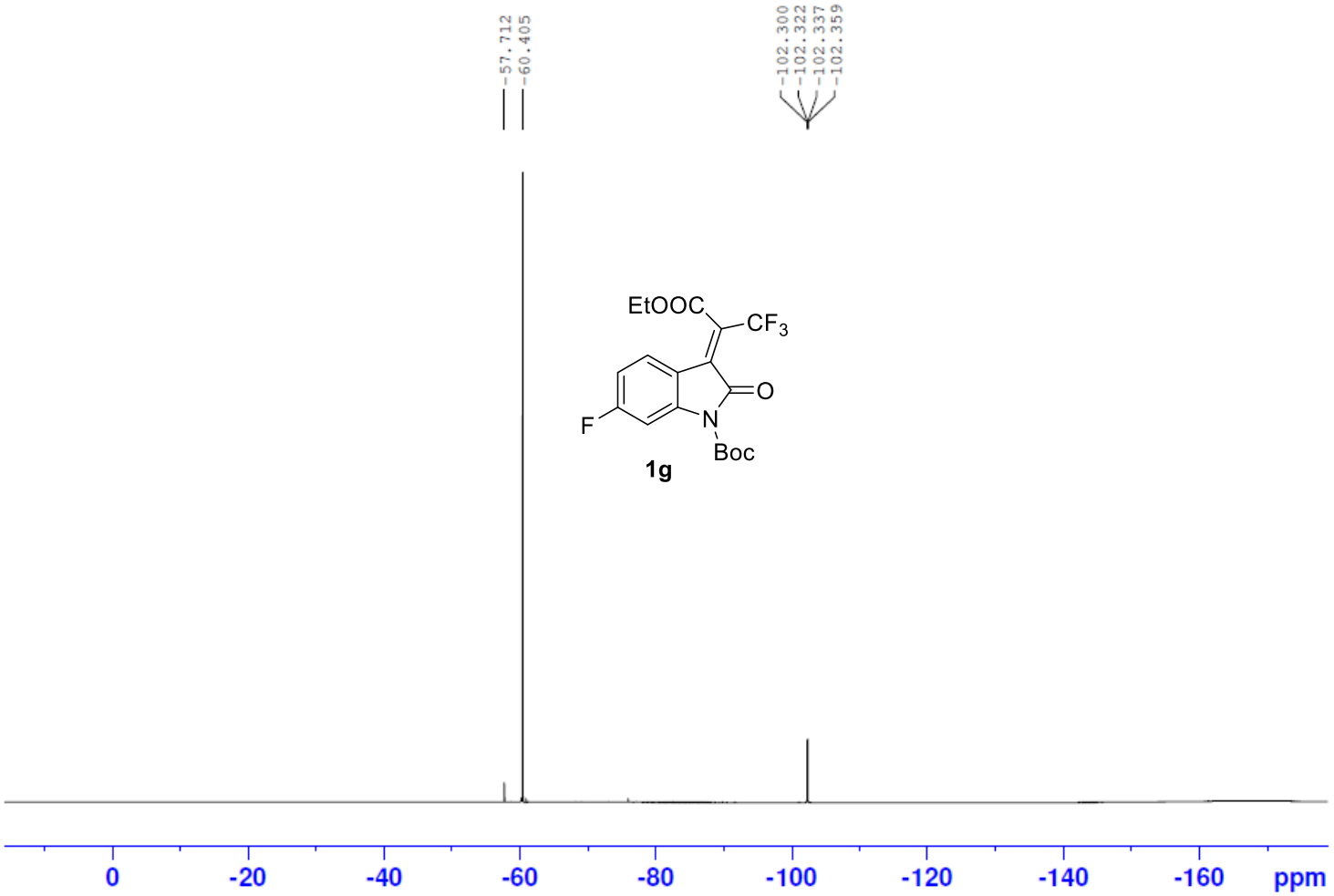

```
Current Data Parameters
NAME          512003c5424
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200321
Time                3.35 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                 110.59
DW                 62.400 usec
DE                  6.50 usec
TE                 298.2 K
D1              1.00000000 sec
TD0                    1
SFO1           400.1324710 MHz
NUC1                  1H
P1                 10.00 usec
PLW1           16.43099976 W

F2 - Processing parameters
SI                 65536
SF             400.1300092 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-001



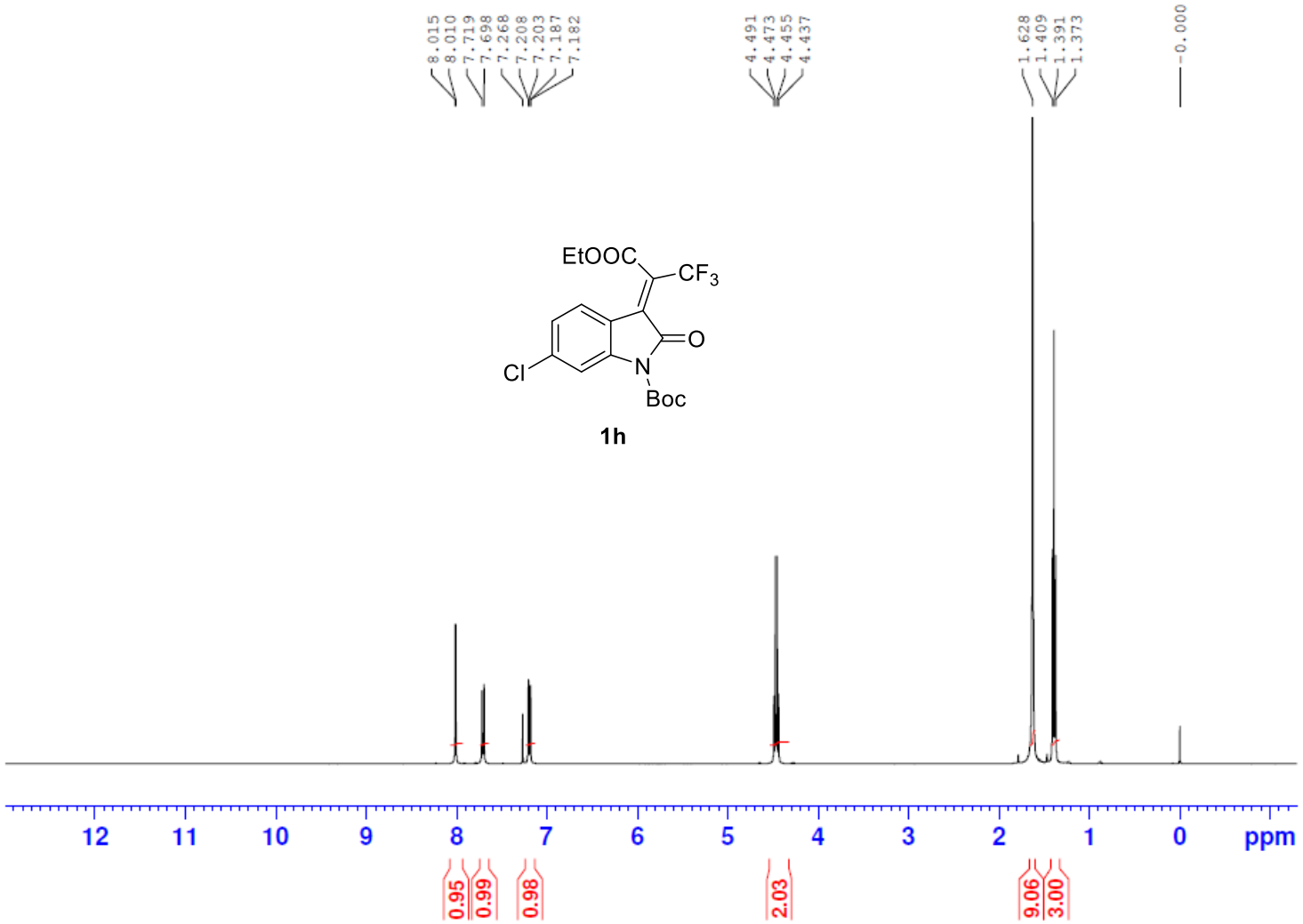

```
Current Data Parameters
NAME          512003c5424
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200321
Time                4.36 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                195.29
DW                20.800 usec
DE                 6.50 usec
TE                298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          77.83999634 W
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                 32768
SF            100.6127713 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

ANL-MCL5-NMR-001



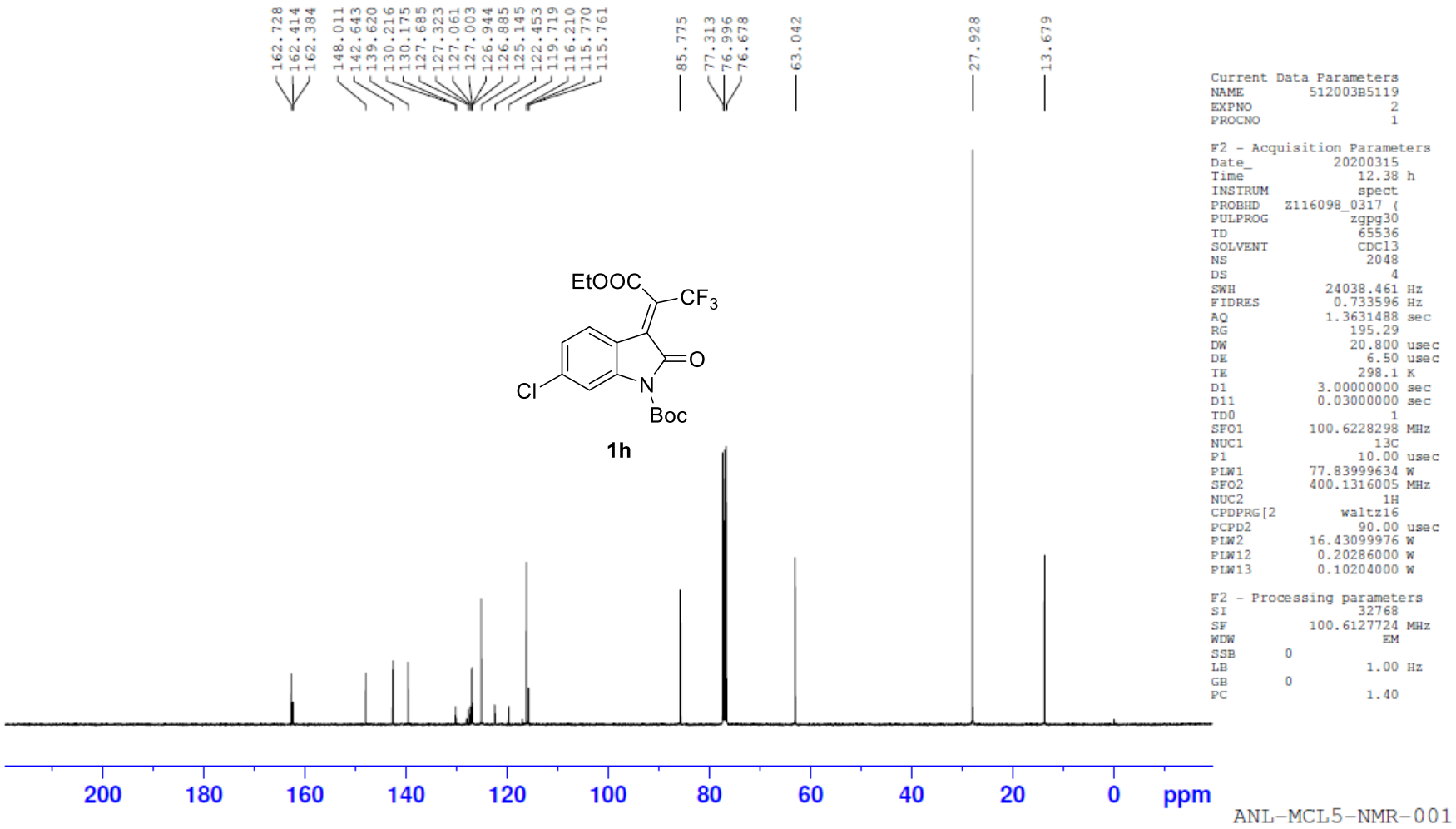

```
Current Data Parameters
NAME         512003C5424
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200321
Time               3.36 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG          zgflqn
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES         1.362392 Hz
AQ             0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          376.4607164 MHz
NUC1                  19F
P1                 18.00 usec
PLW1          19.85499954 W

F2 - Processing parameters
SI                 65536
SF            376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

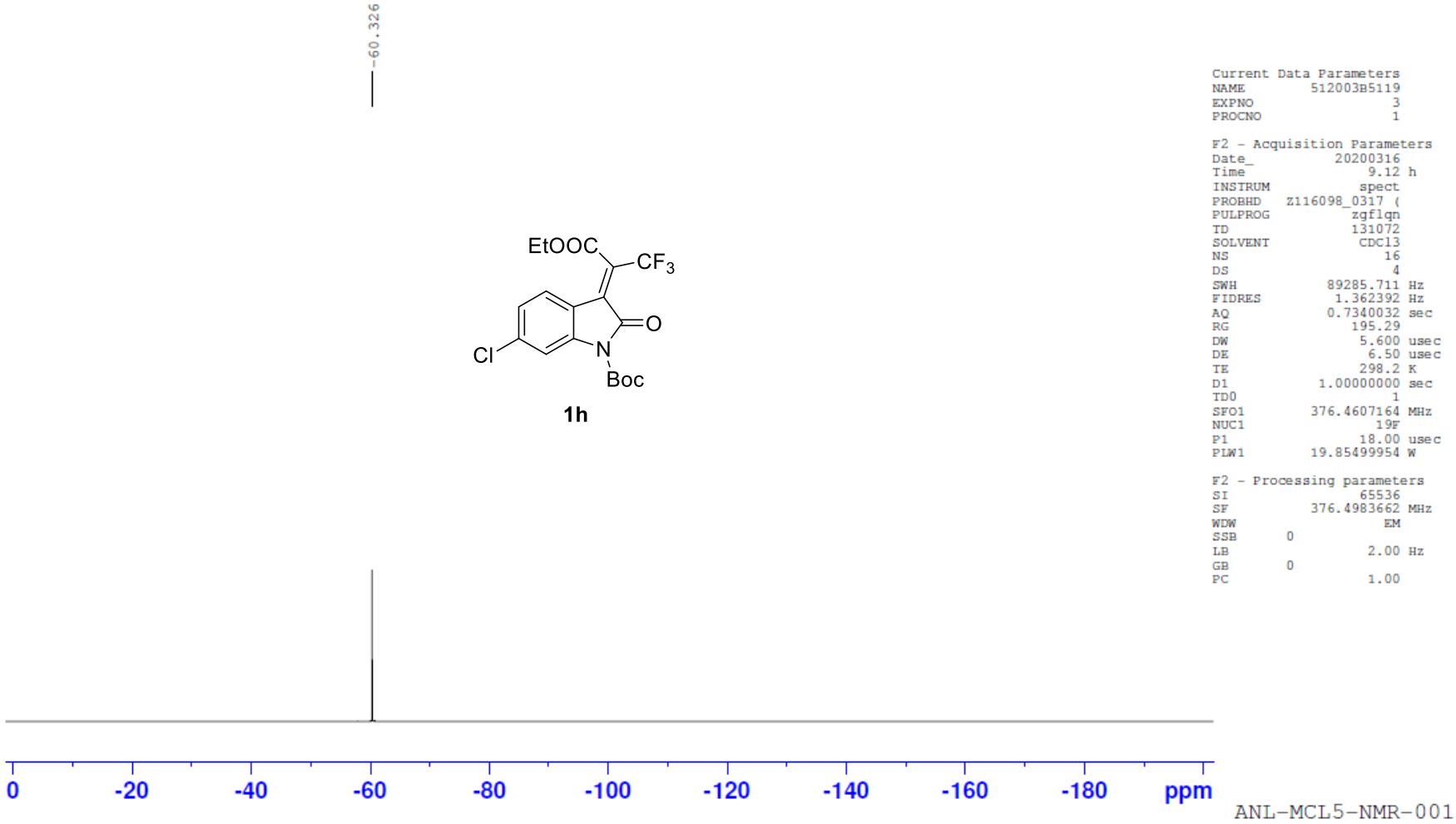
ANL-MCL5-NMR-001

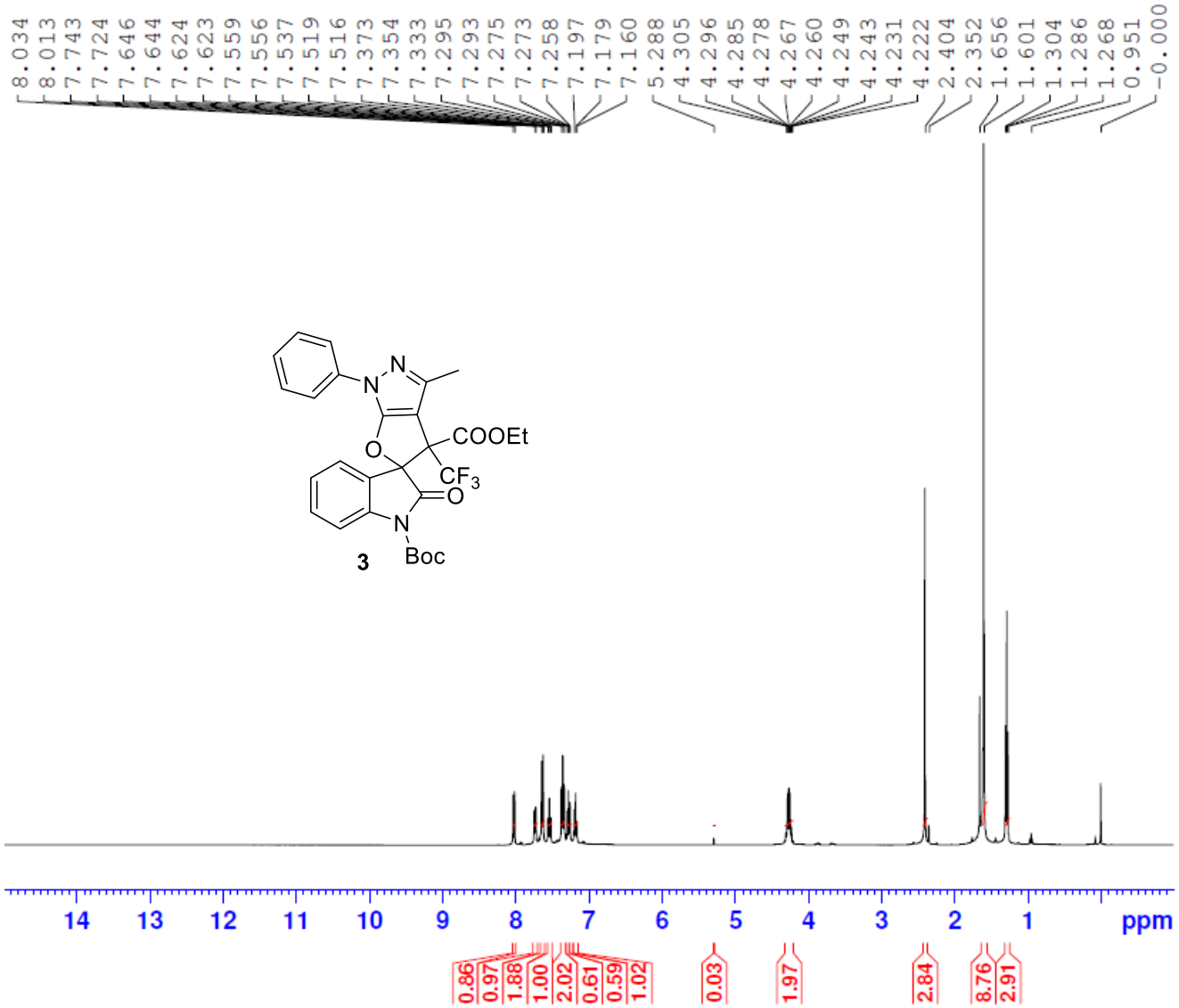



```
Current Data Parameters
NAME         512003B5119
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200315
Time               10.05 h
INSTRUM            spect
PROBHD   Z116098_0317 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES             0.244532 Hz
AQ              4.0894465 sec
RG                 62.17
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1              1.00000000 sec
TD0                    1
SFO1          400.1324710 MHz
NUC1                  1H
P1                10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                 65536
SF            400.1300071 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-001

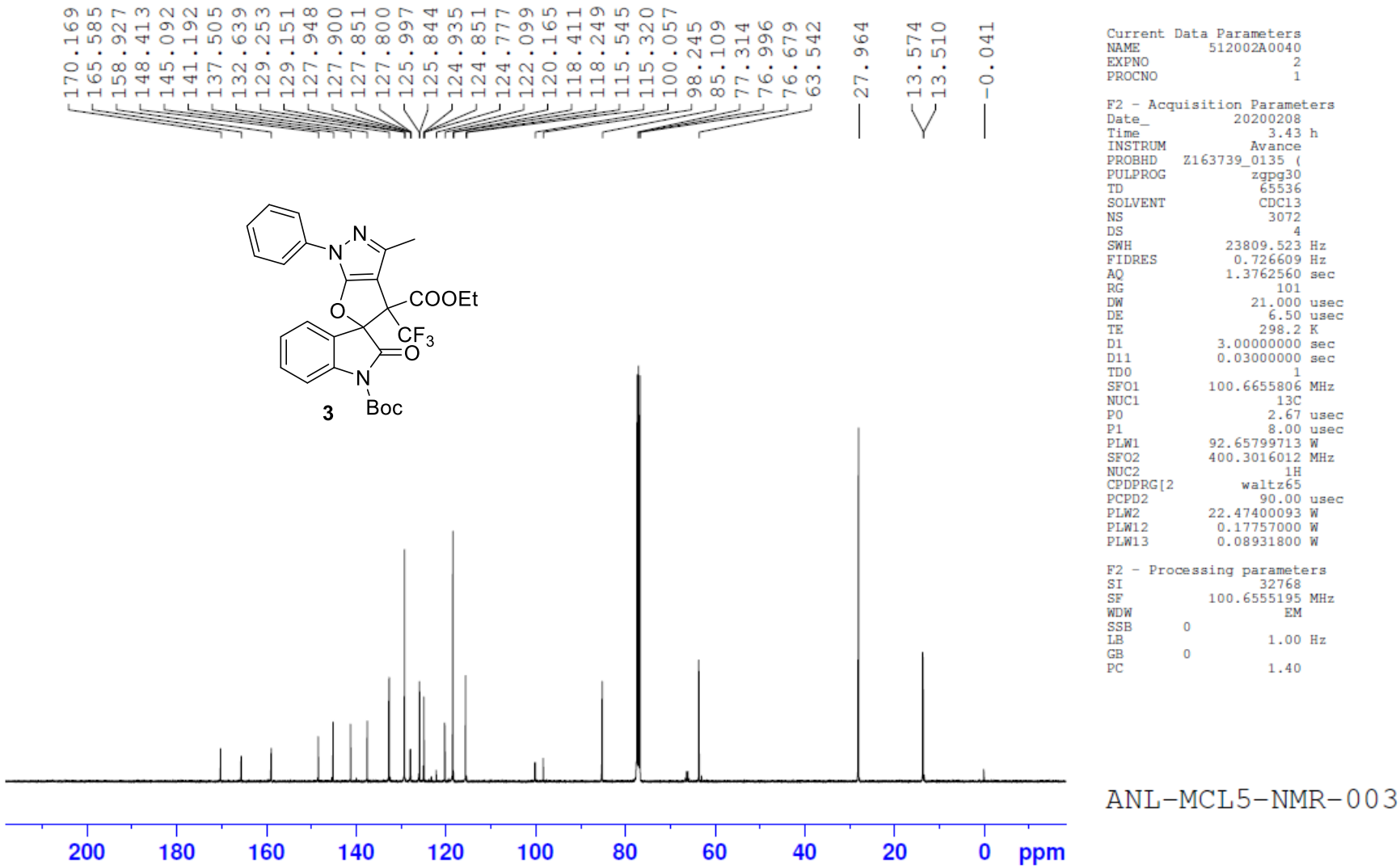

162.728
162.414
162.384
148.011
142.643
139.620
130.216
130.175
127.685
127.323
127.061
127.003
126.944
126.885
125.145
122.453
119.719
116.210
115.770
115.761
85.775
77.313
76.996
76.678
63.042
27.928
13.679
EtOOC
CF3
O
N
Cl
Boc
1h
Current Data Parameters
NAME 512003B5119
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200315
Time 12.38 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 195.29
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 77.83999634 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.43099976 W
PLW12 0.20286000 W
PLW13 0.10204000 W
F2 - Processing parameters
SI 32768
SF 100.6127724 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm
ANL-MCL5-NMR-001


-60.326
EtOOC
CF3
O
Cl
N
Boc
1h
Current Data Parameters
NAME 512003B5119
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200316
Time 9.12 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 1.362392 Hz
AQ 0.7340032 sec
RG 195.29
DW 5.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 18.00 usec
PLW1 19.85499954 W
F2 - Processing parameters
SI 65536
SF 376.4983662 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm
ANL-MCL5-NMR-001



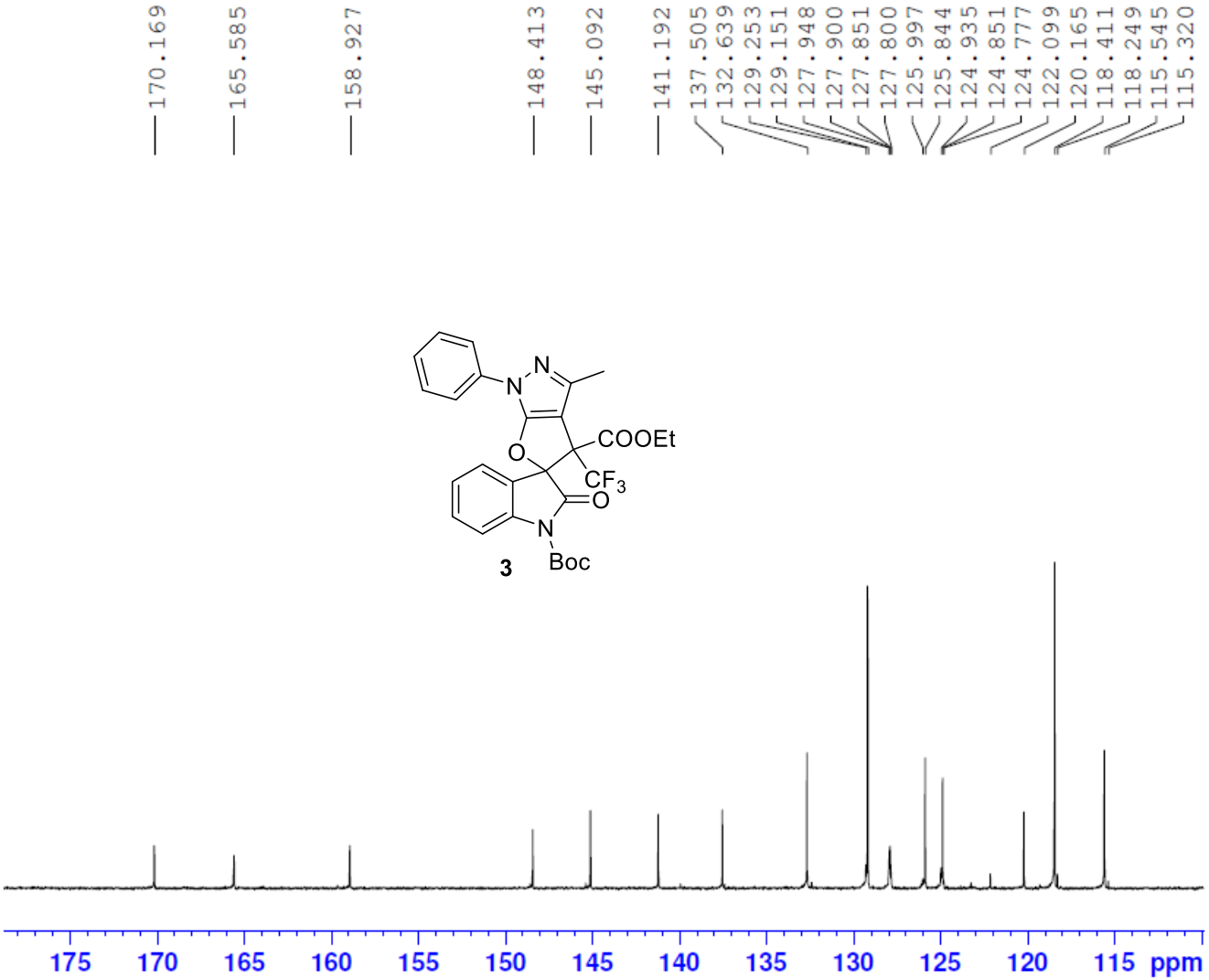

```
Current Data Parameters
NAME         512002A0040
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200207
Time              21.38 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                61.4354
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.3024719 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                 65536
SF            400.3000107 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-003



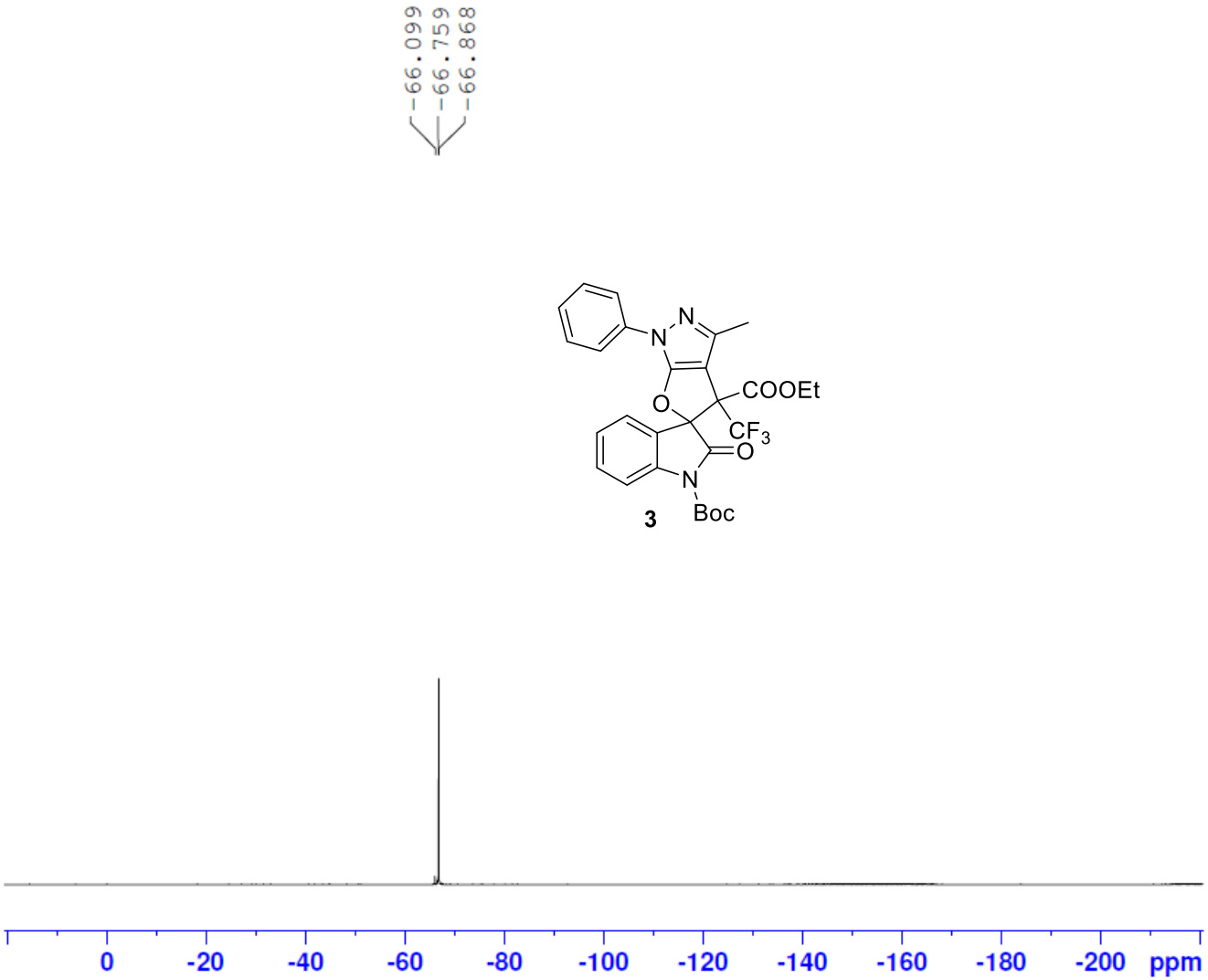

```
Current Data Parameters
NAME         512002A0040
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time                3.43 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         100.6655806 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1         92.65799713 W
SFO2         400.3016012 MHz
NUC2                 1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.47400093 W
PLW12         0.17757000 W
PLW13         0.08931800 W

F2 - Processing parameters
SI                 32768
SF           100.6555195 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

ANL-MCL5-NMR-003



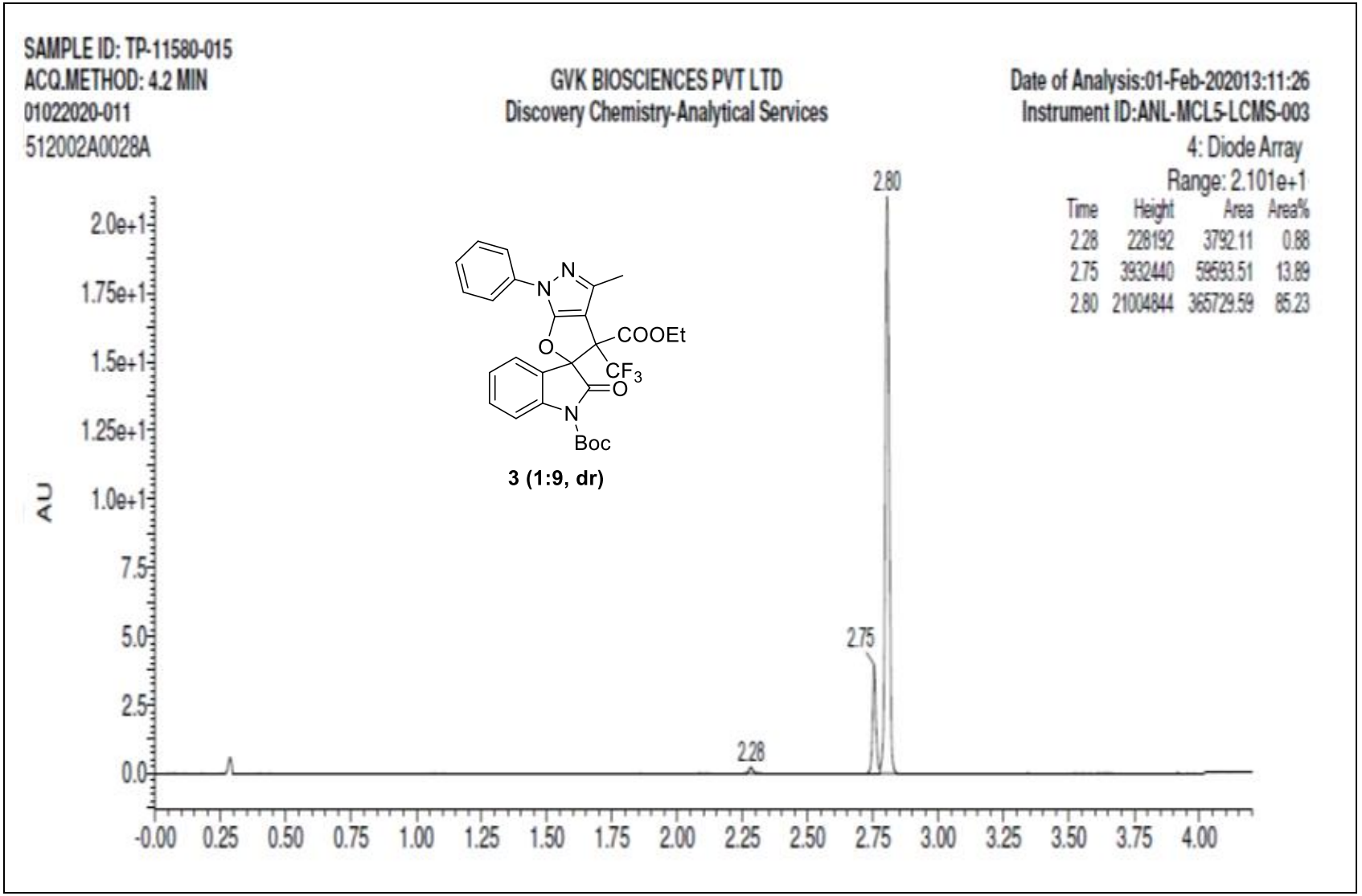

```
Current Data Parameters
NAME         512002A0040
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time                3.43 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 3072
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6655806 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1         92.65799713 W
SFO2         400.3016012 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.47400093 W
PLW12         0.17757000 W
PLW13         0.08931800 W

F2 - Processing parameters
SI                 32768
SF           100.6555195 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

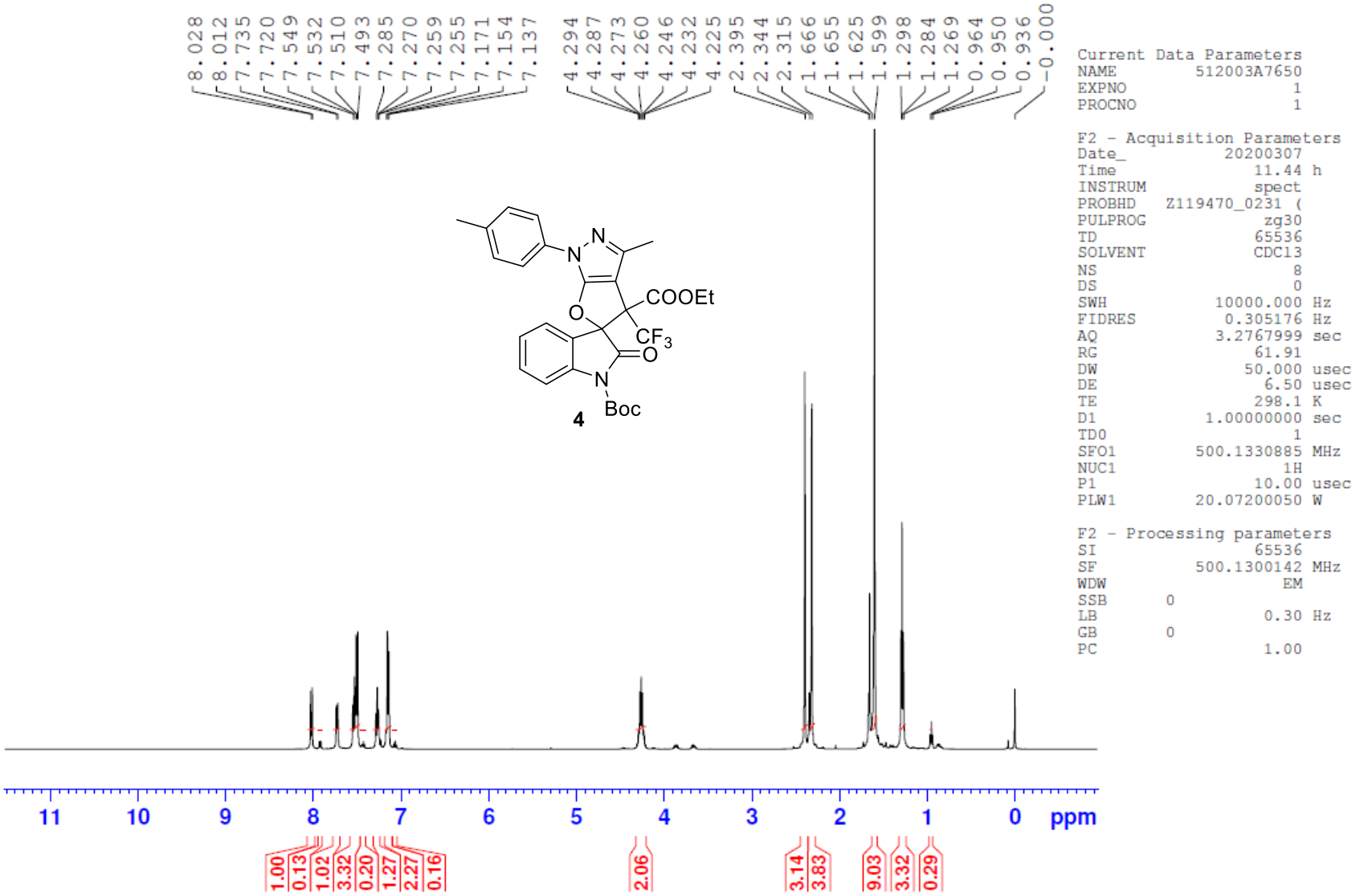
ANL-MCL5-NMR-003



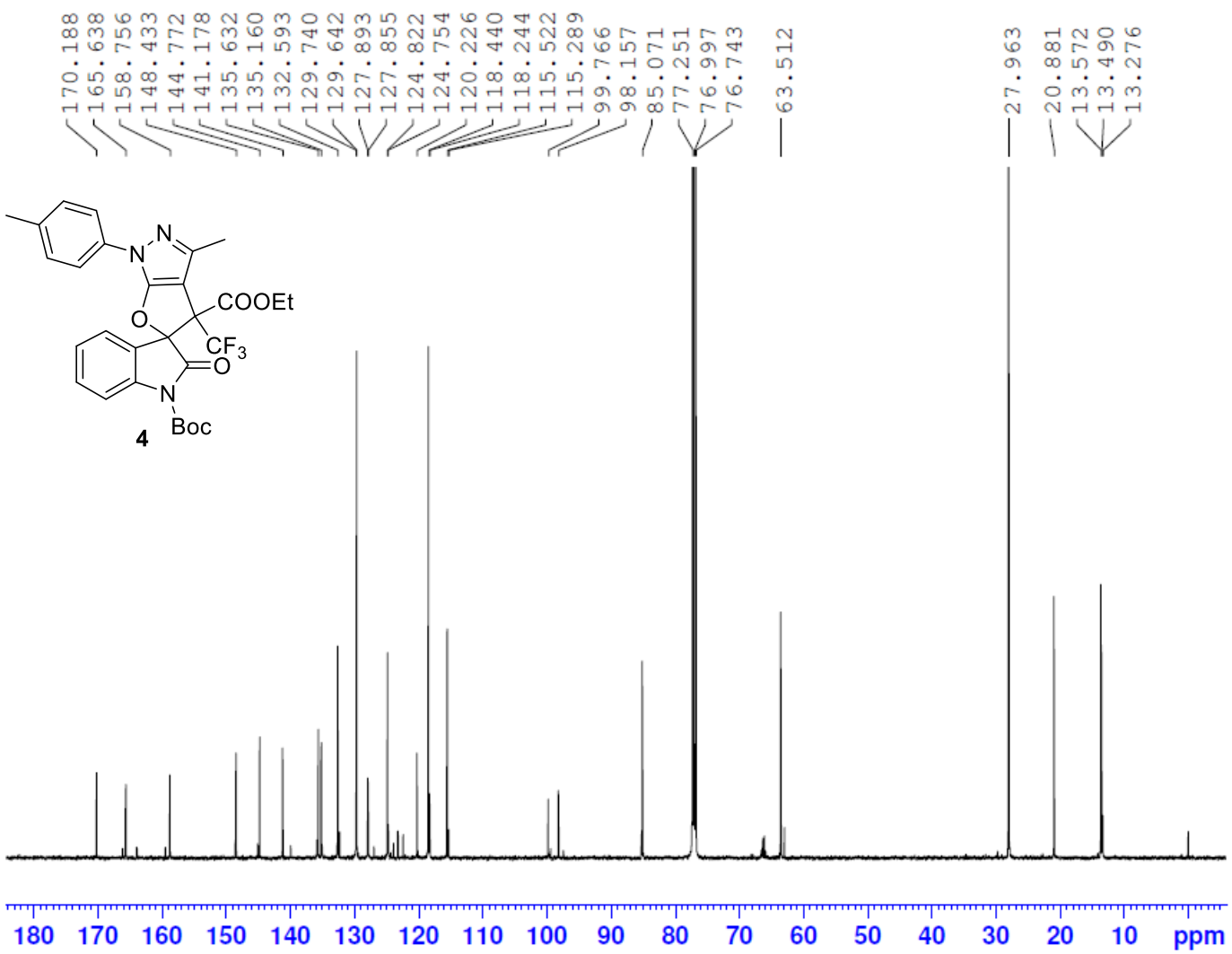

```
Current Data Parameters
NAME         512002A0040
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time                3.45 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zgig
TD                131072
SOLVENT            CDC13
NS                    16
DS                     4
SWH           90909.094 Hz
FIDRES         1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                 298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1         37.49499893 W
SFO2         400.3016012 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         22.47400093 W
PLW12         0.17757000 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-003


SAMPLE ID: TP-11580-015
ACQ.METHOD: 4.2 MIN
01022020-011
512002A0028A
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:01-Feb-202013:11:26
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 2.101e+1
Time Height Area Area%
2.28 228192 3792.11 0.88
2.75 3932440 59593.51 13.89
2.80 21004844 365729.59 85.23
COOEt
CF3
O
N
Boc
3 (1:9, dr)
AU
2.80
2.75
2.28

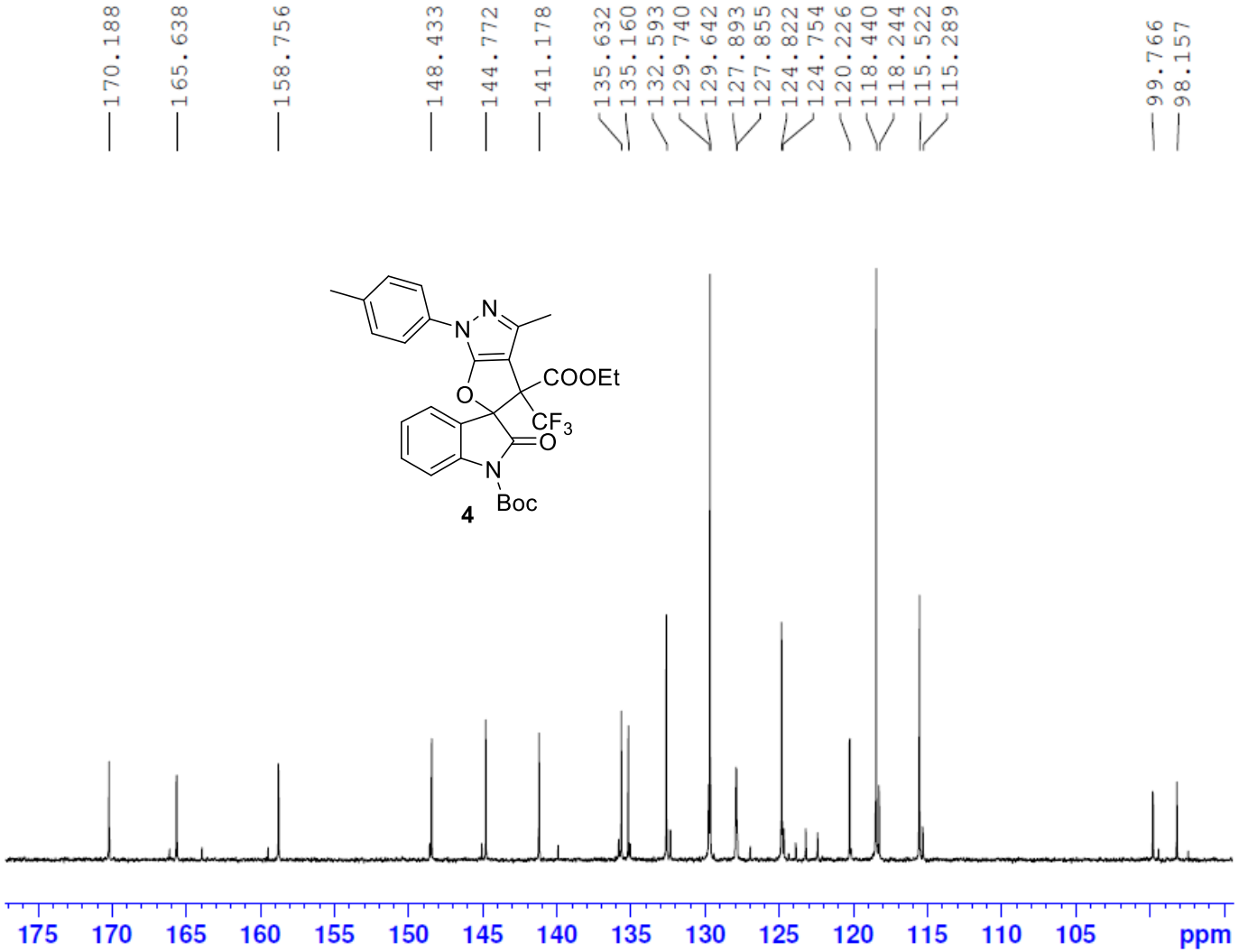



```
Current Data Parameters
NAME         512003A7650
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200307
Time               11.44 h
INSTRUM            spect
PROBHD   Z119470_0231 (
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     0
SWH            10000.000 Hz
FIDRES             0.305176 Hz
AQ              3.2767999 sec
RG                 61.91
DW                 50.000 usec
DE                  6.50 usec
TE                 298.1 K
D1              1.00000000 sec
TD0                    1
SFO1           500.1330885 MHz
NUC1                  1H
P1                 10.00 usec
PLW1           20.07200050 W

F2 - Processing parameters
SI                 65536
SF             500.1300142 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

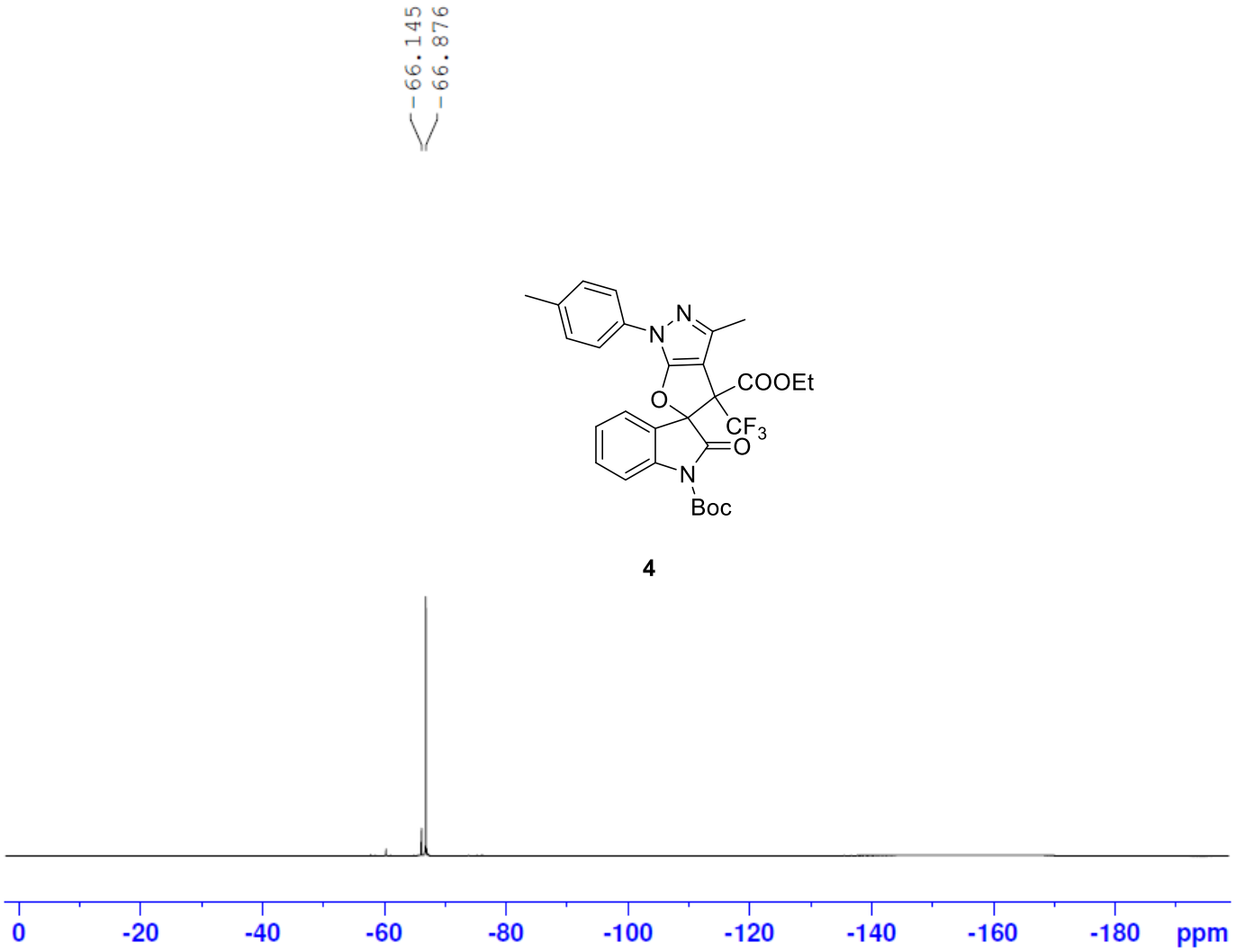



```
Current Data Parameters
NAME          512003A7650
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200308
Time                21.57 h
INSTRUM              spect
PROBHD    Z119470_0231 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    3072
DS                       4
SWH              29761.904 Hz
FIDRES           0.908261 Hz
AQ               1.1010048 sec
RG                  197.72
DW                  16.800 usec
DE                   6.50 usec
TE                  298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                      1
SFO1         125.7703643 MHz
NUC1                  13C
P1                  10.00 usec
PLW1         86.78700256 W
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         20.07200050 W
PLW12         0.31363001 W
PLW13         0.15775000 W

F2 - Processing parameters
SI                   32768
SF           125.7577944 MHz
WDW                     EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   1.40
```

TP-11580-012




```
Current Data Parameters
NAME          512003A7650
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200308
Time                21.57 h
INSTRUM             spect
PROBHD    Z119470_0231 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                197.72
DW                16.800 usec
DE                 6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          86.78700256 W
SFO2          500.1320005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          20.07200050 W
PLW12          0.31363001 W
PLW13          0.15775000 W

F2 - Processing parameters
SI                 32768
SF            125.7577944 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```



4

```
Current Data Parameters
NAME         512003A7650
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200308
Time               21.59 h
INSTRUM             spect
PROBHD    Z119470_0231 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ              0.5767168 sec
RG                 197.72
DW                 4.400 usec
DE                 6.50 usec
TE                 298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1
SFO1         470.5453180 MHz
NUC1                  19F
P1                 15.00 usec
PLW1         34.86600113 W
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         20.07200050 W
PLW12         0.31363001 W

F2 - Processing parameters
SI                 65536
SF           470.5923772 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

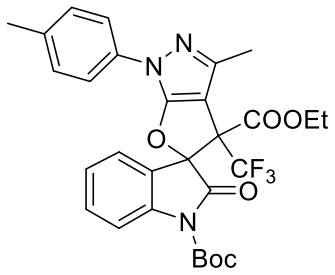
GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services

SAMPLE ID:TP-11580-012
06032020-123
ACQ.METHOD:FA-4-2 MIN
512003A7652A

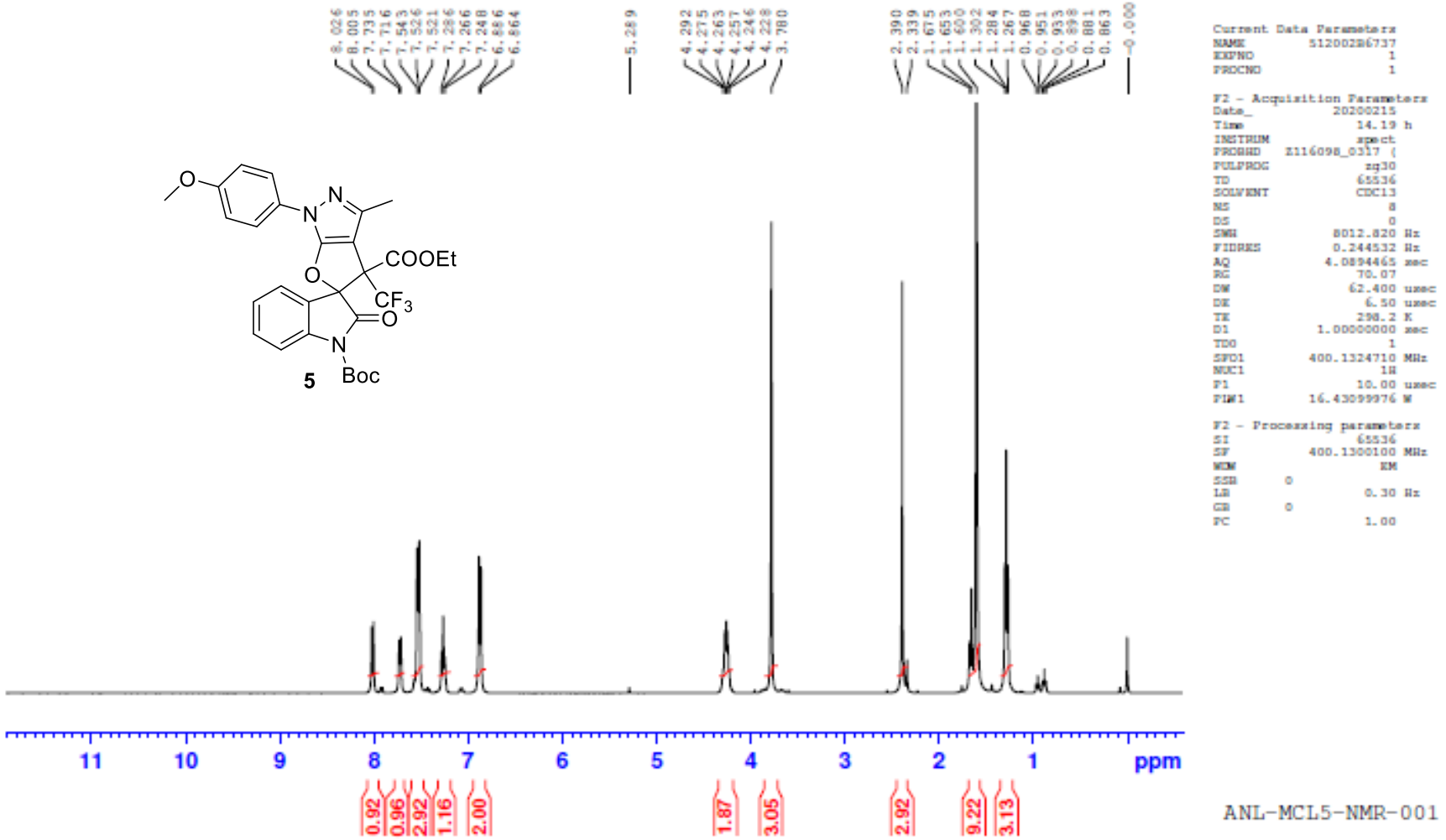
Date of analysis: 06-Mar-202020:04:03
Instrument ID :ANL-MCL4-LCMS-SQD-1

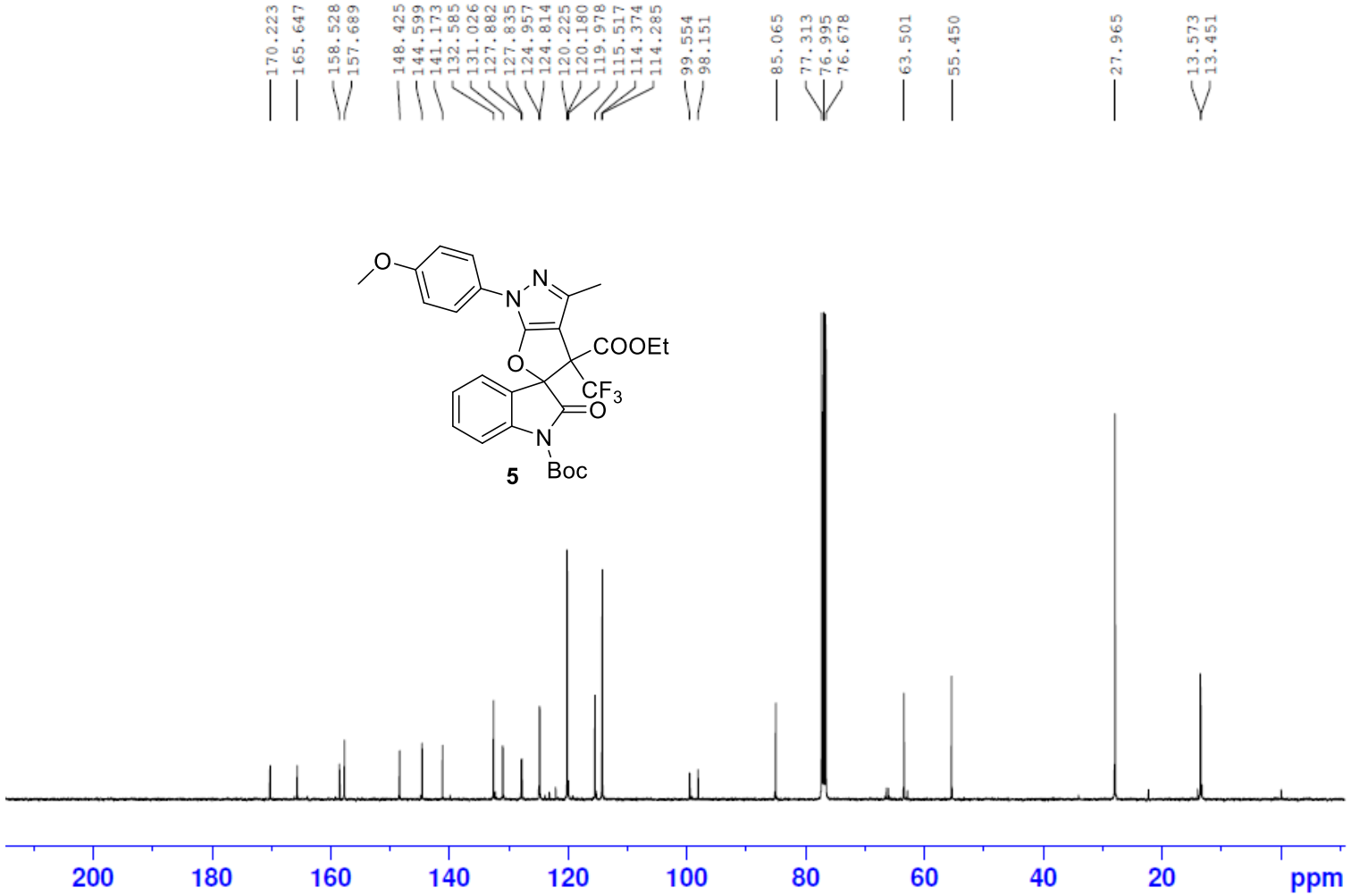
4: Diode Array
Range: 1.265e+2

Time	Height	Area	Area%
2.35	102595	2726.00	0.06
2.50	148760	4098.82	0.08
2.57	797183	22559.94	0.46
2.66	126229	3763.10	0.08
2.82	17644118	526135.69	10.76
2.87	126538760	4326154.00	88.51
3.05	64886	2483.80	0.05



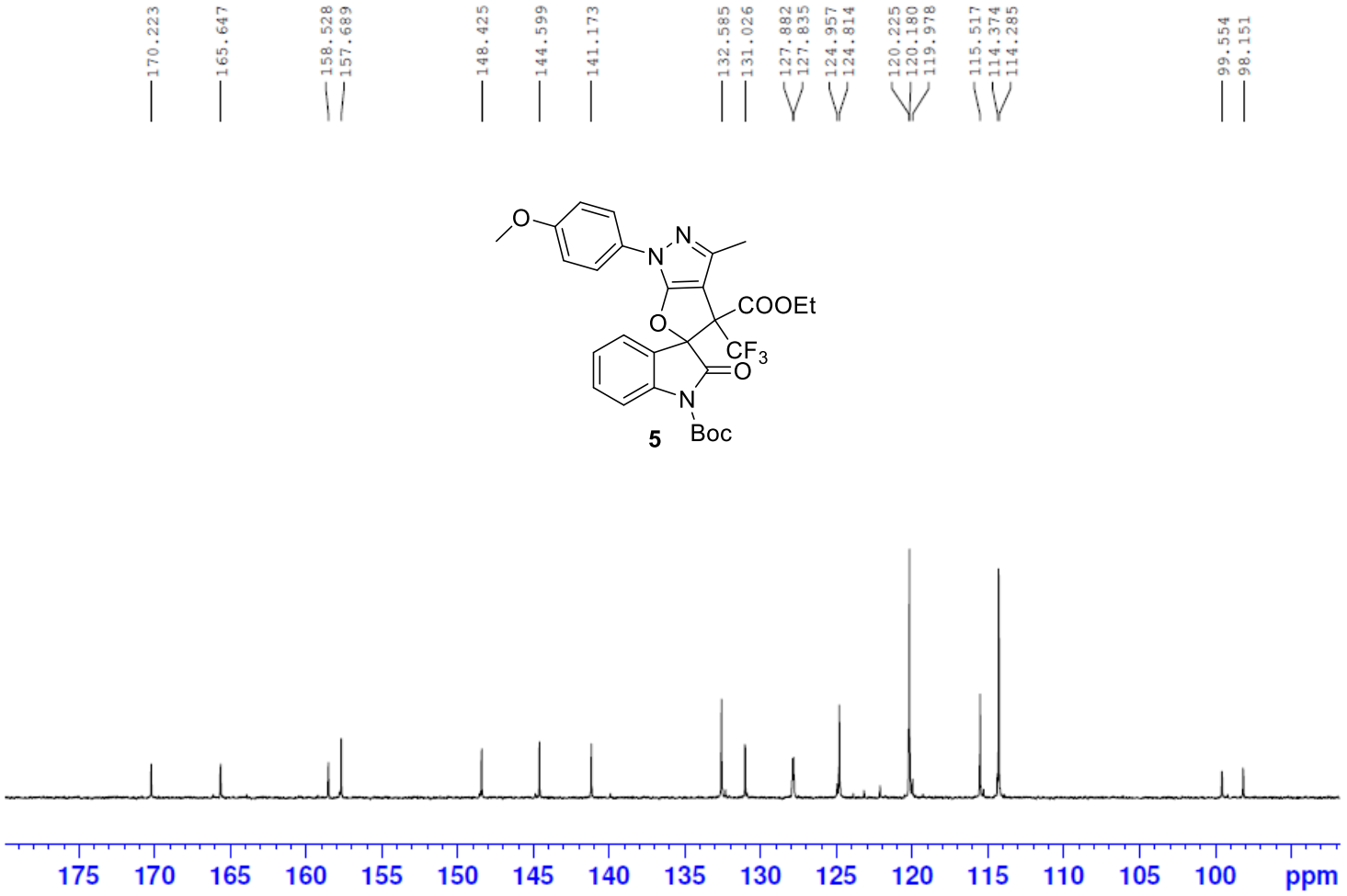
4 (1:9, dr)


8.026
8.005
7.735
7.716
7.543
7.526
7.521
7.286
7.266
7.248
6.886
6.864
5.289
4.292
4.275
4.263
4.257
4.246
4.228
3.780
2.390
2.339
1.675
1.653
1.600
1.302
1.284
1.267
0.968
0.951
0.933
0.898
0.881
0.863
-0.000
COOEt
CF3
O
N
Boc
5
11
10
9
8
7
6
5
4
3
2
1
ppm
0.92
0.96
2.92
1.16
2.00
1.87
3.05
2.92
9.22
3.13
Current Data Parameters
NAME 51200286737
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200215
Time 14.19 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 70.07
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ANL-MCL5-NMR-001



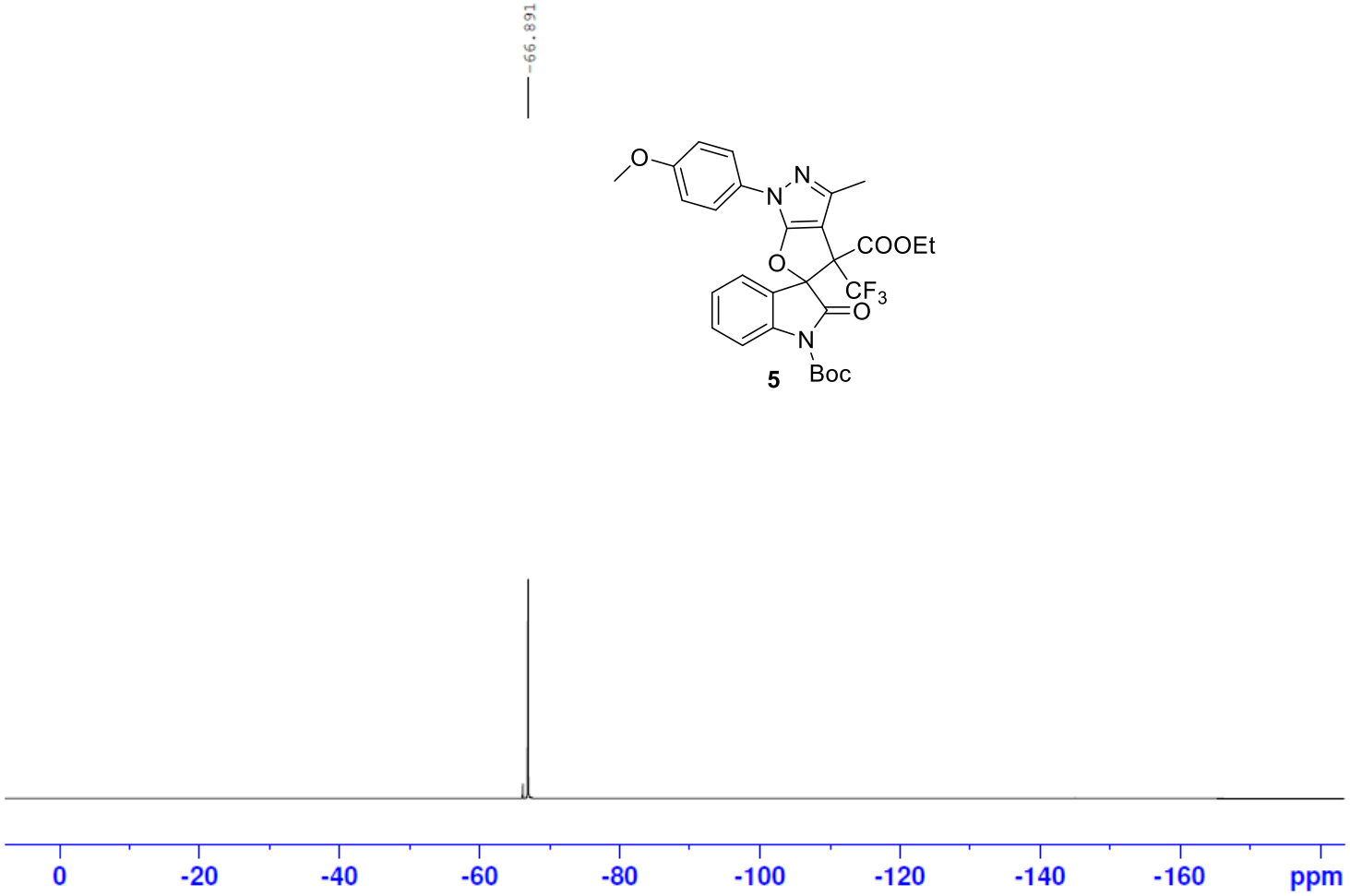

```
Current Data Parameters
NAME          512002B6737
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200215
Time               21.02 h
INSTRUM             spect
PROBHD   Z116098_0317 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3000
DS                      4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ            1.3631488 sec
RG               195.29
DW               20.800 usec
DE                6.50 usec
TE               300.1 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                     1
SFO1         100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1         77.83999634 W
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         16.43099976 W
PLW12         0.20286000 W
PLW13         0.10204000 W

F2 - Processing parameters
SI                  32768
SF           100.6127729 MHz
WDW                    EM
SSB      0
LB                 2.00 Hz
GB       0
PC                 1.40
```

ANL-MCL5-NMR-001




```
Current Data Parameters
NAME          512002B6737
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200215
Time                21.02 h
INSTRUM              spect
PROBHD    Z116098_0317 (
PULPROG            zqpg30
TD                  65536
SOLVENT             CDCl3
NS                   3000
DS                      4
SWH             24038.461 Hz
FIDRES          0.733596 Hz
AQ              1.3631488 sec
RG                 195.29
DW                 20.800 usec
DE                  6.50 usec
TE                 300.1 K
D1              3.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           100.6228298 MHz
NUC1                  13C
P1                 10.00 usec
PLW1           77.83999634 W
SFO2           400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           16.43099976 W
PLW12           0.20286000 W
PLW13           0.10204000 W

F2 - Processing parameters
SI                    32768
SF             100.6127729 MHz
WDW                      EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.40
```

ANL-MCL5-NMR-001



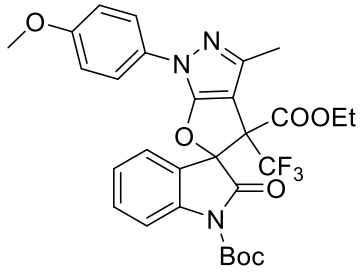

```
Current Data Parameters
NAME         512002B6737
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200215
Time               17.18 h
INSTRUM             spect
PROBHD   Z116098_0317 (
PULPROG             zgflqn
TD                  131072
SOLVENT               CDCl3
NS                     16
DS                      4
SWH              89285.711 Hz
FIDRES           1.362392 Hz
AQ               0.7340032 sec
RG                  195.29
DW                    5.600 usec
DE                    6.50 usec
TE                   298.1 K
D1             1.00000000 sec
TD0                    1
SFO1           376.4607164 MHz
NUC1                  19F
P1                   18.00 usec
PLW1          19.85499954 W

F2 - Processing parameters
SI                    65536
SF             376.4983662 MHz
WDW                     EM
SSB       0
LB                    2.00 Hz
GB        0
PC                    1.00
```

ANL-MCL5-NMR-001

GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services

SAMPLE ID:TP-11580-017
14022020-129
ACQ.METHOD:FA- 4.2 MIN
512002B6736A

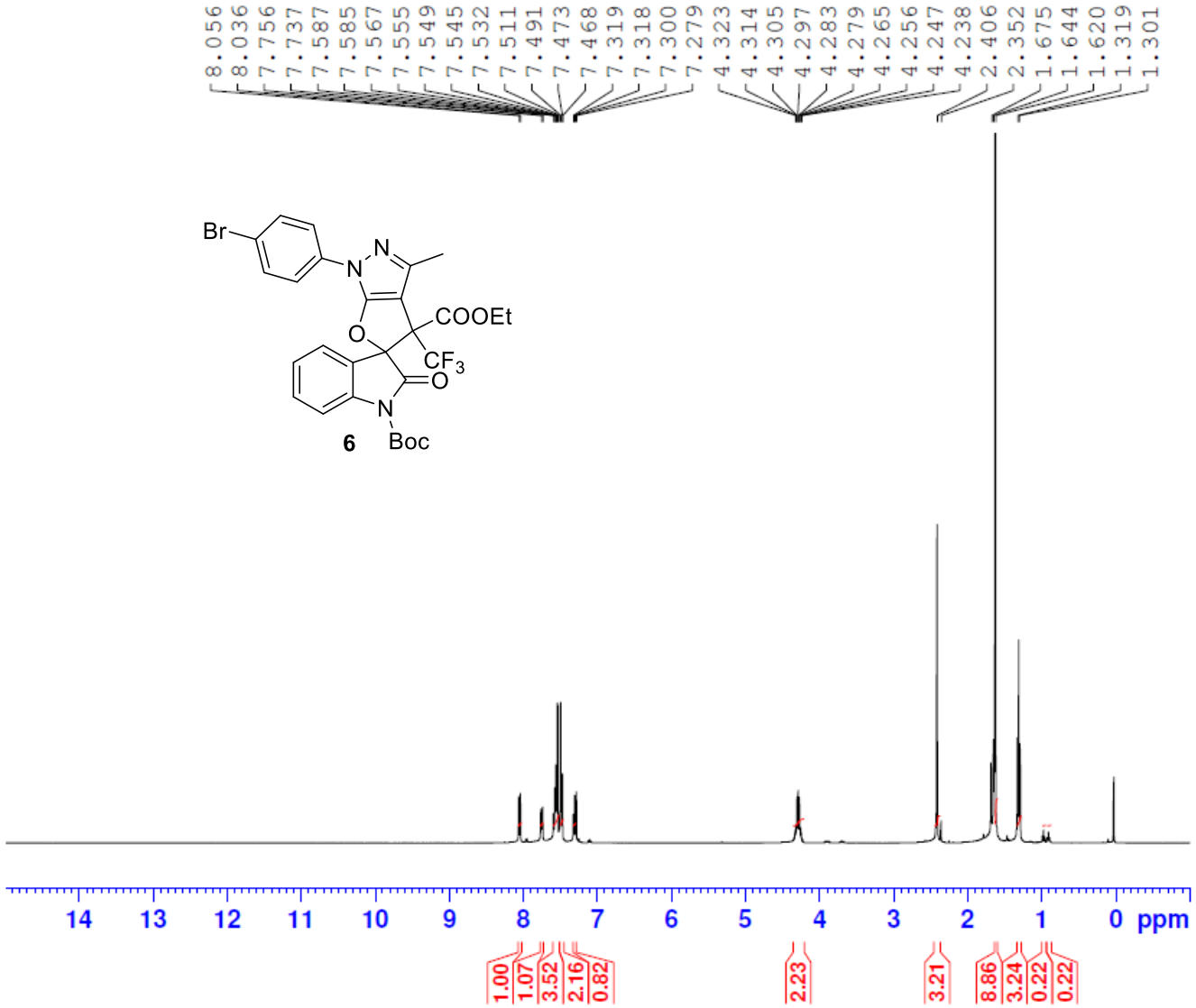
Date of analysis: 14-Feb-202021:42:18
Instrument ID :ANL-MCL4-LCMS-SQD-1



5 (1:9, dr)

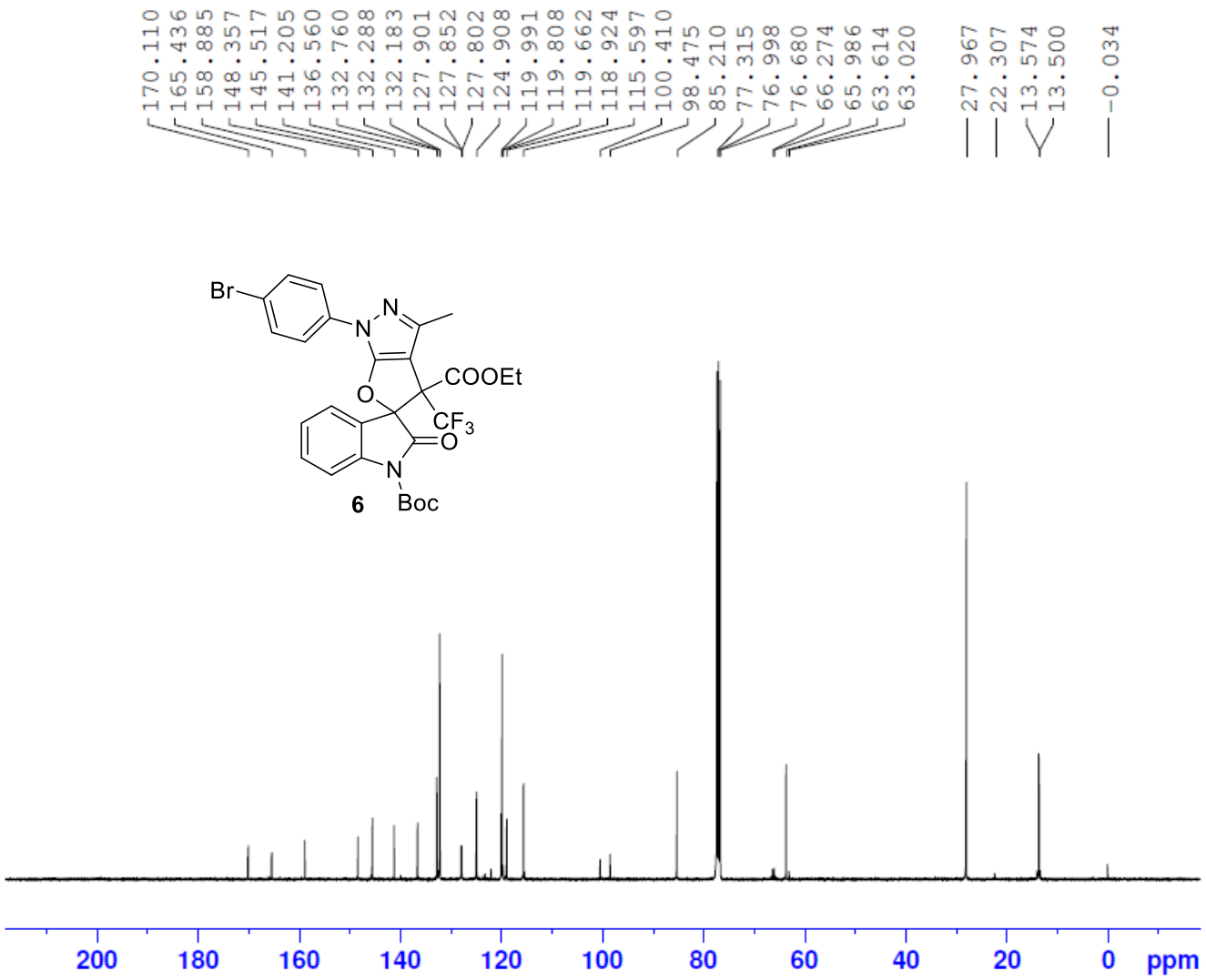
4: Diode Array
Range: 1.526e+2

Time	Height	Area	Area%
1.90	19780796	395161.50	9.77
1.97	2849348	210361.95	5.20
2.11	502126	23578.44	0.58
2.18	280289	7312.86	0.18
2.21	274476	4993.27	0.12
2.25	340984	14923.98	0.37
2.28	368767	13635.05	0.34
2.33	419813	21273.03	0.53
2.40	1135492	18640.18	0.46
2.41	1164010	65485.70	1.62
2.67	349125	5229.05	0.13
2.71	16149302	286525.38	7.09
2.76	152456080	2973384.00	73.54
2.86	165019	2975.28	0.07



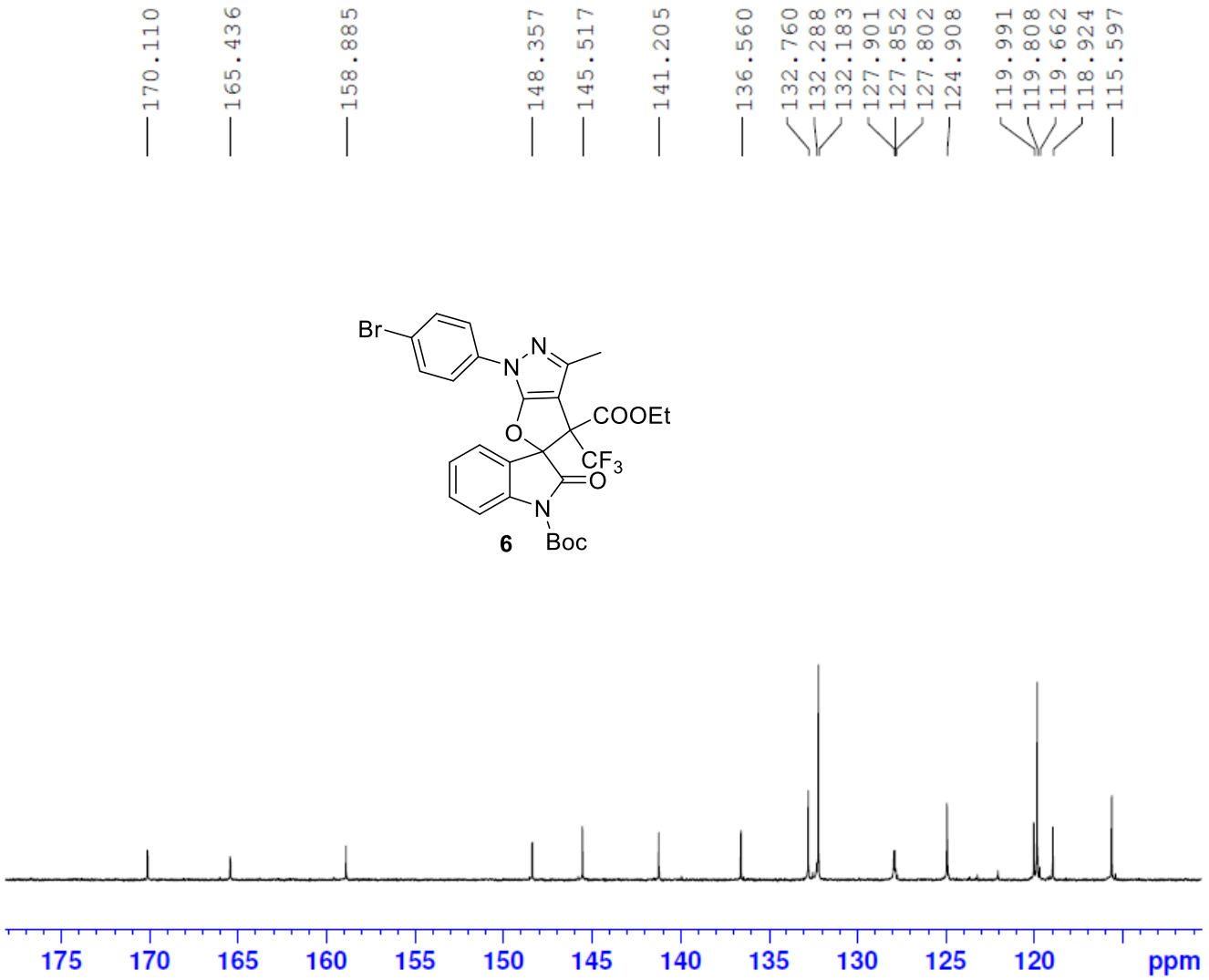

```
Current Data Parameters
NAME         512002A8067
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200207
Time              21.32 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                74.2626
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.3024719 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                 65536
SF            400.3000026 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-003



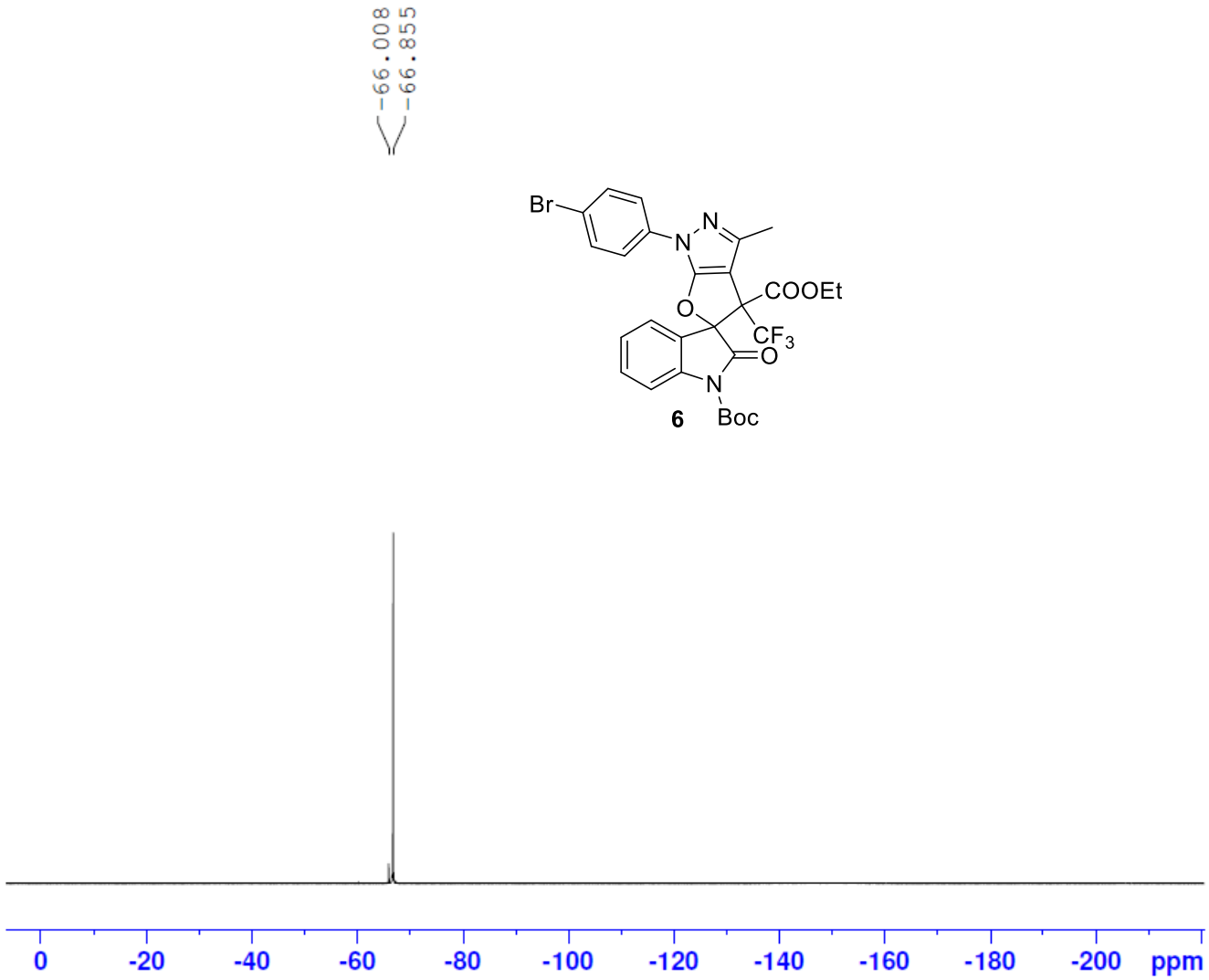

```
Current Data Parameters
NAME          512002A8067
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200208
Time                11.34 h
INSTRUM            Avance
PROBHD    Z163739_0135 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3072
DS                      4
SWH             23809.523 Hz
FIDRES          0.726609 Hz
AQ              1.3762560 sec
RG                    101
DW                 21.000 usec
DE                  6.50 usec
TE                 298.2 K
D1              3.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           100.6655806 MHz
NUC1                  13C
P0                  2.67 usec
P1                  8.00 usec
PLW1           92.65799713 W
SFO2           400.3016012 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2              90.00 usec
PLW2           22.47400093 W
PLW12           0.17757000 W
PLW13           0.08931800 W

F2 - Processing parameters
SI                   32768
SF             100.6555189 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

ANL-MCL5-NMR-003



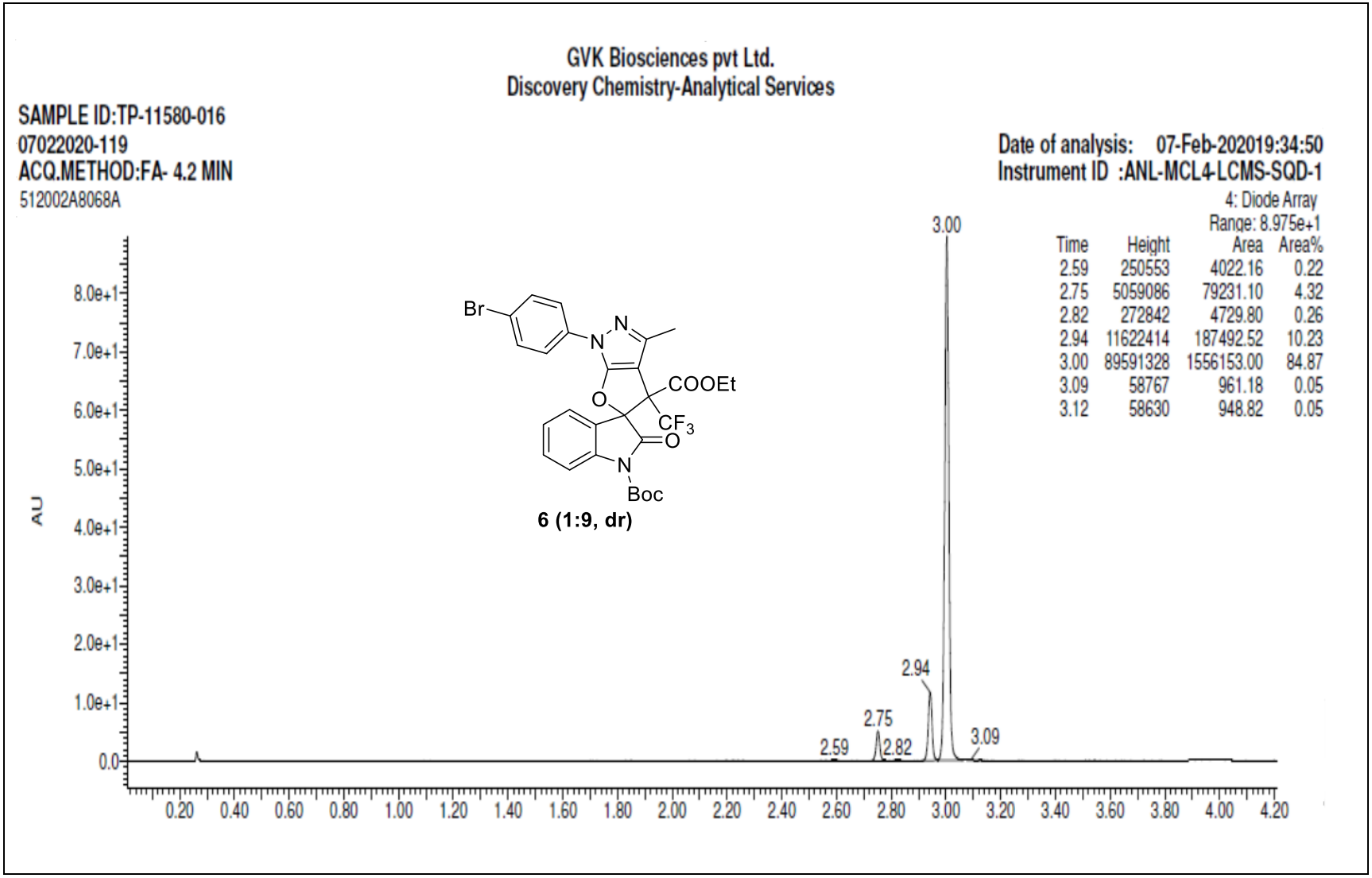

```
Current Data Parameters
NAME         512002A8067
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time              11.34 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 3072
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.2 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6655806 MHz
NUC1                13C
P0                2.67 usec
P1                8.00 usec
PLW1         92.65799713 W
SFO2        400.3016012 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.47400093 W
PLW12         0.17757000 W
PLW13         0.08931800 W

F2 - Processing parameters
SI                 32768
SF          100.6555189 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

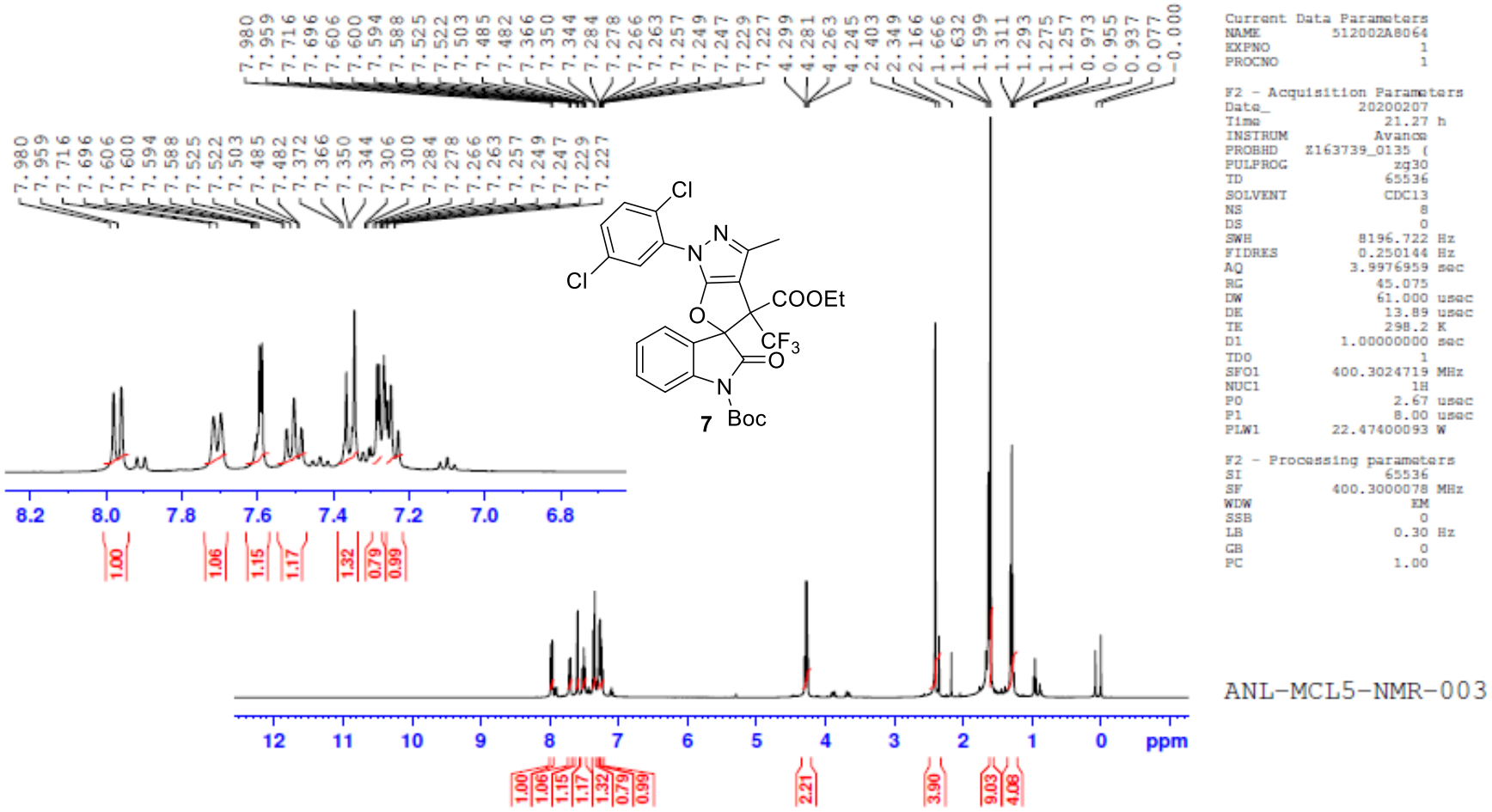
ANL-MCL5-NMR-003




```
Current Data Parameters
NAME         512002A8067
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time                7.45 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG               zg
TD                131072
SOLVENT            CDC13
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                298.2 K
D1            1.00000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1         37.49499893 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

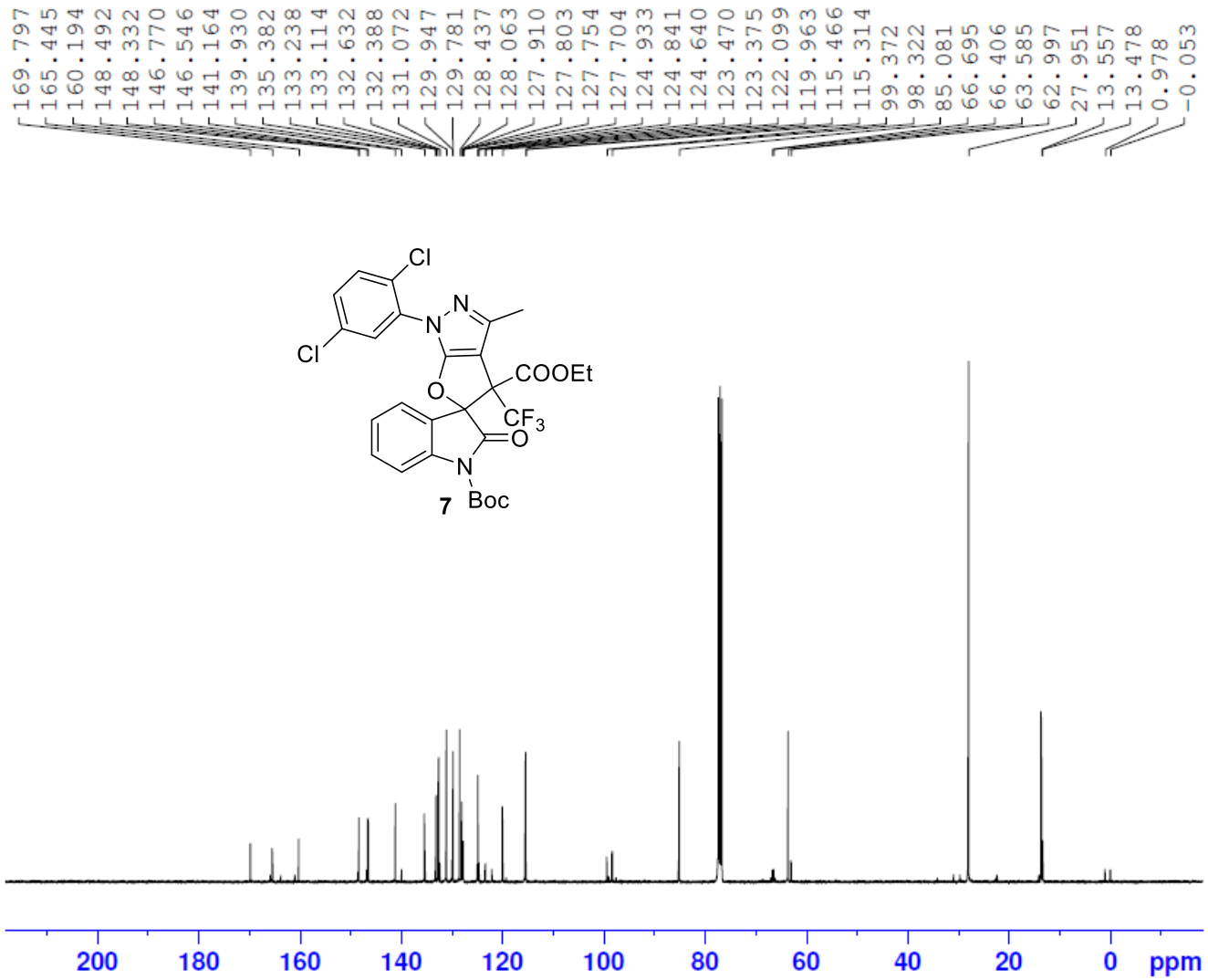
ANL-MCL5-NMR-003


GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services
SAMPLE ID:TP-11580-016
07022020-119
ACQ.METHOD:FA- 4.2 MIN
512002A8068A
Date of analysis: 07-Feb-202019:34:50
Instrument ID :ANL-MCL4-LCMS-SQD-1
4: Diode Array
Range: 8.975e+1
Time Height Area Area%
2.59 250553 4022.16 0.22
2.75 5059086 79231.10 4.32
2.82 272842 4729.80 0.26
2.94 11622414 187492.52 10.23
3.00 89591328 1556153.00 84.87
3.09 58767 961.18 0.05
3.12 58630 948.82 0.05
Br
N
N
O
COOEt
CF3
O
N
Boc
6 (1:9, dr)
AU
8.0e+1
7.0e+1
6.0e+1
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
3.00
2.94
2.75
2.59
2.82
3.09
0.20 0.40 0.60 0.80 1.00 1.20 1.40 1.60 1.80 2.00 2.20 2.40 2.60 2.80 3.00 3.20 3.40 3.60 3.80 4.00 4.20



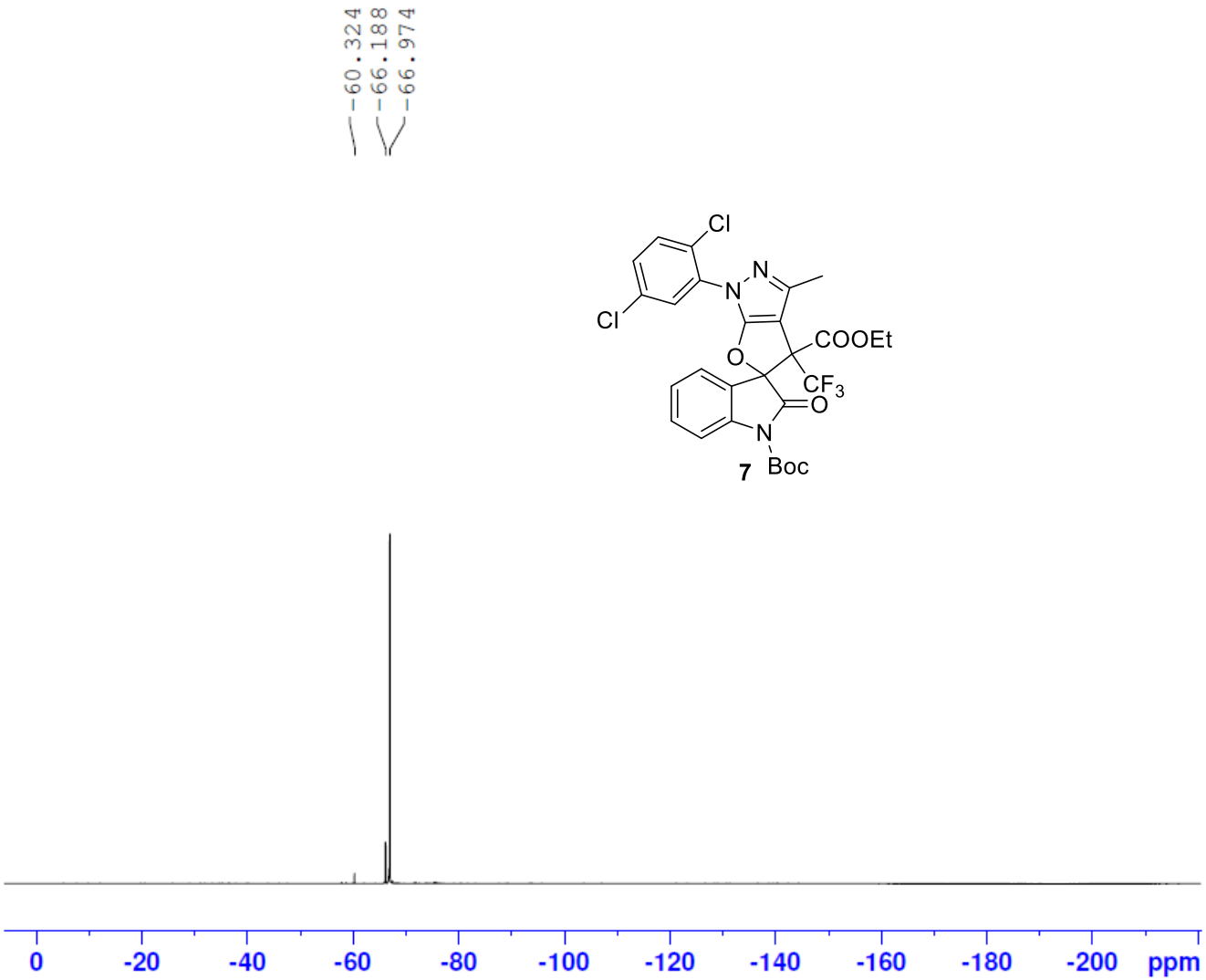

```
Current Data Parameters
NAME          512002A8064
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200207
Time                21.27 h
INSTRUM             Avance
PROBHD    Z163739_0135 (
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8196.722 Hz
FIDRES            0.250144 Hz
AQ                3.9976959 sec
RG                    45.075
DW                    61.000 usec
DE                    13.89 usec
TE                    298.2 K
D1             1.00000000 sec
TD0                      1
SFO1           400.3024719 MHz
NUC1                    1H
P0                    2.67 usec
P1                    8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                   65536
SF             400.3000078 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

ANL-MCL5-NMR-003



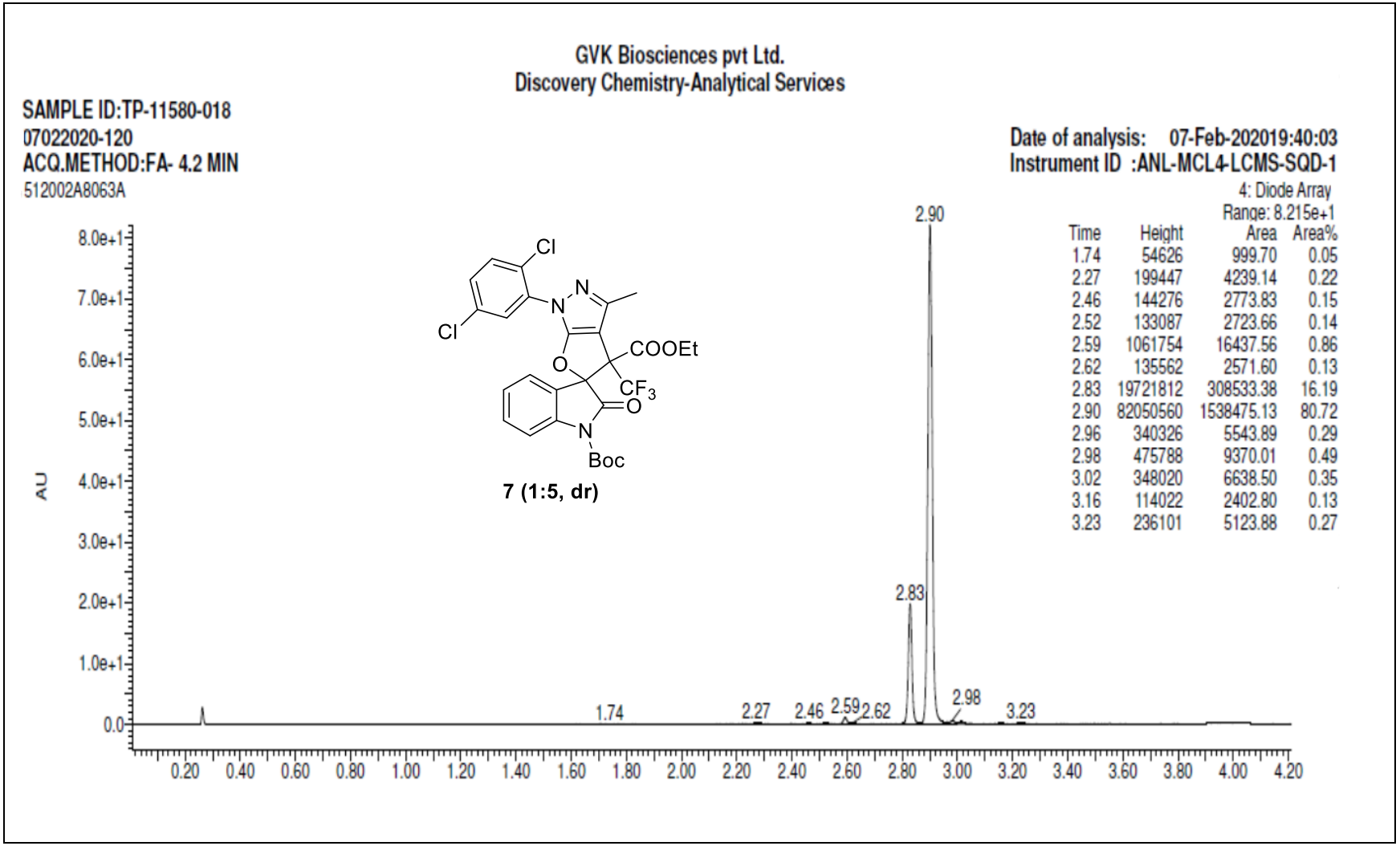

```
Current Data Parameters
NAME         512002A8064
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time                7.38 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 3072
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6655806 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1         92.65799713 W
SFO2         400.3016012 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.47400093 W
PLW12         0.17757000 W
PLW13         0.08931800 W

F2 - Processing parameters
SI                 32768
SF          100.6555203 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

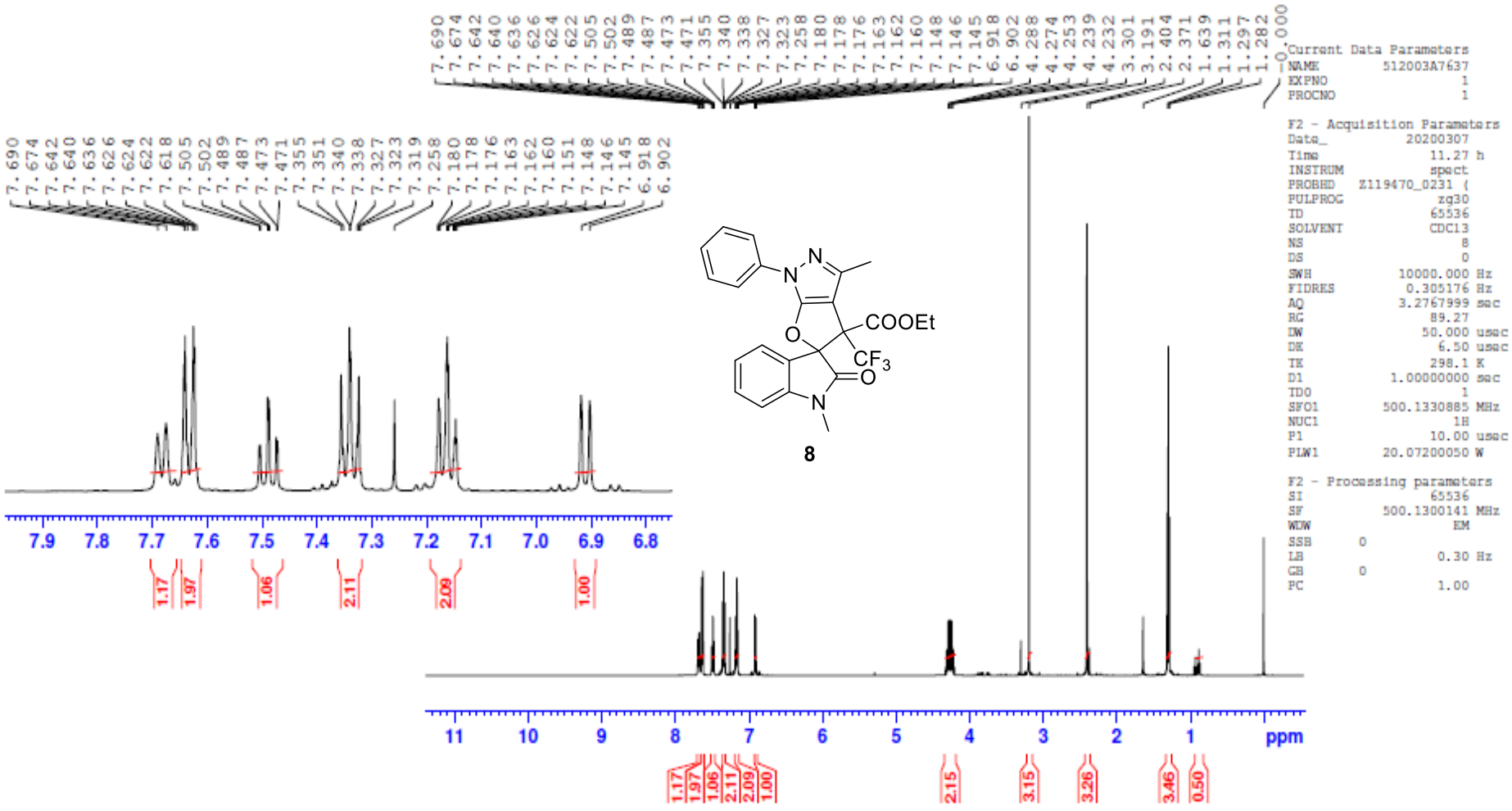
ANL-MCL5-NMR-003



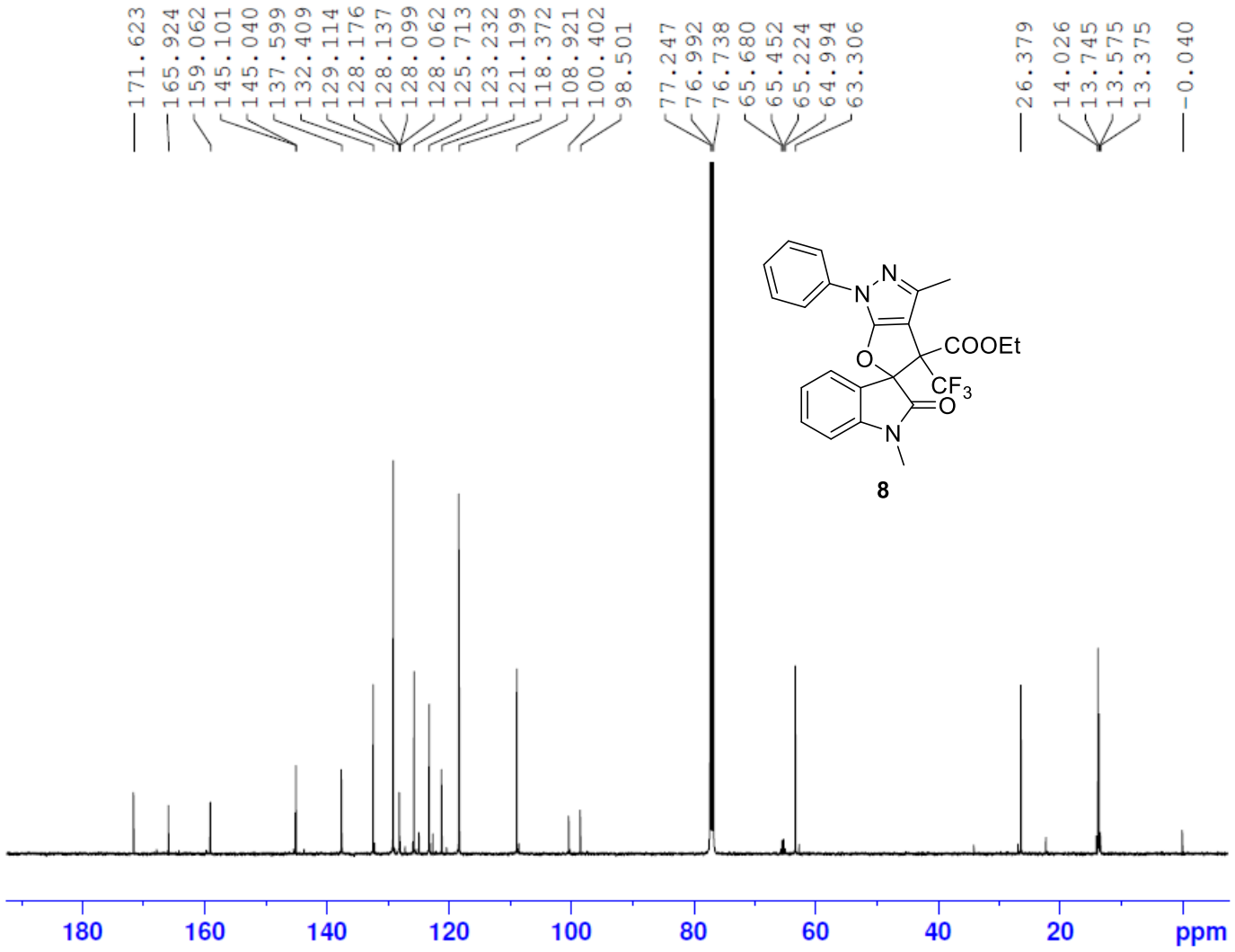

```
Current Data Parameters
NAME         512002A8064
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200208
Time                7.40 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG               zg
TD                131072
SOLVENT            CDC13
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                 12.00 usec
PLW1         37.49499893 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-003


GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services
SAMPLE ID:TP-11580-018
07022020-120
ACQ.METHOD:FA- 4.2 MIN
512002A8063A
Date of analysis: 07-Feb-202019:40:03
Instrument ID :ANL-MCL4-LCMS-SQD-1
4: Diode Array
Range: 8.215e+1
Time Height Area Area%
1.74 54626 999.70 0.05
2.27 199447 4239.14 0.22
2.46 144276 2773.83 0.15
2.52 133087 2723.66 0.14
2.59 1061754 16437.56 0.86
2.62 135562 2571.60 0.13
2.83 19721812 308533.38 16.19
2.90 82050560 1538475.13 80.72
2.96 340326 5543.89 0.29
2.98 475788 9370.01 0.49
3.02 348020 6638.50 0.35
3.16 114022 2402.80 0.13
3.23 236101 5123.88 0.27
Cl
Cl
N
N
O
COOEt
CF3
O
N
Boc
7 (1:5, dr)
AU
8.0e+1
7.0e+1
6.0e+1
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
2.90
2.83
1.74
2.27
2.46
2.59
2.62
2.98
3.23
0.20 0.40 0.60 0.80 1.00 1.20 1.40 1.60 1.80 2.00 2.20 2.40 2.60 2.80 3.00 3.20 3.40 3.60 3.80 4.00 4.20

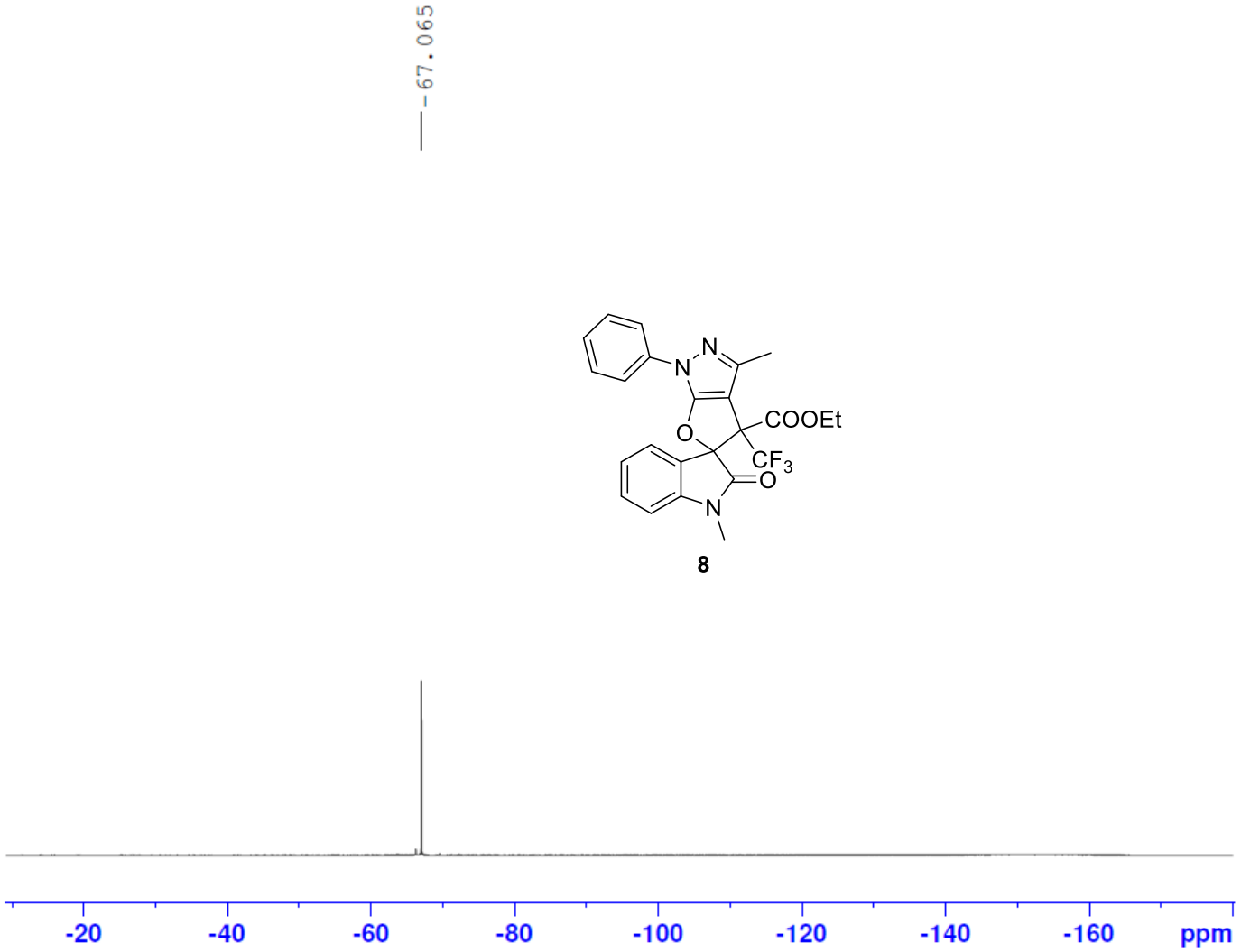

7.690 7.674 7.642 7.640 7.636 7.626 7.624 7.622 7.618 7.505 7.502 7.489 7.487 7.473 7.471 7.355 7.351 7.340 7.338 7.327 7.323 7.319 7.258 7.180 7.178 7.176 7.163 7.162 7.160 7.151 7.148 7.146 7.145 6.918 6.902
7.690 7.674 7.642 7.640 7.636 7.626 7.624 7.622 7.618 7.505 7.502 7.489 7.487 7.473 7.471 7.355 7.351 7.340 7.338 7.327 7.323 7.319 7.258 7.180 7.178 7.176 7.163 7.162 7.160 7.151 7.148 7.146 7.145 6.918 6.902
4.288 4.274 4.253 4.239 4.232 3.301 3.191 2.404 2.371 1.639 1.311 1.297 1.282 0.000
COOEt
CF3
O
N
8
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8
1.17 1.97 1.06 2.11 2.09 1.00
11 10 9 8 7 6 5 4 3 2 1 ppm
1.17 1.97 1.06 2.11 2.09 1.00 2.15 3.15 3.26 3.46 0.50
Current Data Parameters
NAME 512003A7637
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Data_ 20200307
Time 11.27 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 89.27
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 20.07200050 W
F2 - Processing parameters
SI 65536
SF 500.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TP-11580-024




```
Current Data Parameters
NAME          512003A7637
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200308
Time                7.26 h
INSTRUM             spect
PROBHD   Z119470_0231 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3072
DS                      4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                 197.72
DW                 16.800 usec
DE                   6.50 usec
TE                 298.2 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                     1
SFO1         125.7703643 MHz
NUC1                  13C
P1                  10.00 usec
PLW1         86.78700256 W
SFO2         500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         20.07200050 W
PLW12         0.31363001 W
PLW13         0.15775000 W

F2 - Processing parameters
SI                  32768
SF           125.7577943 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.40
```

TP-11580-024



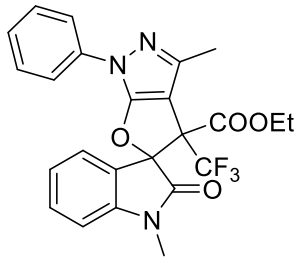
8

```
Current Data Parameters
NAME          512003A7637
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200308
Time                3.51 h
INSTRUM             spect
PROBHD    Z119470_0231 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ              0.5767168 sec
RG                 197.72
DW                  4.400 usec
DE                  6.50 usec
TE                 298.2 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                     1
SFO1          470.5453180 MHz
NUC1                  19F
P1                  15.00 usec
PLW1          34.86600113 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2          20.07200050 W
PLW12          0.31363001 W

F2 - Processing parameters
SI                  65536
SF            470.5923772 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services

SAMPLE ID:TP-11580-024
06032020-122
ACQ.METHOD:FA-4-2 MIN
512003A7636A

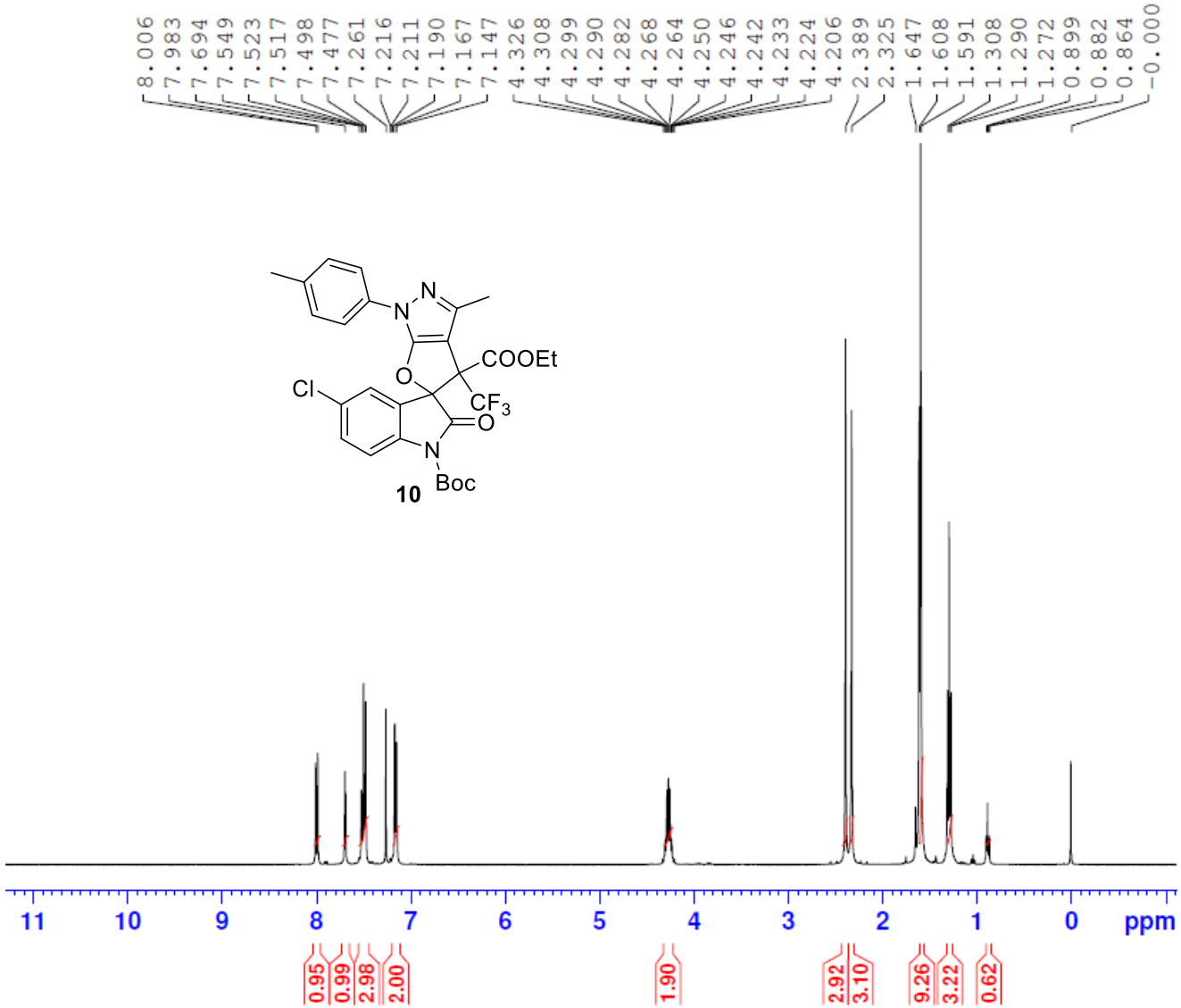
Date of analysis: 06-Mar-202019:58:48
Instrument ID :ANL-MCL4-LCMS-SQD-1



8 (6:92, dr)

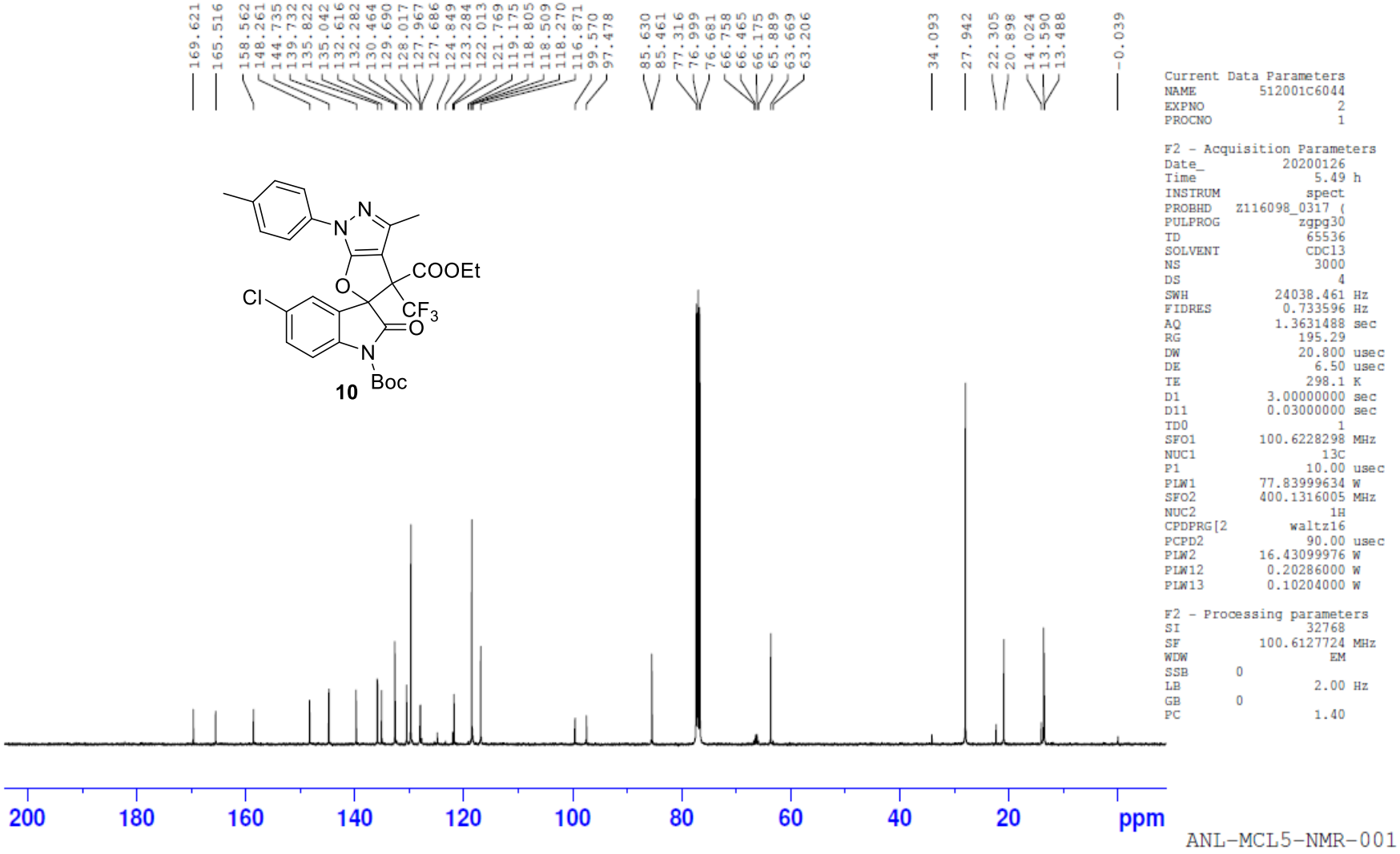
4: Diode Array
Range: 1.216e+2

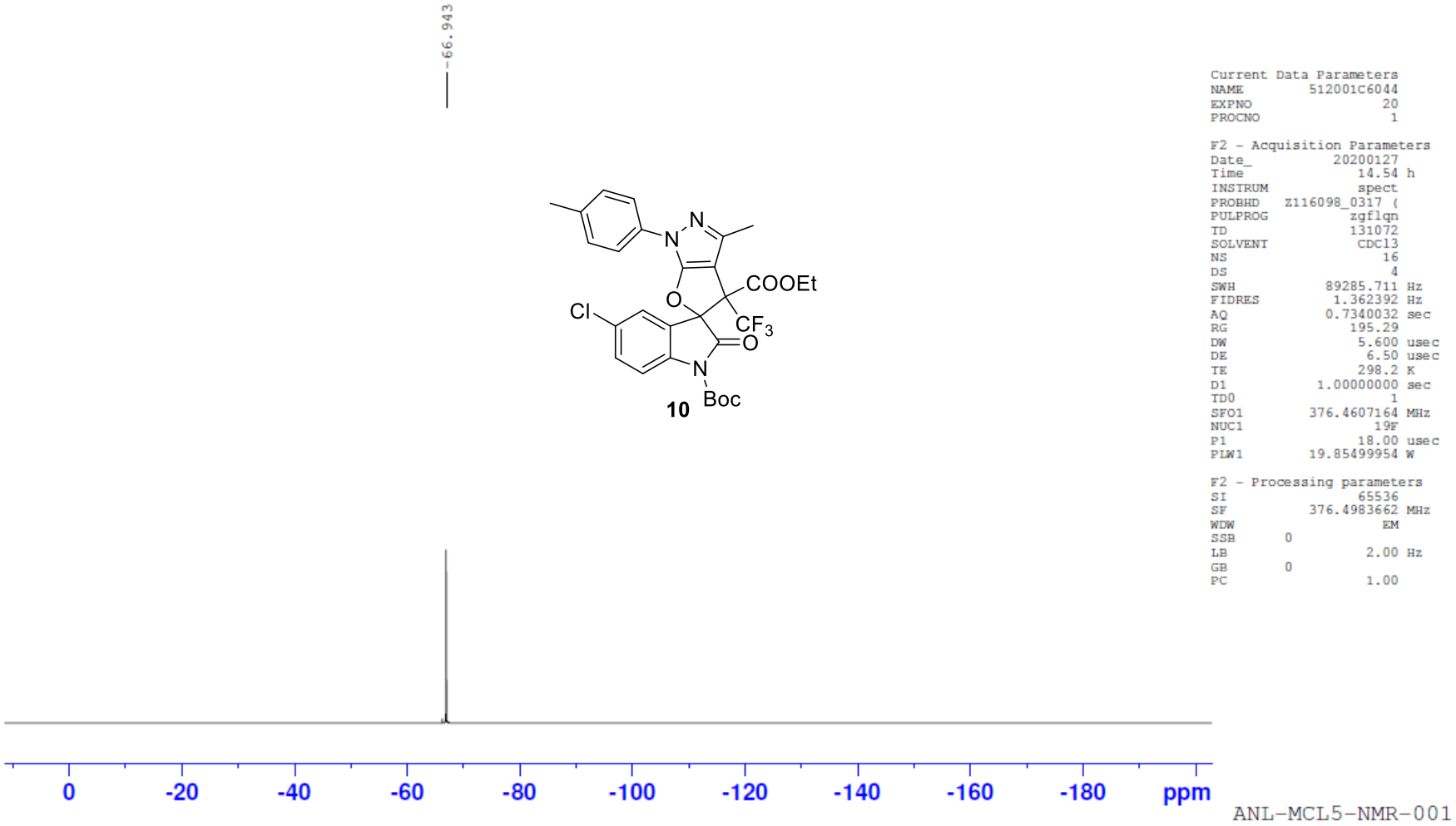
Time	Height	Area	Area%
1.86	371492	10955.77	0.26
1.92	148982	6587.56	0.16
2.01	99696	2282.14	0.05
2.16	185599	5735.08	0.14
2.27	220748	5194.82	0.12
2.29	252645	6955.57	0.17
2.40	7990242	236631.16	5.68
2.46	121521880	3850541.75	92.50
2.70	301652	12297.66	0.30
2.74	138224	3762.32	0.09
2.81	174660	5134.13	0.12
2.87	196286	6627.27	0.16
3.21	212627	9874.71	0.24




```
Current Data Parameters
NAME         512001B7990
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200120
Time                7.59 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.3024719 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                 65536
SF            400.3000097 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

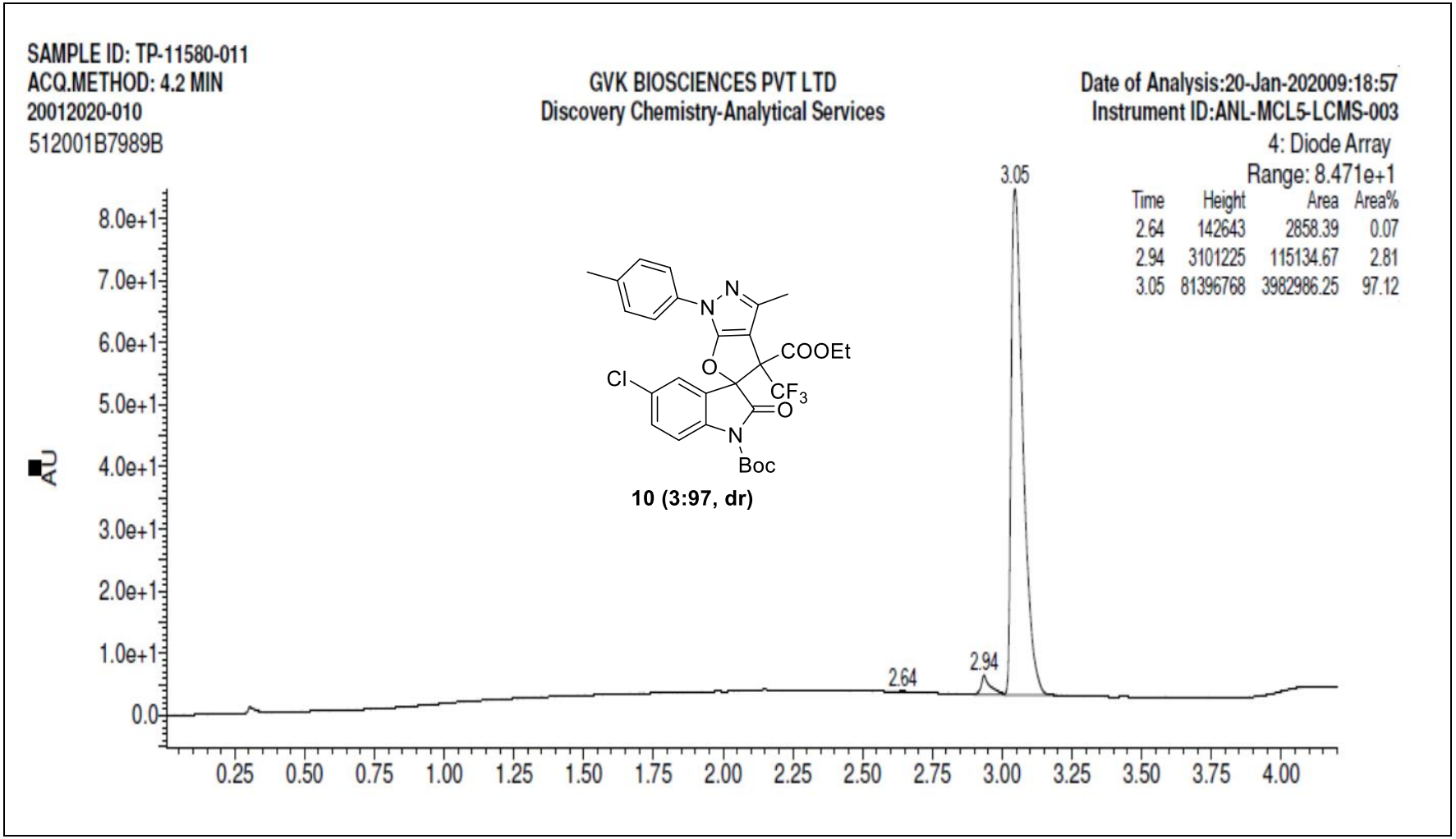
ANL-MCL5-NMR-003

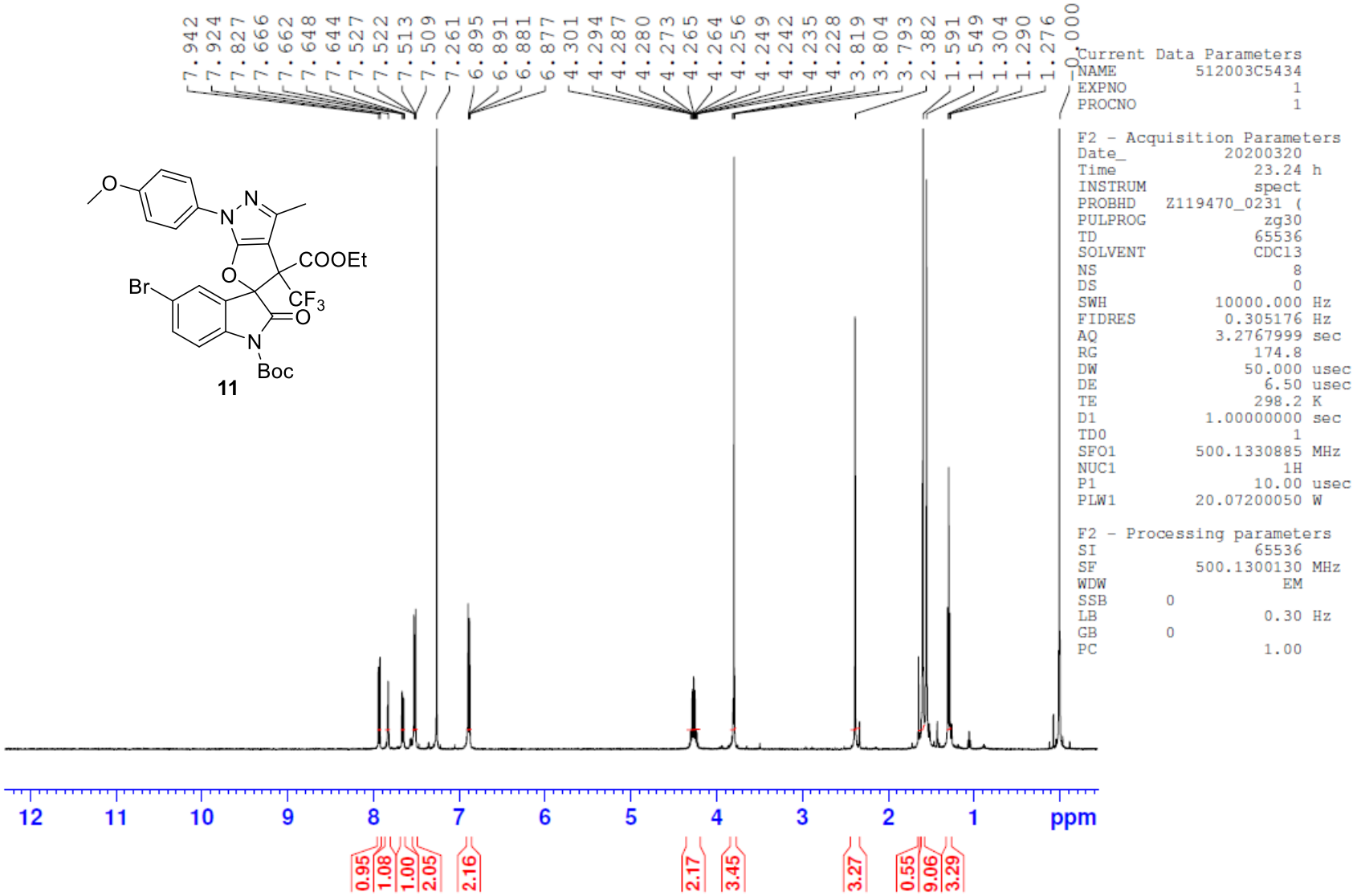

169.621
165.516
158.562
148.261
144.735
139.732
135.822
135.042
132.616
132.282
130.464
129.690
128.017
127.967
127.686
124.849
123.284
122.013
121.769
119.175
118.805
118.509
118.270
116.871
99.570
97.478
85.630
85.461
77.316
76.999
76.681
66.758
66.465
66.175
65.889
63.669
63.206
34.093
27.942
22.305
20.898
14.024
13.590
13.488
-0.039
COOEt
Cl
CF3
O
N
Boc
10
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME 512001c6044
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200126
Time 5.49 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 195.29
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 77.83999634 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.43099976 W
PLW12 0.20286000 W
PLW13 0.10204000 W
F2 - Processing parameters
SI 32768
SF 100.6127724 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
ANL-MCL5-NMR-001

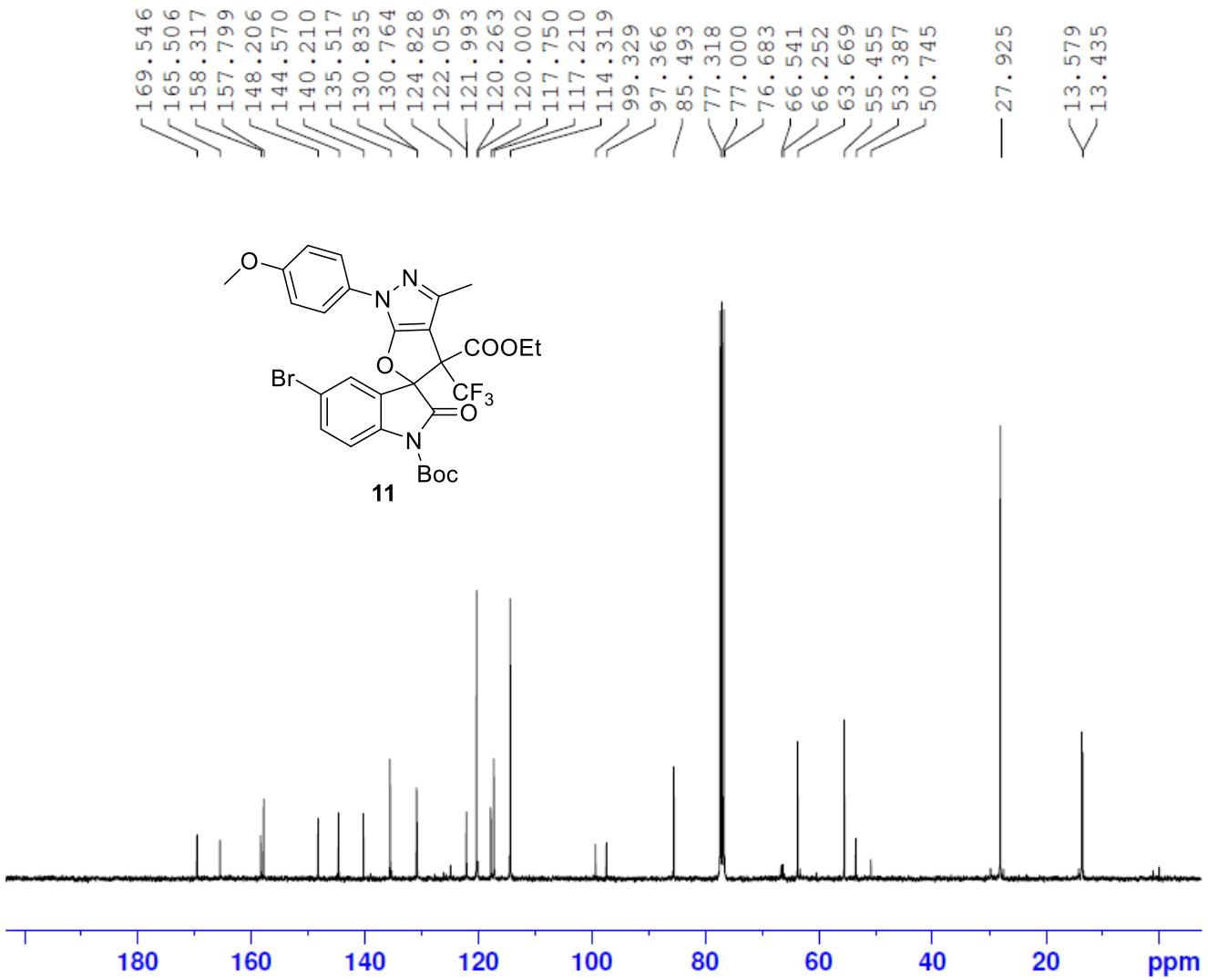



```
Current Data Parameters
NAME         512001C6044
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200127
Time              14.54 h
INSTRUM             spect
PROBHD   Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT             CDCl3
NS                    16
DS                     4
SWH           89285.711 Hz
FIDRES         1.362392 Hz
AQ             0.7340032 sec
RG                195.29
DW                 5.600 usec
DE                 6.50 usec
TE                298.2 K
D1            1.00000000 sec
TD0                    1
SFO1         376.4607164 MHz
NUC1                 19F
P1                18.00 usec
PLW1         19.85499954 W

F2 - Processing parameters
SI                 65536
SF           376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-001

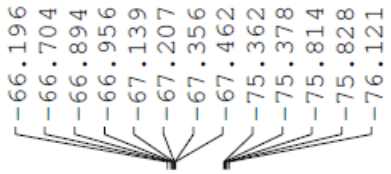

SAMPLE ID: TP-11580-011
ACQ.METHOD: 4.2 MIN
20012020-010
512001B7989B
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:20-Jan-202009:18:57
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 8.471e+1
Time Height Area Area%
2.64 142643 2858.39 0.07
2.94 3101225 115134.67 2.81
3.05 81396768 3982986.25 97.12
3.05
2.64
2.94
AU
8.0e+1
7.0e+1
6.0e+1
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
0.25 0.50 0.75 1.00 1.25 1.50 1.75 2.00 2.25 2.50 2.75 3.00 3.25 3.50 3.75 4.00
COOEt
Cl
CF3
O
N
Boc
10 (3:97, dr)

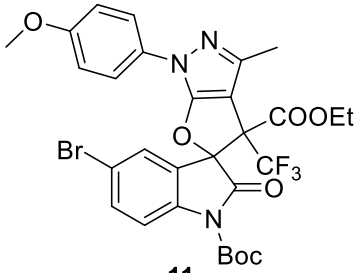

7.942
7.924
7.827
7.666
7.662
7.648
7.644
7.527
7.522
7.513
7.509
7.261
6.895
6.891
6.881
6.877
4.301
4.294
4.287
4.280
4.273
4.265
4.264
4.256
4.249
4.242
4.235
4.228
3.819
3.804
3.793
2.382
1.591
1.549
1.304
1.290
1.276
0.000
Current Data Parameters
NAME 512003C5434
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200320
Time 23.24 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 174.8
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 20.07200050 W
F2 - Processing parameters
SI 65536
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
COOEt
Br
CF3
Boc
11
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.95
1.08
1.00
2.05
2.16
2.17
3.45
3.27
0.55
9.06
3.29




```
Current Data Parameters
NAME         512002D2025
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200302
Time                7.47 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  669
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         100.6655806 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1         92.65799713 W
SFO2         400.3016012 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2         22.47400093 W
PLW12         0.17757000 W
PLW13         0.08931800 W

F2 - Processing parameters
SI                 32768
SF           100.6555199 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

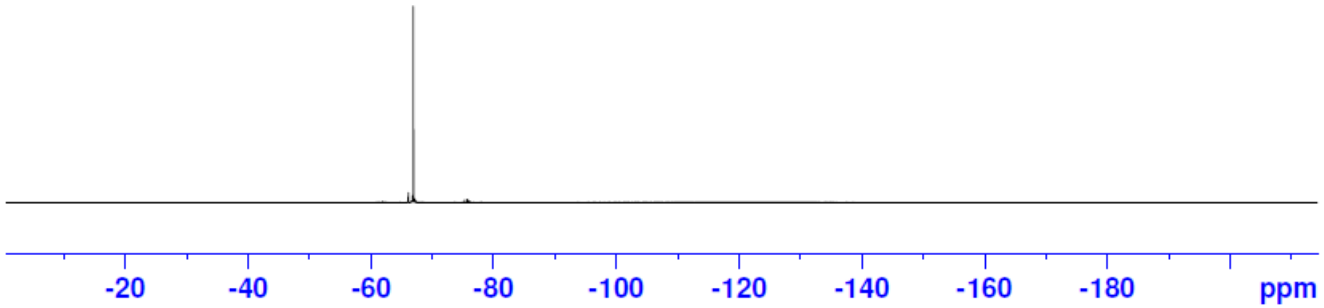
ANL-MCL5-NMR-003





11

```
Current Data Parameters
NAME         512002D2025
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200229
Time              13.13 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG               zg
TD                131072
SOLVENT             CDCl3
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                    1
SFO1          376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1          37.49499893 W

F2 - Processing parameters
SI                 65536
SF            376.6583260 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```



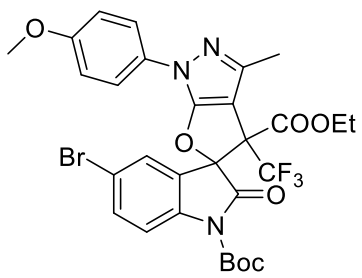
ANL-MCL5-NMR-003

SAMPLE ID: TP-11580-023
ACQ.METHOD: FA: 4.2 MIN
28022020-105
512002D2026A

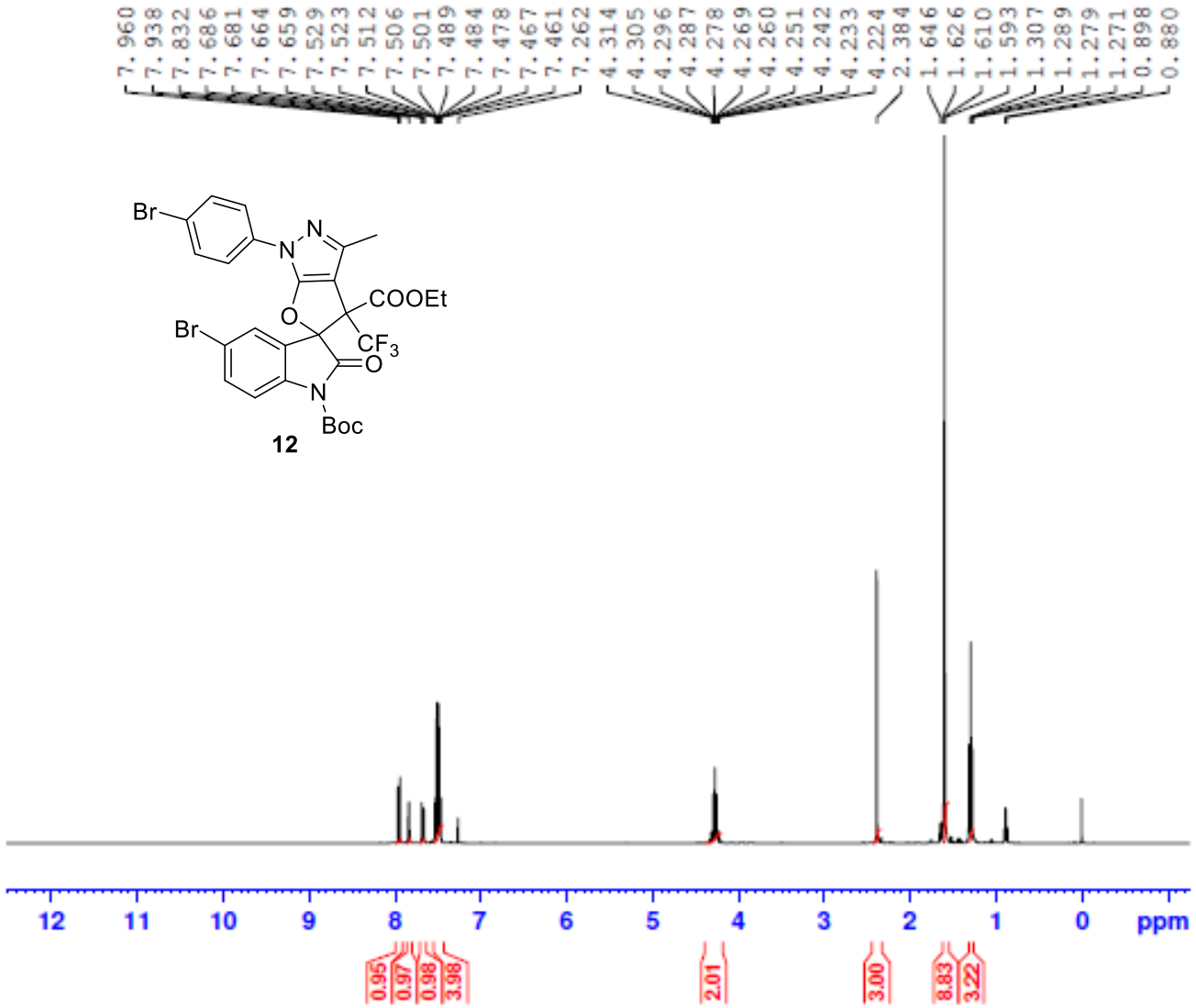
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services

Date of Analysis:28-Feb-202020:58:58
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 1.169e+2

Time	Height	Area	Area%
2.56	153200	2294.66	0.08
2.70	1888821	36096.11	1.22
2.77	1039072	34837.01	1.17
2.83	8349268	170205.09	5.74
2.93	116764920	2623951.00	88.49
3.02	2491206	78029.54	2.63
3.09	261843	7325.84	0.25
3.11	255981	10195.68	0.34
3.19	69405	2159.57	0.07

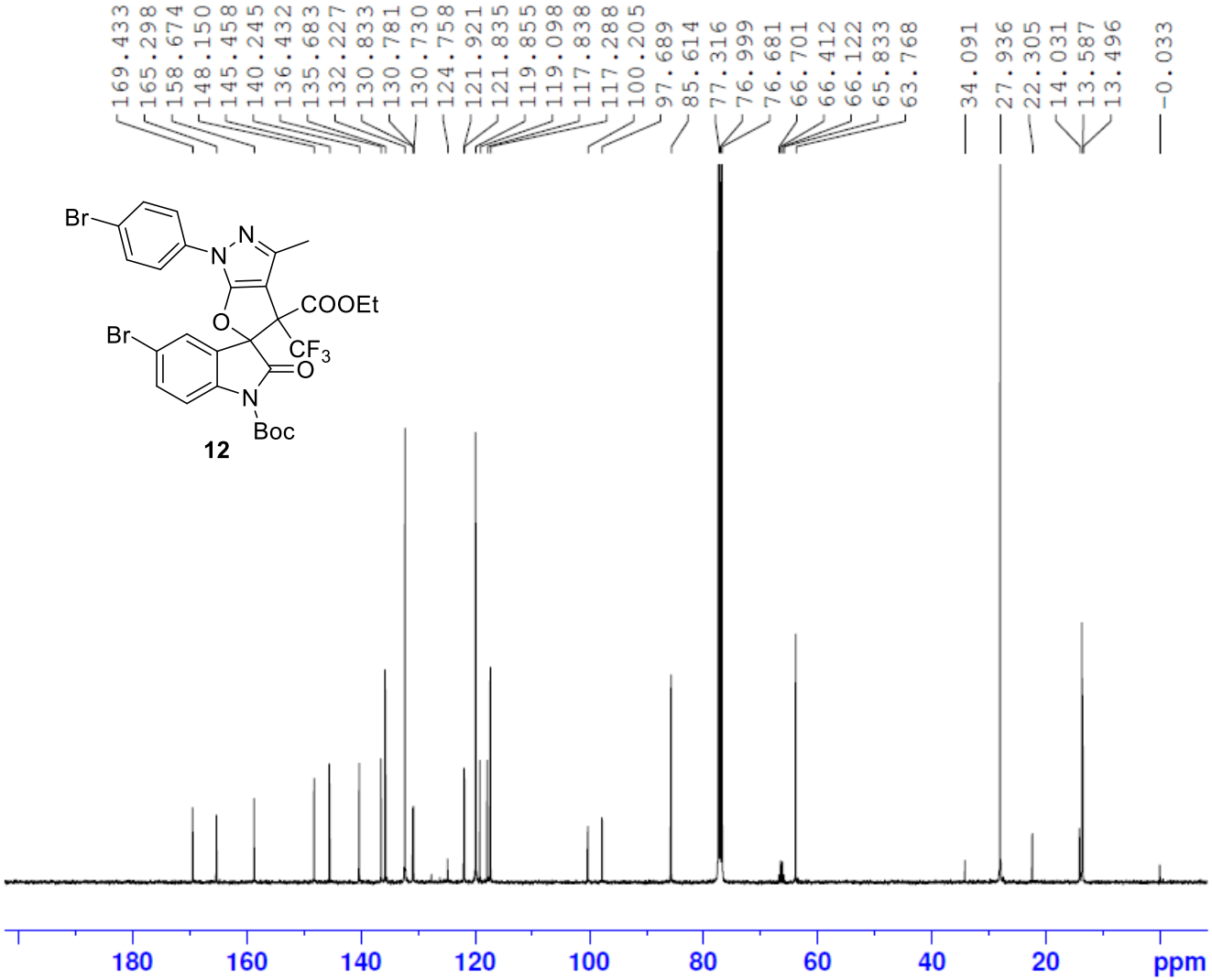


11 (6:88, dr)



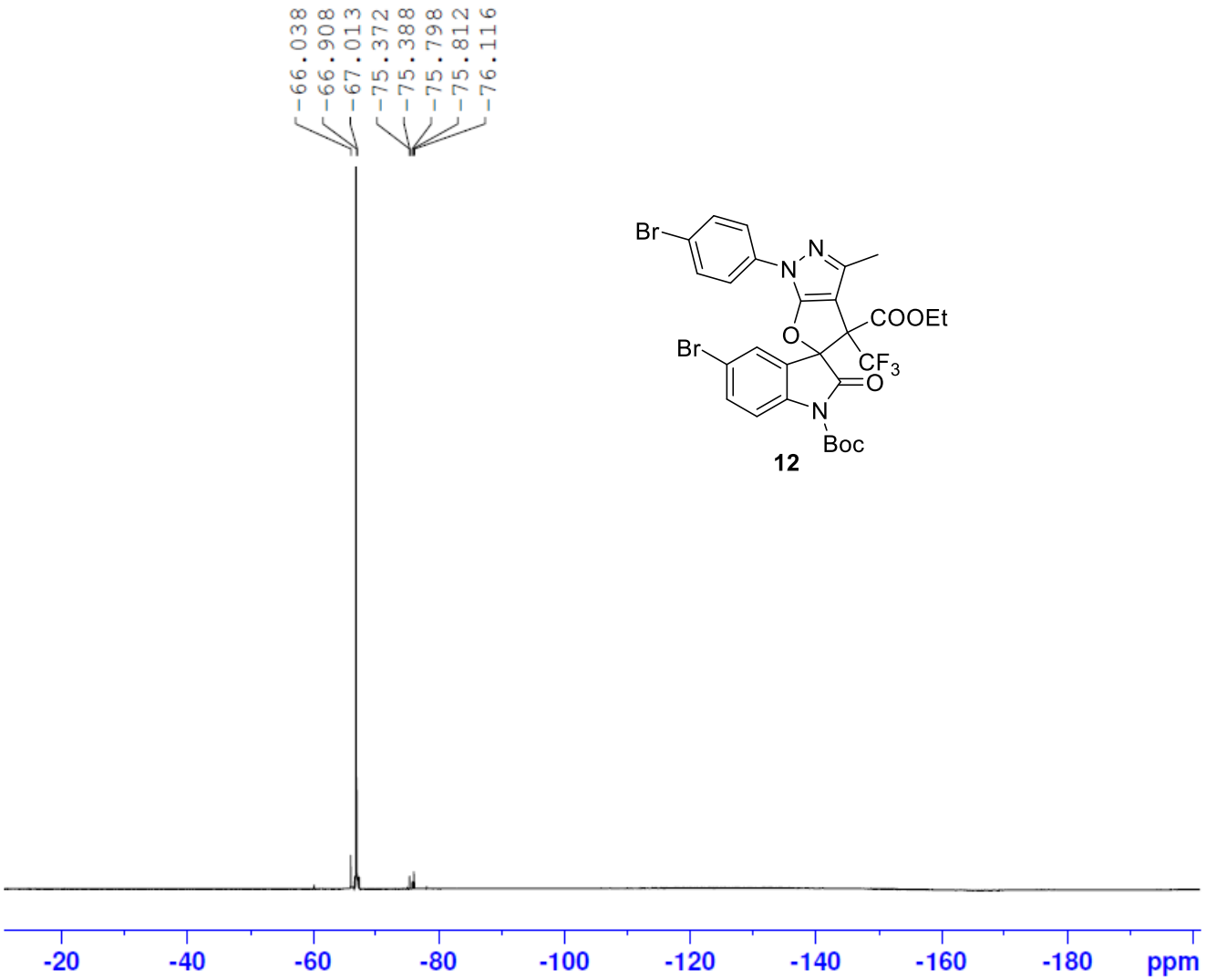

```
Current Data Parameters
NAME         512002D2027
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Data_             20200229
Time                12.50 h
INSTRUM             Avance
PROBHD    Z163739_0135 (
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       0
SWH               8196.722 Hz
FIDRES            0.250144 Hz
AQ                3.9976959 sec
RG                  56.4203
DW                  61.000 usec
DE                  13.89 usec
TE                  298.1 K
D1             1.00000000 sec
TD0                      1
SFO1          400.3024719 MHz
NUC1                    1H
P0                  2.67 usec
P1                  8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                   65536
SF            400.3000093 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

ANL-MCL5-NMR-003



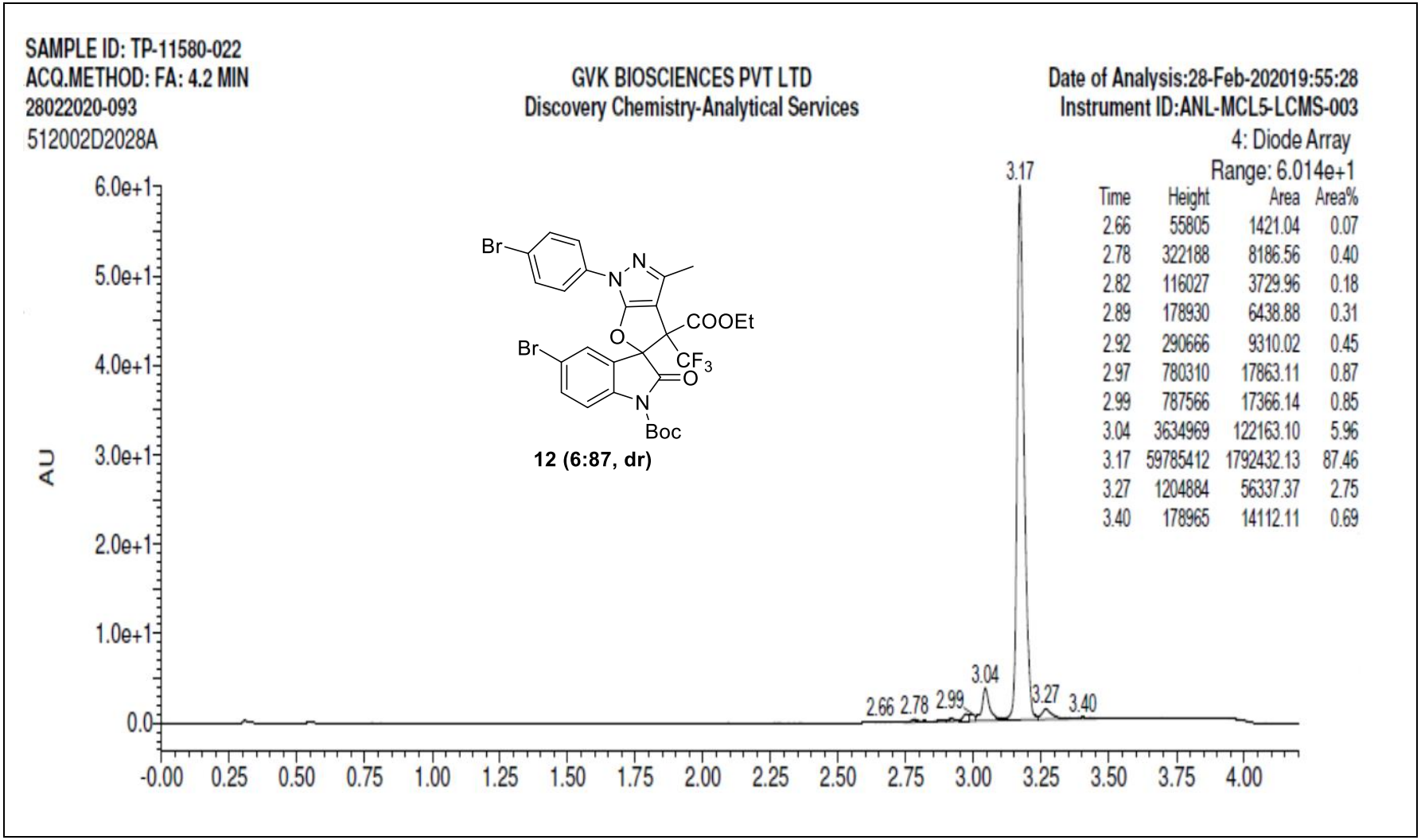

```
Current Data Parameters
NAME          512002D2027
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_              20200301
Time                  1.28 h
INSTRUM             Avance
PROBHD    Z163739_0135 (
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    3000
DS                       4
SWH             23809.523 Hz
FIDRES           0.726609 Hz
AQ               1.3762560 sec
RG                     101
DW                  21.000 usec
DE                   6.50 usec
TE                  298.1 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                      1
SFO1          100.6655806 MHz
NUC1                   13C
P0                   2.67 usec
P1                   8.00 usec
PLW1          92.65799713 W
SFO2          400.3016012 MHz
NUC2                   1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          22.47400093 W
PLW12          0.17757000 W
PLW13          0.08931800 W

F2 - Processing parameters
SI                   32768
SF            100.6555191 MHz
WDW                     EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   1.40
```

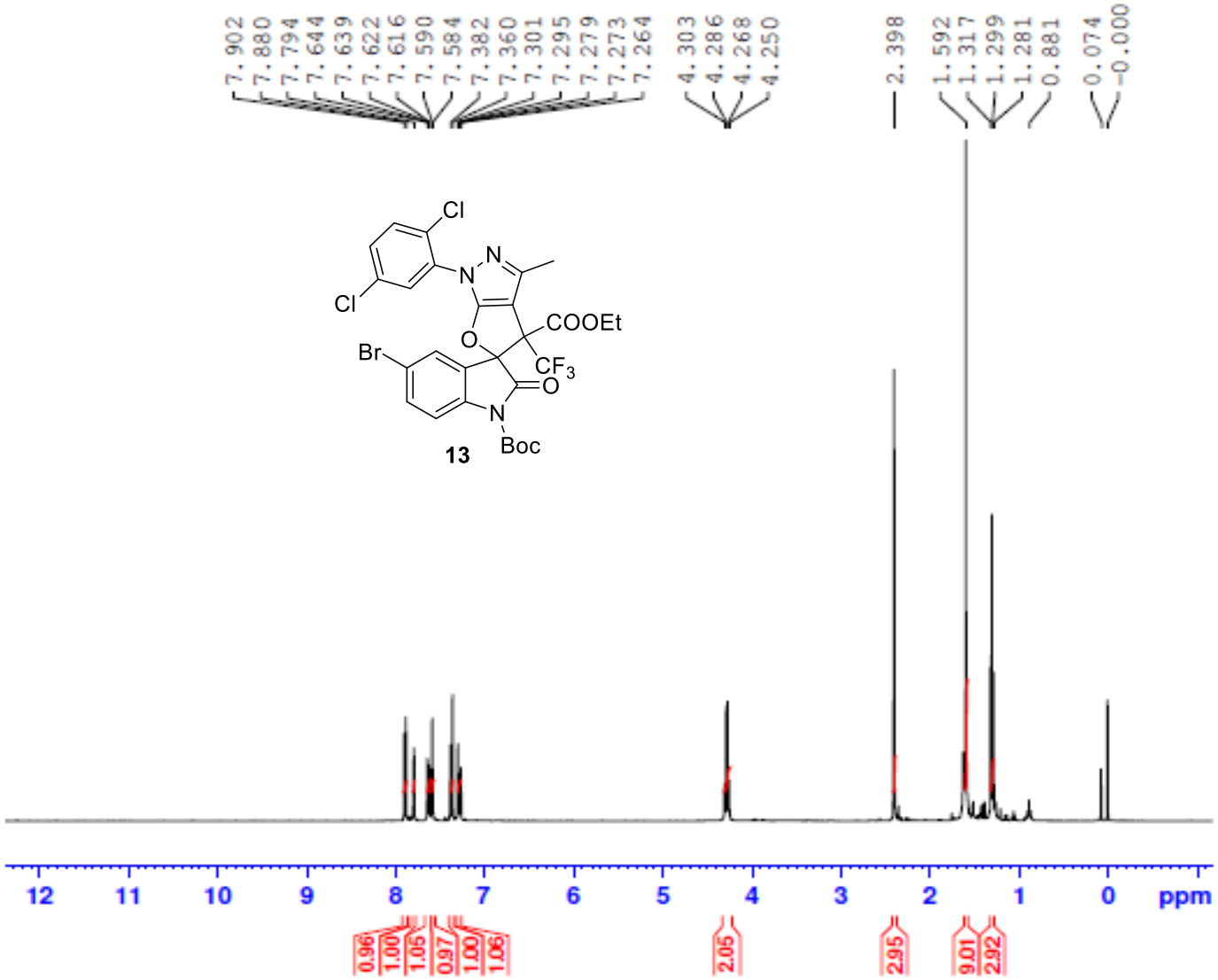
ANL-MCL5-NMR-003




```
Current Data Parameters
NAME         512002D2027
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200229
Time              12.53 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG               zg
TD                131072
SOLVENT             CDCl3
NS                    16
DS                     4
SWH           90909.094 Hz
FIDRES         1.387163 Hz
AQ            0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1         37.49499893 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                  EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

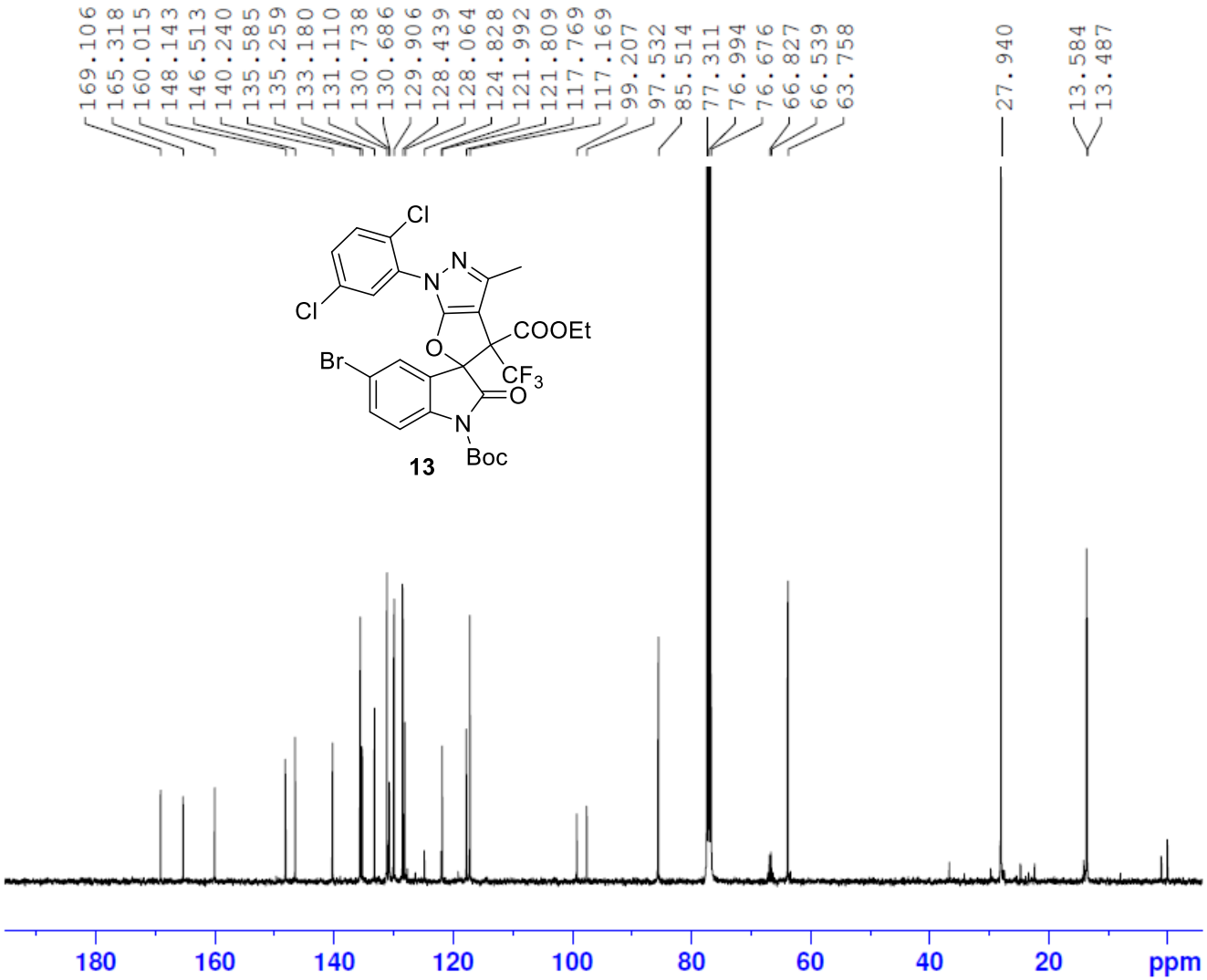
ANL-MCL5-NMR-003


SAMPLE ID: TP-11580-022
ACQ.METHOD: FA: 4.2 MIN
28022020-093
512002D2028A
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:28-Feb-202019:55:28
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 6.014e+1
Time Height Area Area%
2.66 55805 1421.04 0.07
2.78 322188 8186.56 0.40
2.82 116027 3729.96 0.18
2.89 178930 6438.88 0.31
2.92 290666 9310.02 0.45
2.97 780310 17863.11 0.87
2.99 787566 17366.14 0.85
3.04 3634969 122163.10 5.96
3.17 59785412 1792432.13 87.46
3.27 1204884 56337.37 2.75
3.40 178965 14112.11 0.69
Br
N
N
COOEt
O
Br
CF3
O
N
Boc
12 (6:87, dr)
AU
6.0e+1
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
3.17
3.04
2.66 2.78 2.99
3.27
3.40
-0.00 0.25 0.50 0.75 1.00 1.25 1.50 1.75 2.00 2.25 2.50 2.75 3.00 3.25 3.50 3.75 4.00



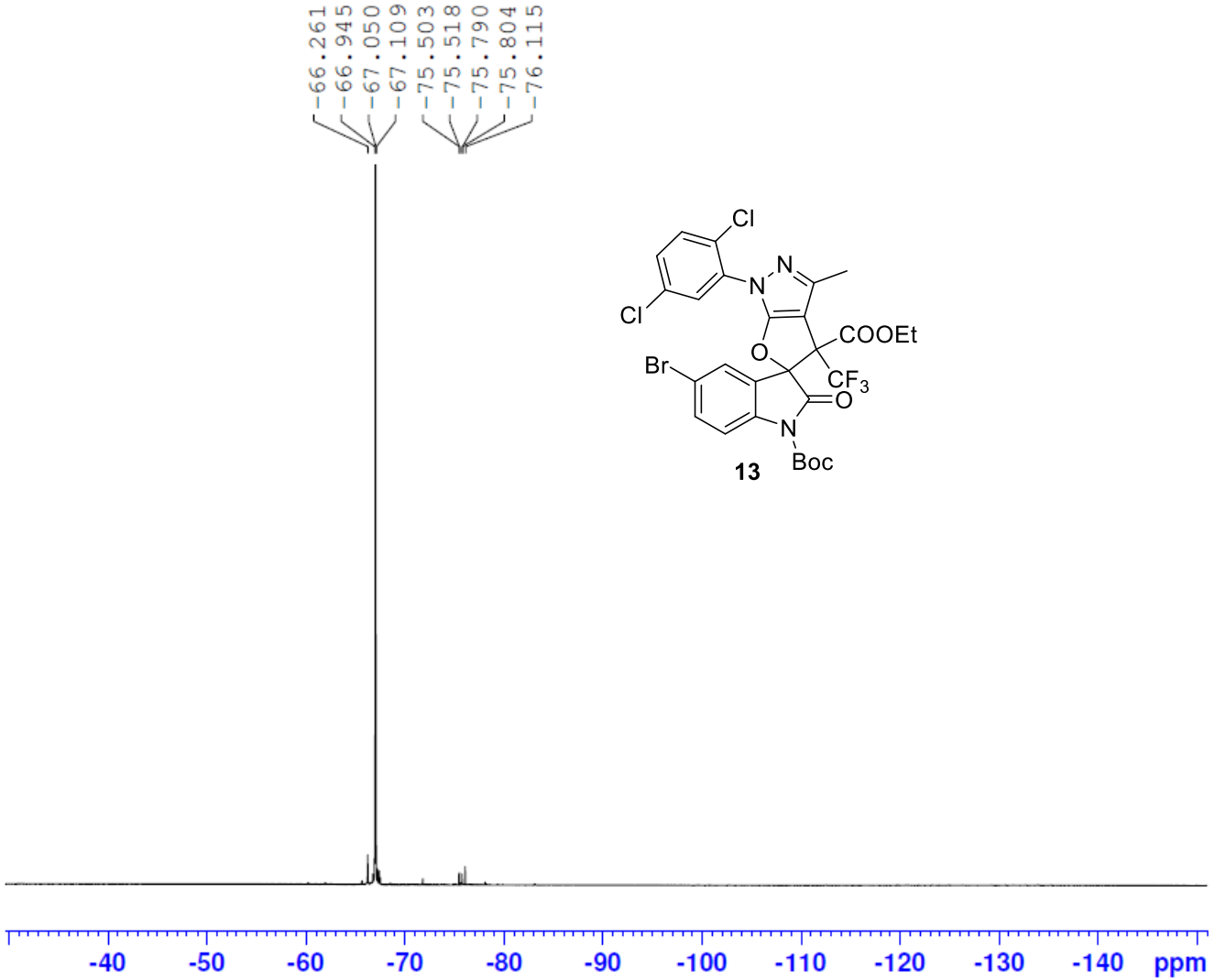

```
Current Data Parameters
NAME          512002D2022
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Data_              20200229
Time                 13.03 h
INSTRUM              Avance
PROBHD    Z163739_0135 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8196.722 Hz
FIDRES            0.250144 Hz
AQ                3.9976959 sec
RG                   66.3623
DW                   61.000 usec
DE                   13.89 usec
TE                   298.2 K
D1                1.00000000 sec
TD0                       1
SFO1           400.3024719 MHz
NUC1                     1H
P0                    2.67 usec
P1                    8.00 usec
PLW1           22.47400093 W

F2 - Processing parameters
SI                    65536
SF             400.3000085 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

ANL-MCL5-NMR-003



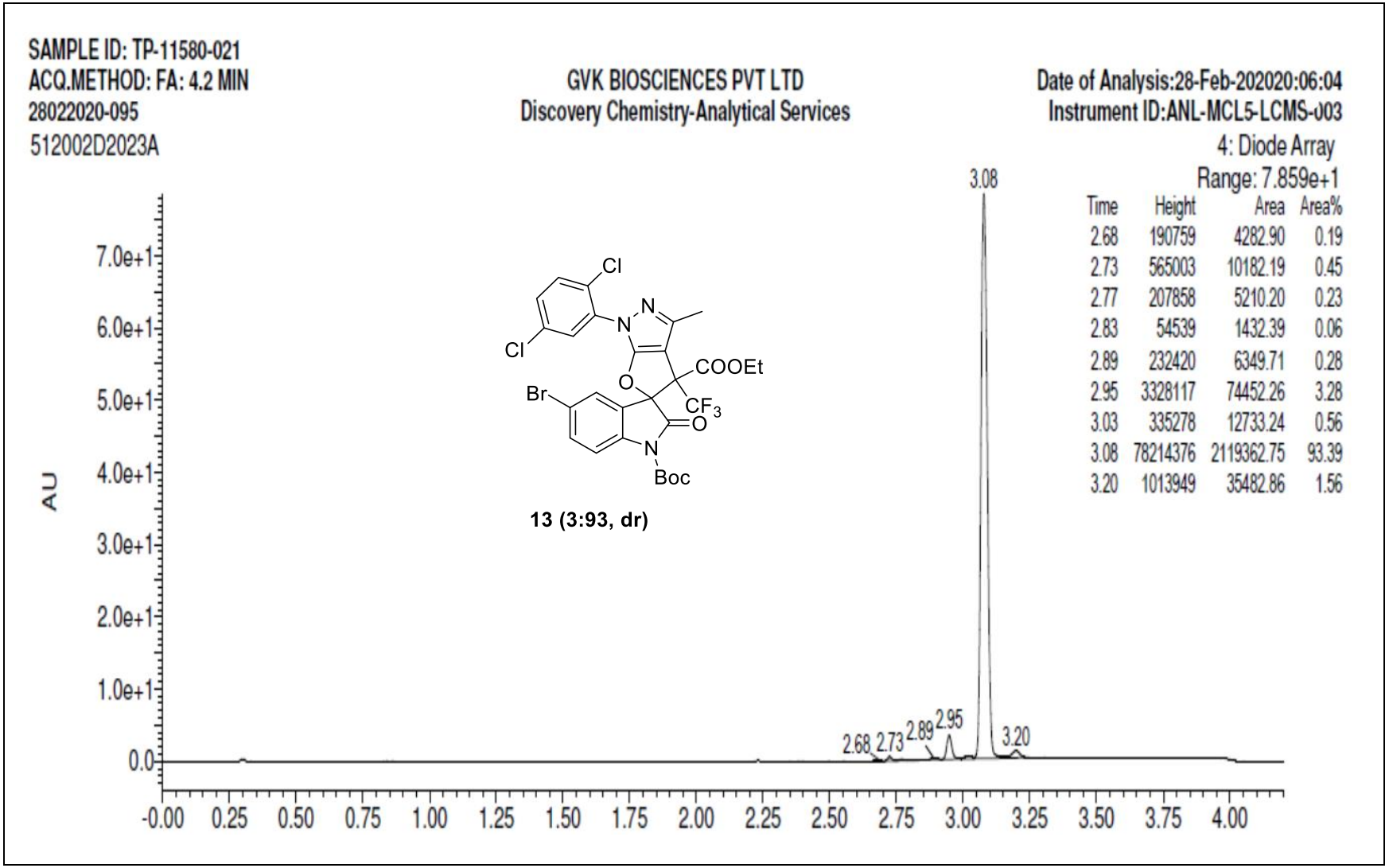

```
Current Data Parameters
NAME          512002D2022
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20200301
Time                5.15 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3000
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ             1.3762560 sec
RG                  101
DW                21.000 usec
DE                 6.50 usec
TE                298.2 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                   1
SFO1         100.6655806 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1          92.65799713 W
SFO2         400.3016012 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2          22.47400093 W
PLW12          0.17757000 W
PLW13          0.08931800 W

F2 - Processing parameters
SI                 32768
SF           100.6555192 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

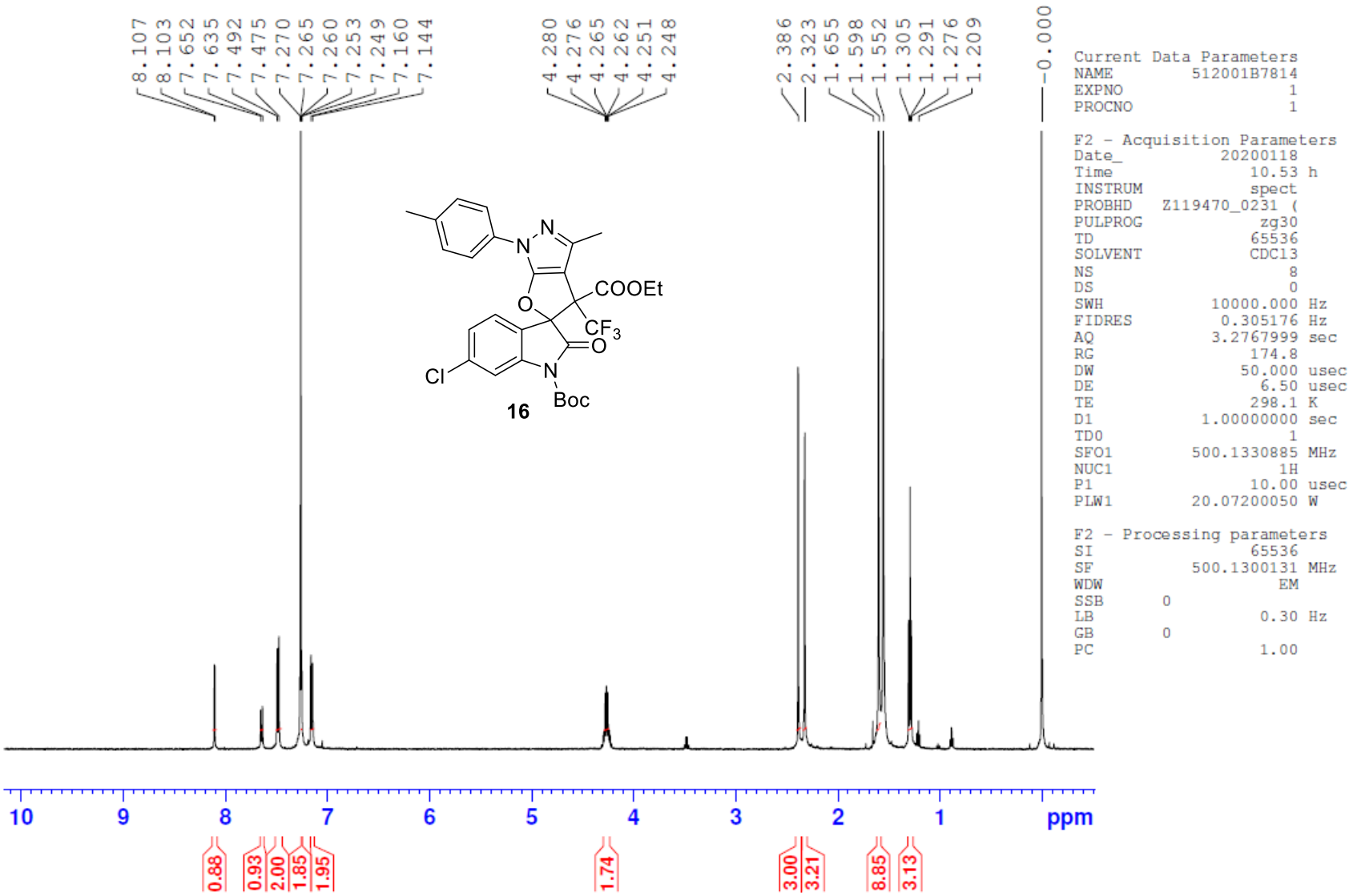
ANL-MCL5-NMR-003




```
Current Data Parameters
NAME         512002D2022
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200229
Time               13.05 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG               zg
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ              0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1         37.49499893 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

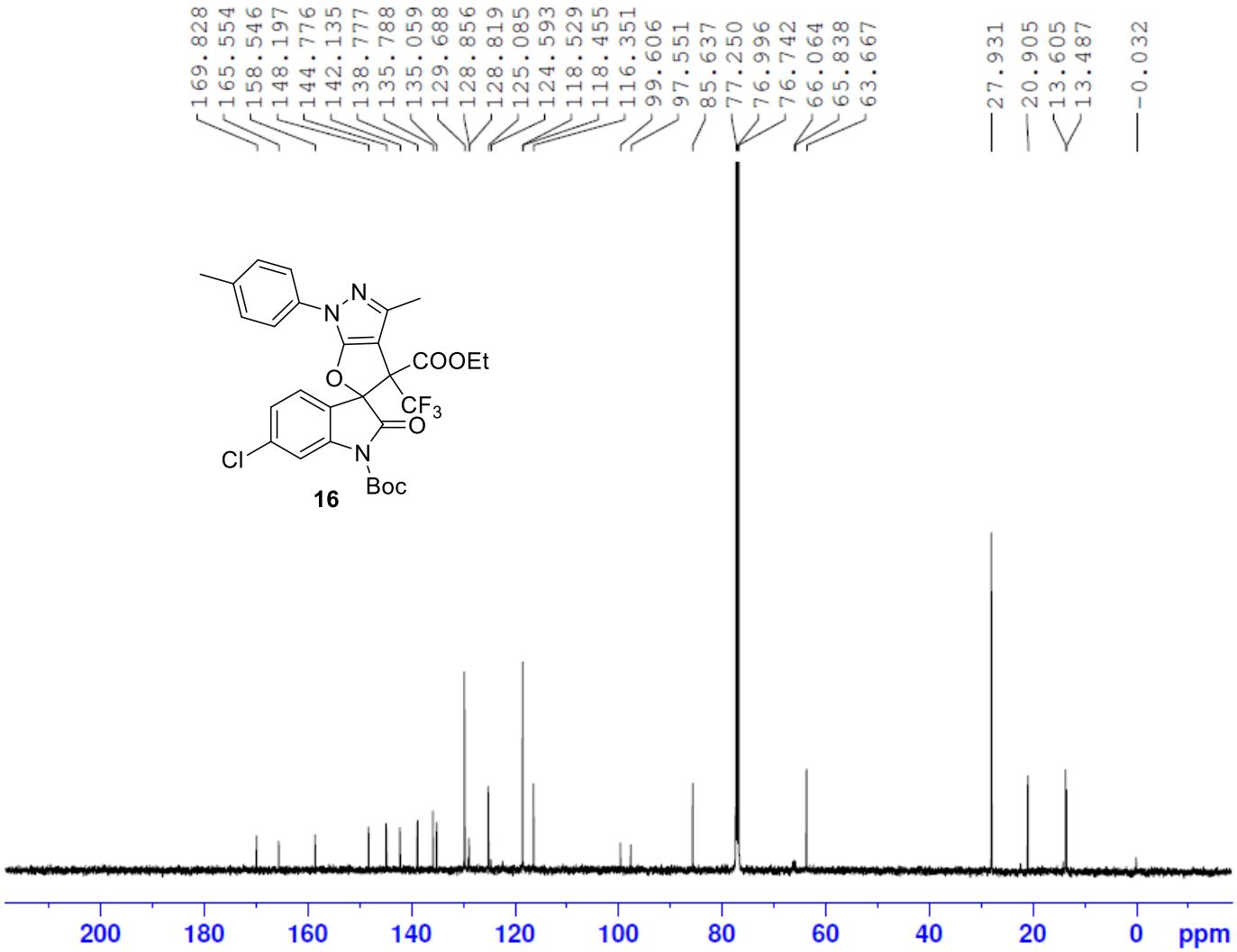
ANL-MCL5-NMR-003


SAMPLE ID: TP-11580-021
ACQ.METHOD: FA: 4.2 MIN
28022020-095
512002D2023A
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:28-Feb-202020:06:04
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 7.859e+1
Time Height Area Area%
2.68 190759 4282.90 0.19
2.73 565003 10182.19 0.45
2.77 207858 5210.20 0.23
2.83 54539 1432.39 0.06
2.89 232420 6349.71 0.28
2.95 3328117 74452.26 3.28
3.03 335278 12733.24 0.56
3.08 78214376 2119362.75 93.39
3.20 1013949 35482.86 1.56
Cl
Cl
N
N
O
COOEt
Br
CF3
O
N
Boc
13 (3:93, dr)
AU
7.0e+1
6.0e+1
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
3.08
2.68
2.73
2.89
2.95
3.20
-0.00 0.25 0.50 0.75 1.00 1.25 1.50 1.75 2.00 2.25 2.50 2.75 3.00 3.25 3.50 3.75 4.00



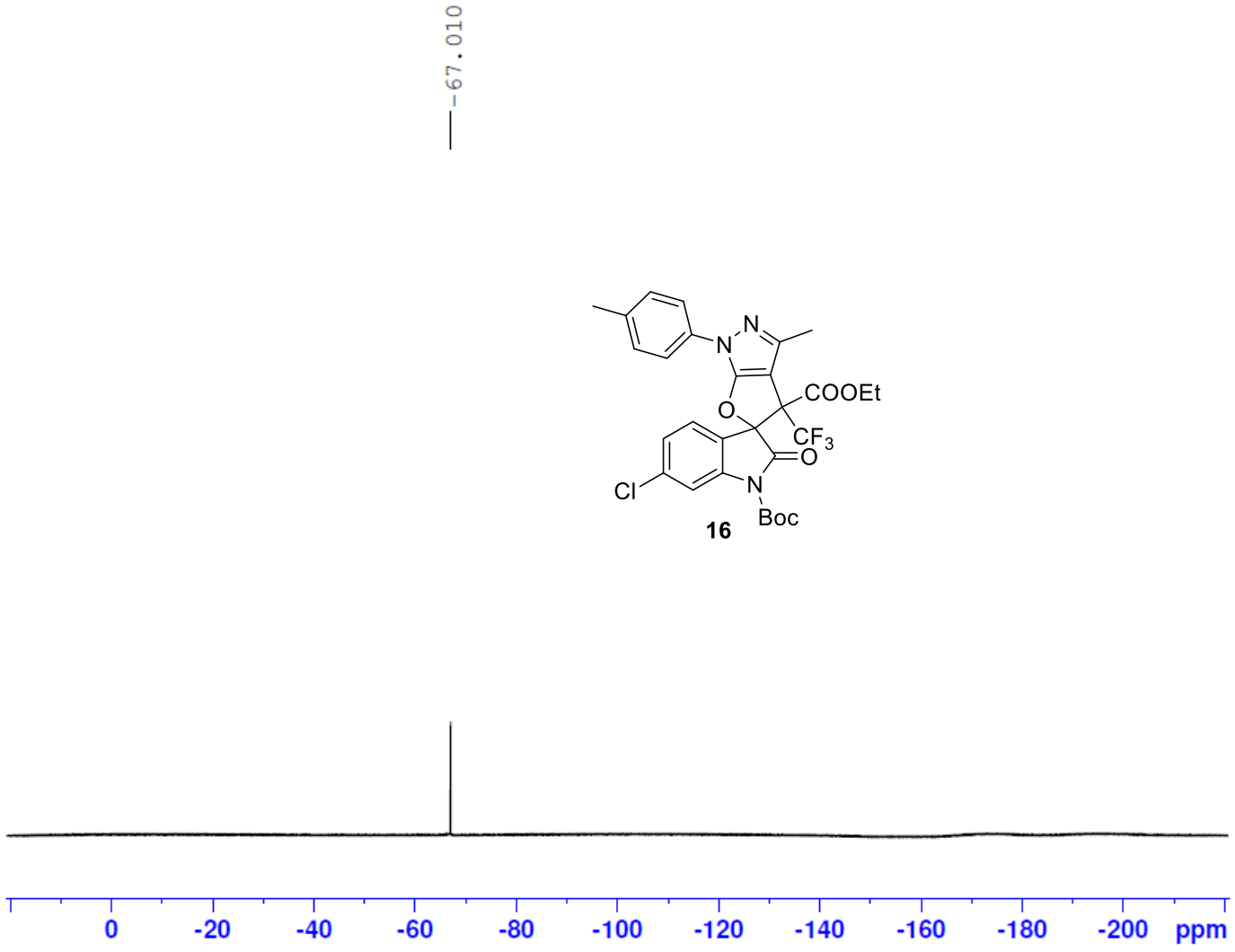

```
Current Data Parameters
NAME         512001B7814
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200118
Time               10.53 h
INSTRUM             spect
PROBHD    Z119470_0231 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            10000.000 Hz
FIDRES             0.305176 Hz
AQ                 3.2767999 sec
RG                   174.8
DW                  50.000 usec
DE                   6.50 usec
TE                  298.1 K
D1              1.00000000 sec
TD0                    1
SFO1           500.1330885 MHz
NUC1                  1H
P1                  10.00 usec
PLW1           20.07200050 W

F2 - Processing parameters
SI                 65536
SF             500.1300131 MHz
WDW                   EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

TP-11580-014

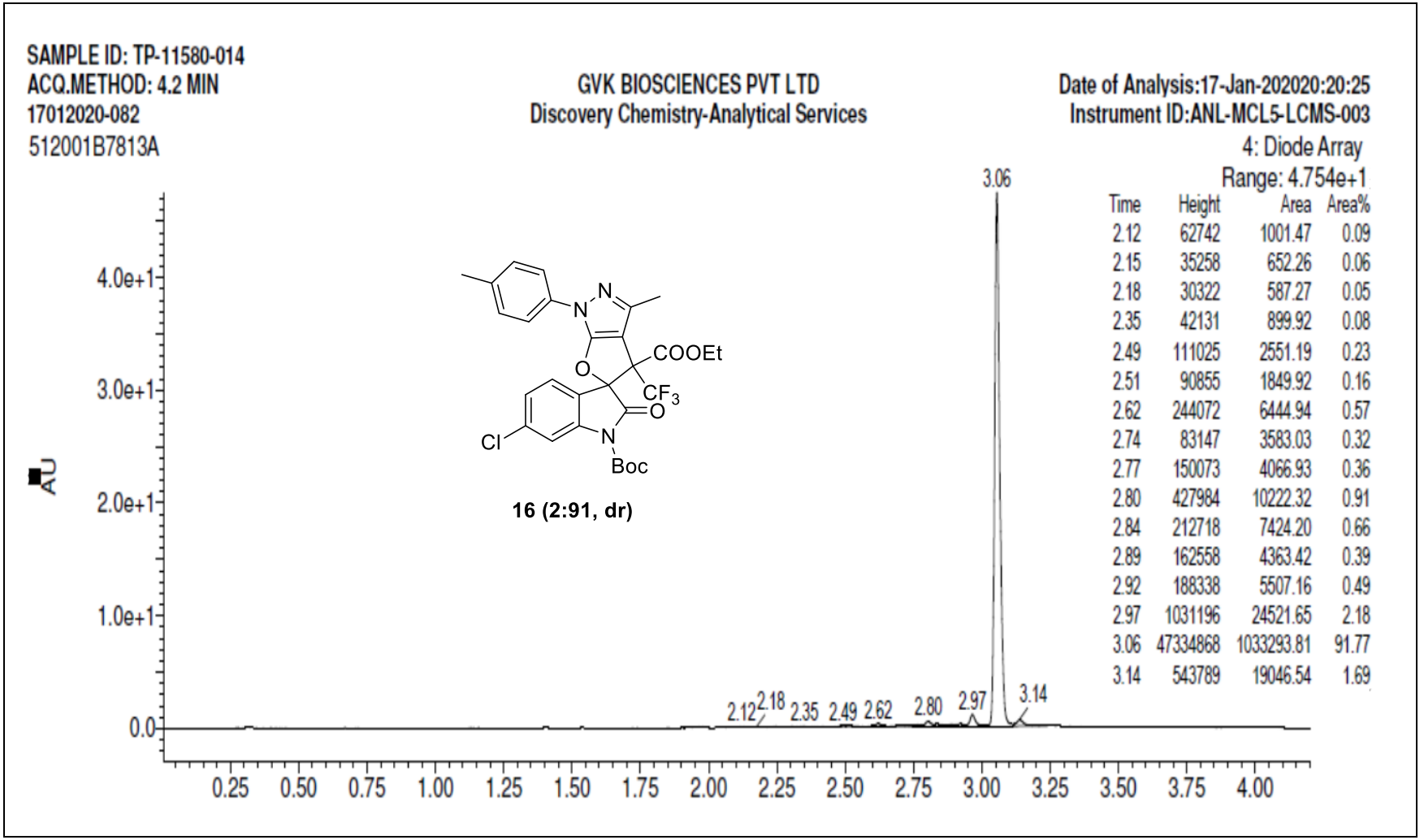


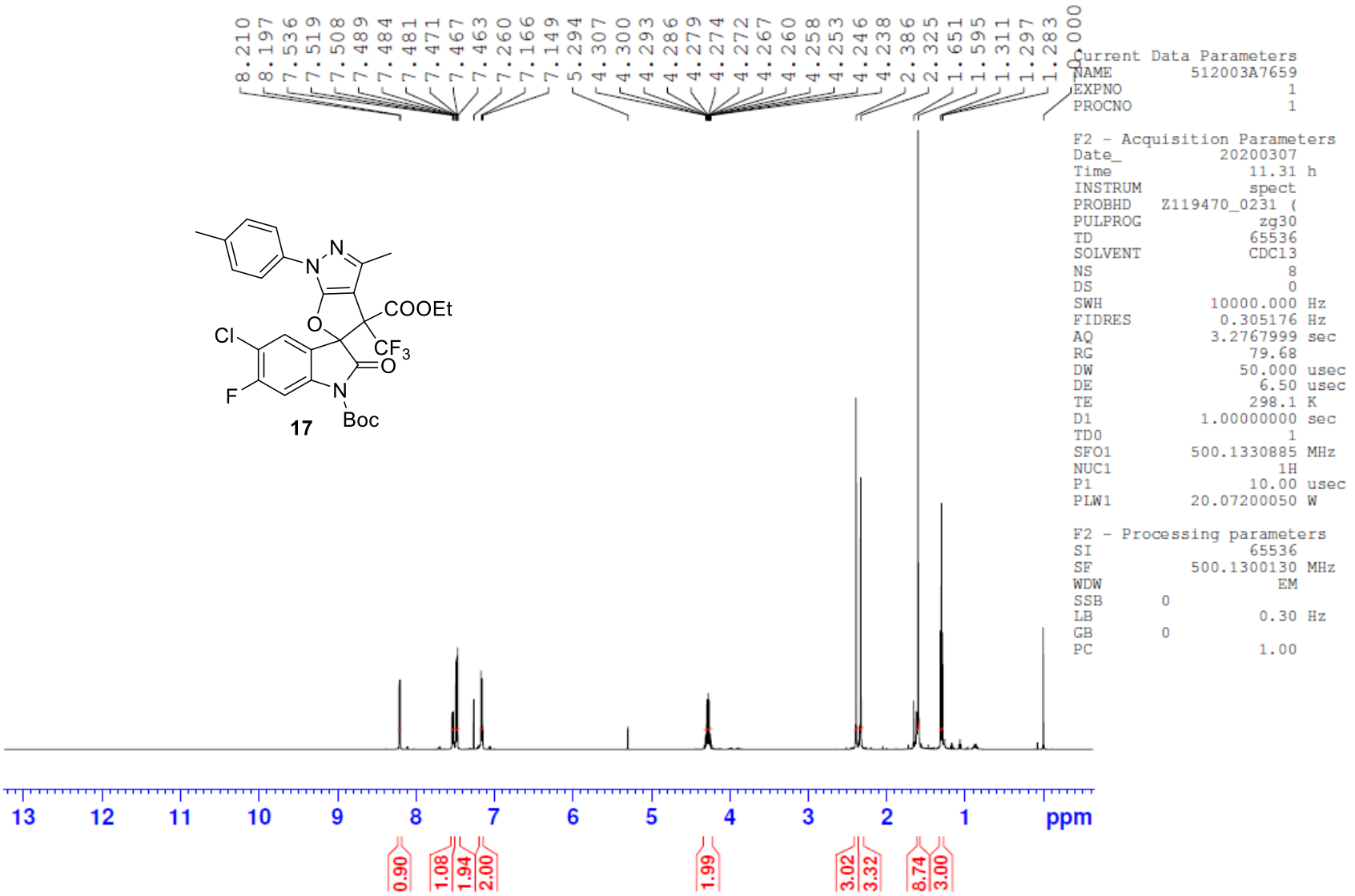

```
Current Data Parameters
NAME          512001c7993
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200122
Time                5.58 h
INSTRUM            spect
PROBHD    Z119470_0231 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  609
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ             1.1010048 sec
RG                197.72
DW                16.800 usec
DE                 6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1         125.7703643 MHz
NUC1                13C
P1                10.00 usec
PLW1         86.78700256 W
SFO2         500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         20.07200050 W
PLW12         0.31363001 W
PLW13         0.15775000 W

F2 - Processing parameters
SI                 32768
SF           125.7577932 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

TP-11580-014

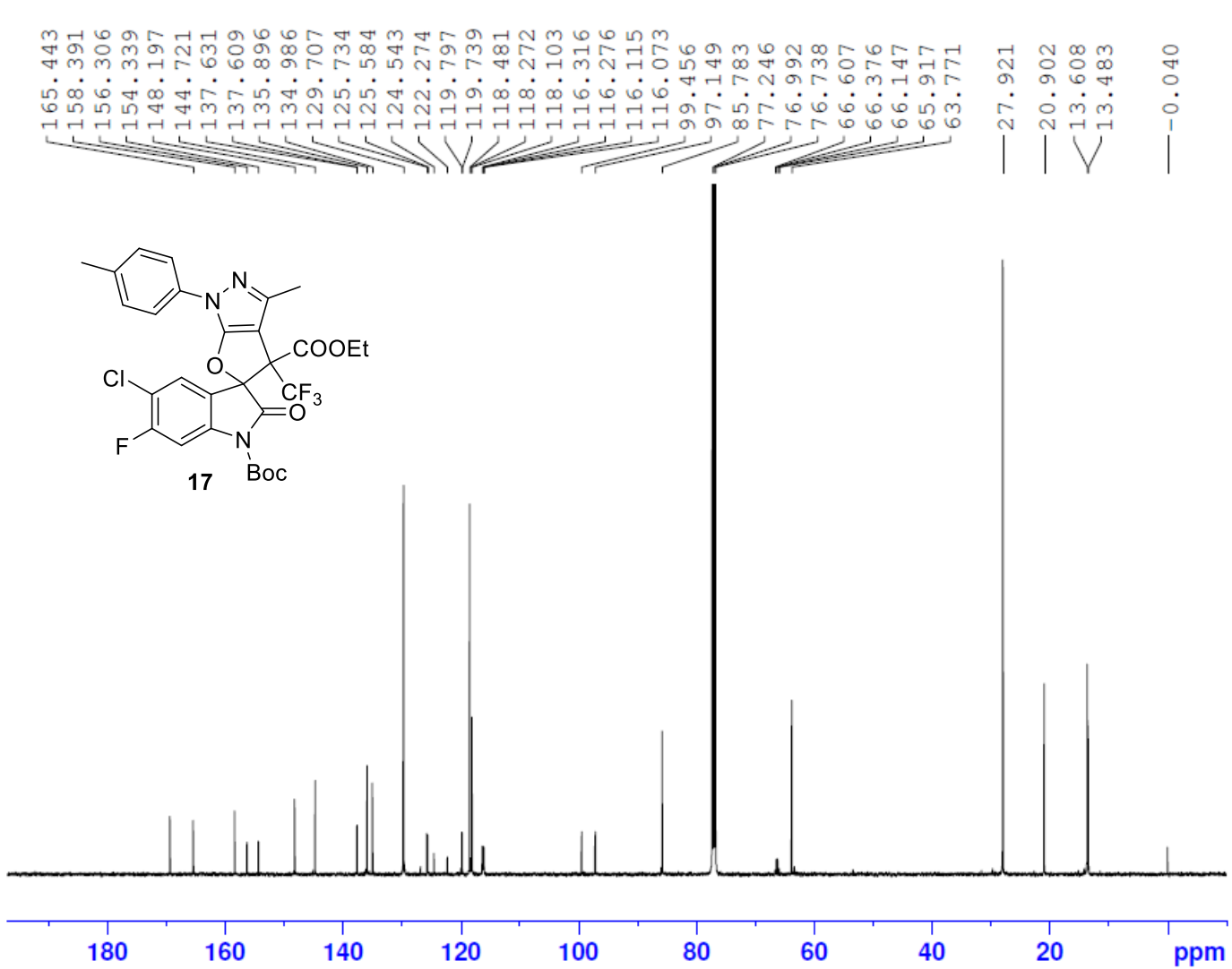



```
Current Data Parameters
NAME         512006A9194
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200608
Time              18.49 h
INSTRUM             spect
PROBHD    Z119470_0294 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ              0.5767168 sec
RG                 197.72
DW                 4.400 usec
DE                  6.50 usec
TE                 301.2 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1
SFO1          470.5453180 MHz
NUC1                  19F
P1                 15.00 usec
PLW1          36.26100159 W
SFO2          500.1320005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          21.30500031 W
PLW12          0.33287999 W

F2 - Processing parameters
SI                 65536
SF            470.5923772 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```


SAMPLE ID: TP-11580-014
ACQ.METHOD: 4.2 MIN
17012020-082
512001B7813A
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:17-Jan-202020:20:25
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 4.754e+1
Time Height Area Area%
2.12 62742 1001.47 0.09
2.15 35258 652.26 0.06
2.18 30322 587.27 0.05
2.35 42131 899.92 0.08
2.49 111025 2551.19 0.23
2.51 90855 1849.92 0.16
2.62 244072 6444.94 0.57
2.74 83147 3583.03 0.32
2.77 150073 4066.93 0.36
2.80 427984 10222.32 0.91
2.84 212718 7424.20 0.66
2.89 162558 4363.42 0.39
2.92 188338 5507.16 0.49
2.97 1031196 24521.65 2.18
3.06 47334868 1033293.81 91.77
3.14 543789 19046.54 1.69
AU
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
0.25 0.50 0.75 1.00 1.25 1.50 1.75 2.00 2.25 2.50 2.75 3.00 3.25 3.50 3.75 4.00
2.12 2.18 2.35 2.49 2.62 2.80 2.97 3.06 3.14
COOEt
CF3
O
Cl
N
Boc
16 (2:91, dr)

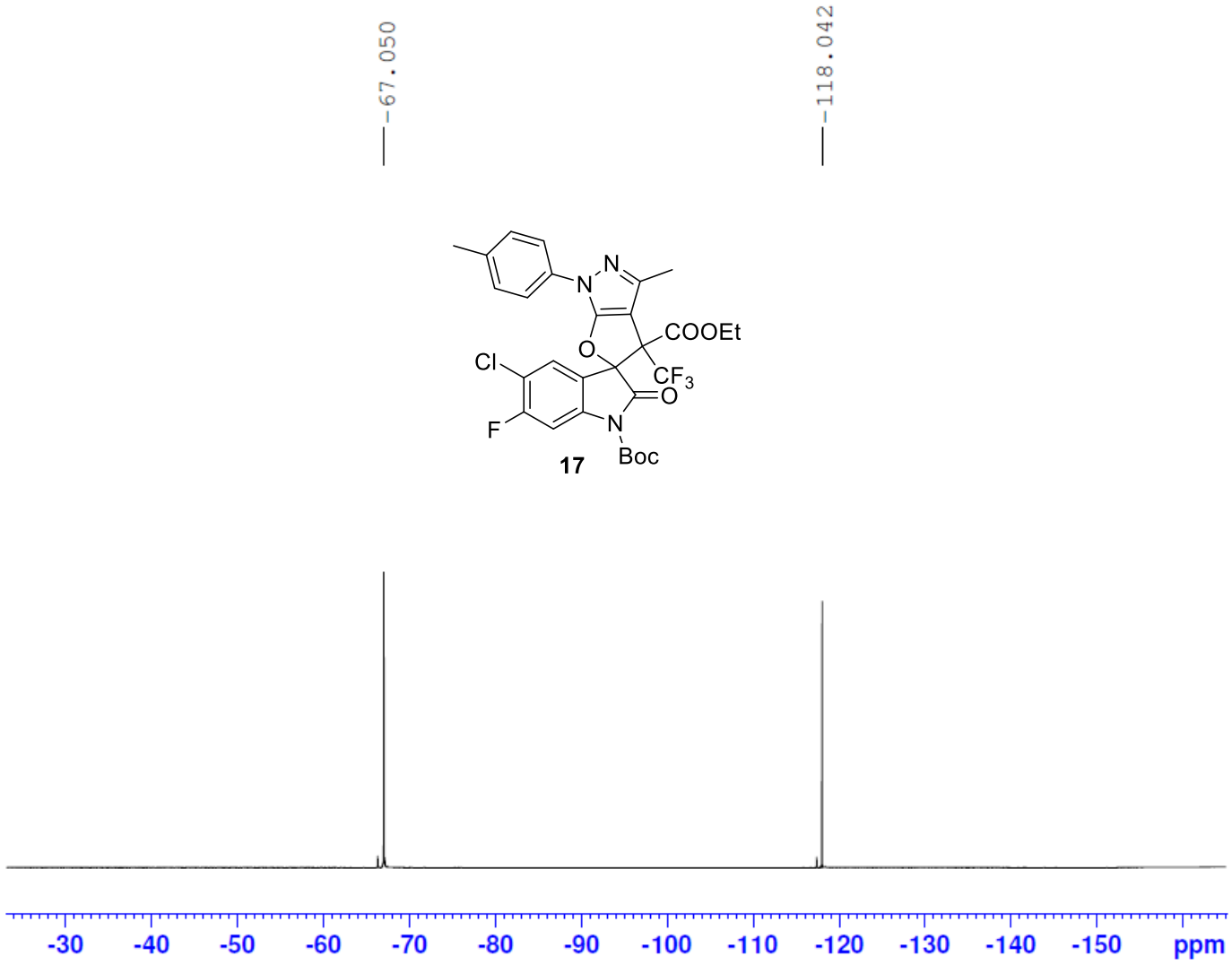

8.210
8.197
7.536
7.519
7.508
7.489
7.484
7.481
7.471
7.467
7.463
7.260
7.166
7.149
5.294
4.307
4.300
4.293
4.286
4.279
4.274
4.272
4.267
4.260
4.258
4.253
4.246
4.238
2.386
2.325
1.651
1.595
1.311
1.297
1.283
0.000
COOEt
CF3
Cl
F
O
N
Boc
17
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.90
1.08
1.94
2.00
1.99
3.02
3.32
8.74
3.00
Current Data Parameters
NAME 512003A7659
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200307
Time 11.31 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 79.68
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 20.07200050 W
F2 - Processing parameters
SI 65536
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TP-11580-013

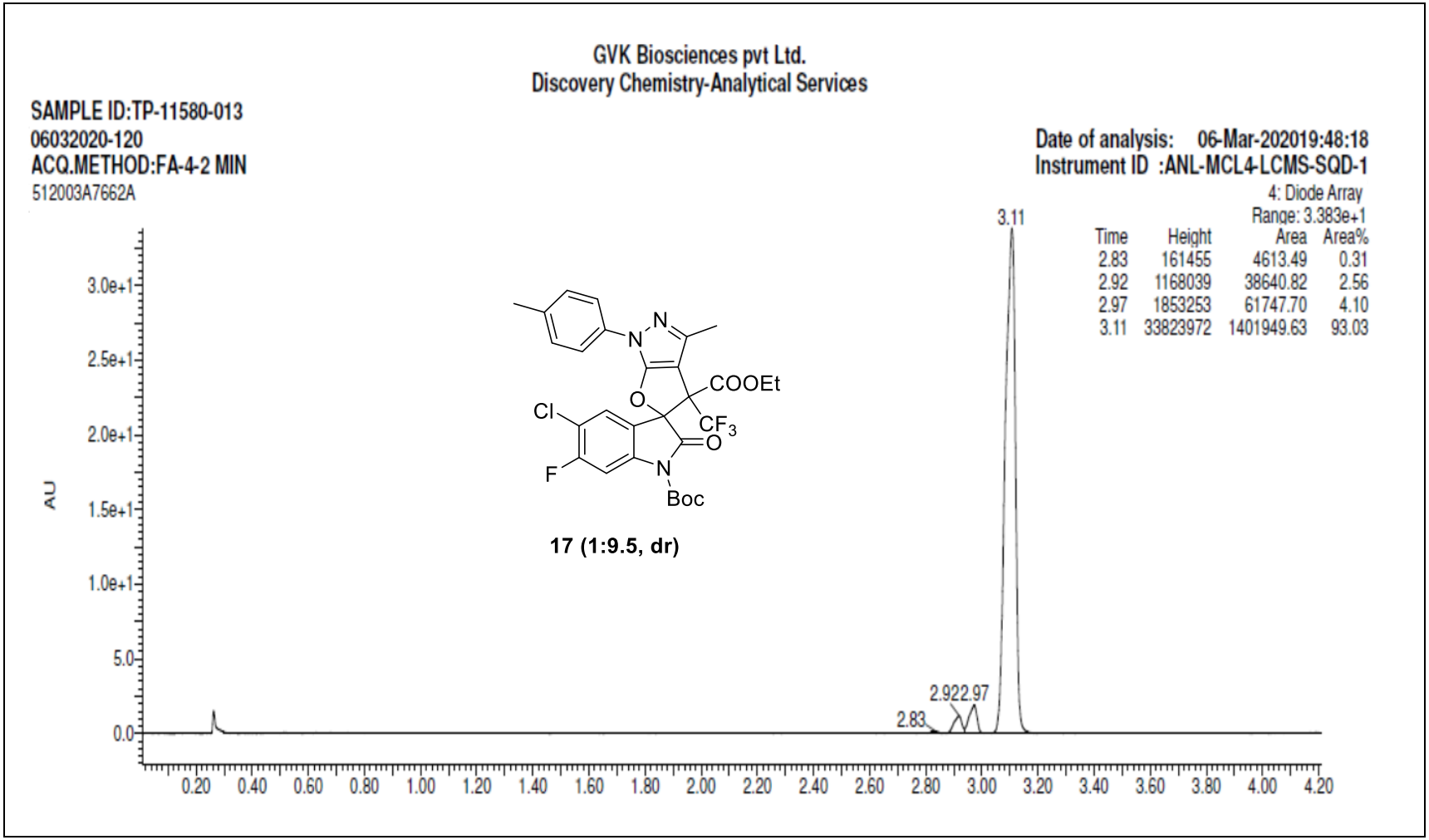


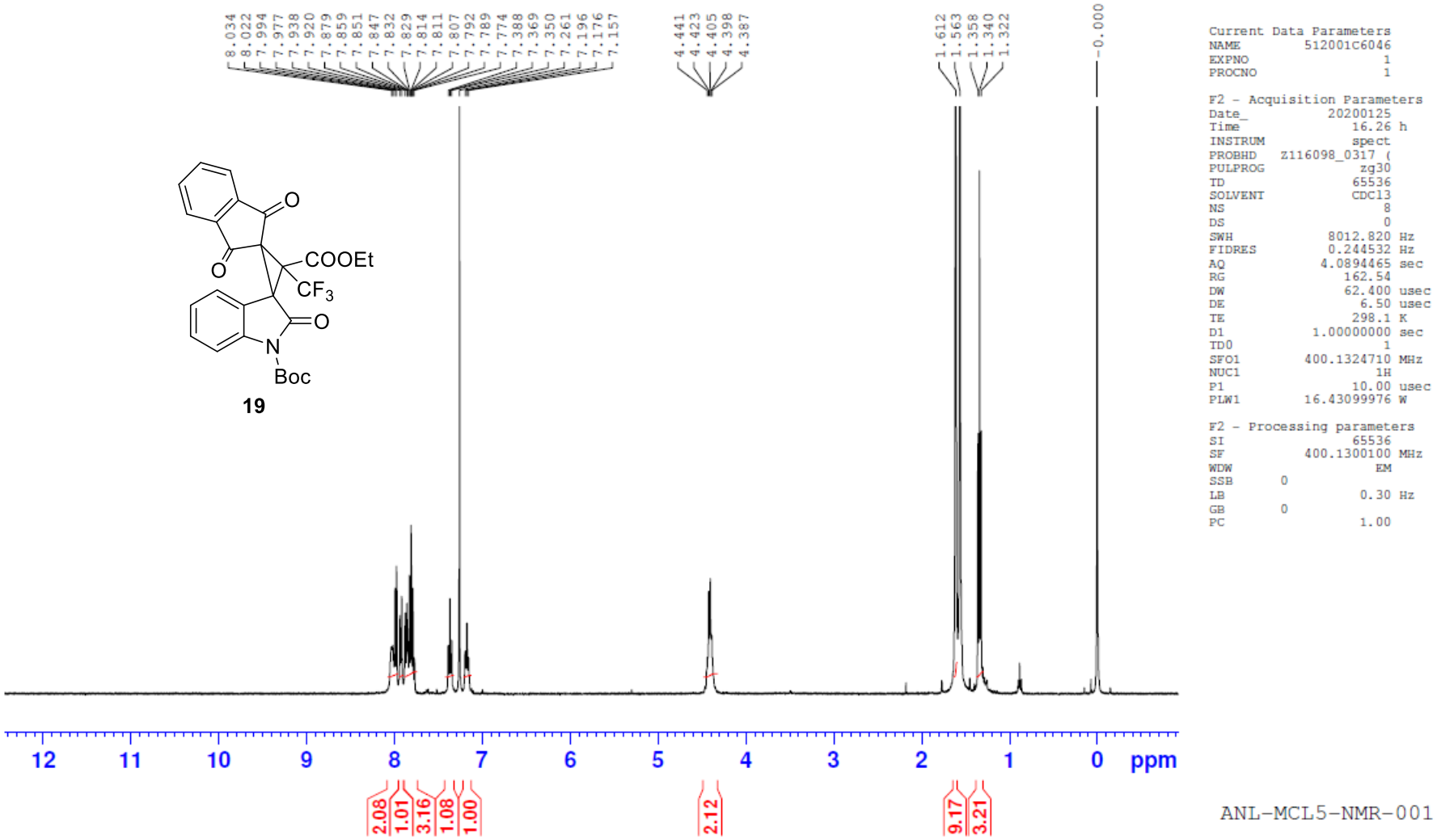

```
Current Data Parameters
NAME          512003A7659
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20200308
Time                11.05 h
INSTRUM             spect
PROBHD    Z119470_0231 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3072
DS                     4
SWH            29761.904 Hz
FIDRES          0.908261 Hz
AQ             1.1010048 sec
RG                197.72
DW                16.800 usec
DE                 6.50 usec
TE                298.1 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                13C
P1                10.00 usec
PLW1          86.78700256 W
SFO2          500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2          20.07200050 W
PLW12          0.31363001 W
PLW13          0.15775000 W

F2 - Processing parameters
SI                 32768
SF            125.7577934 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

TP-11580-013



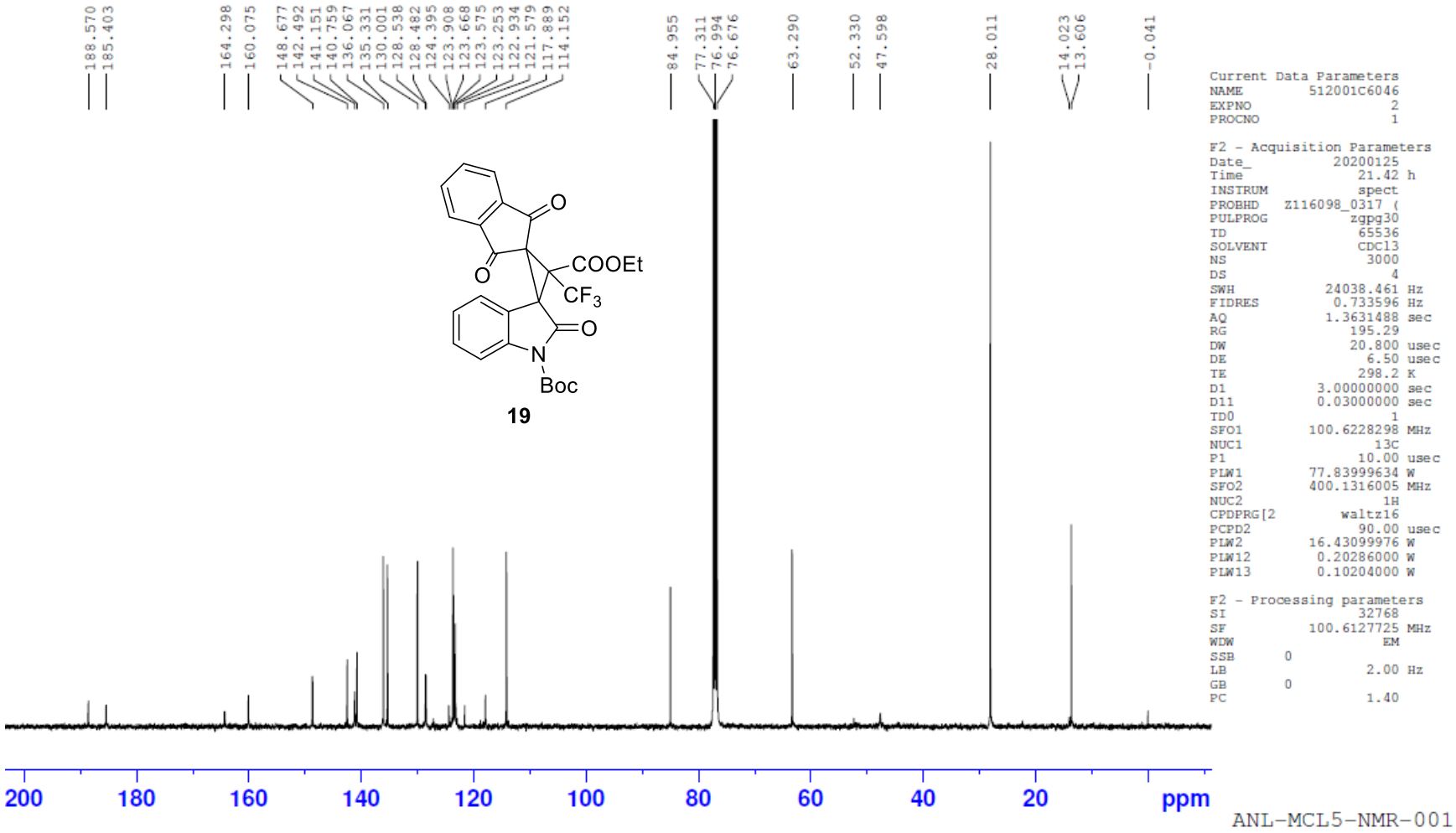

```
Current Data Parameters
NAME         512003A7659
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200308
Time                7.30 h
INSTRUM             spect
PROBHD   Z119470_0231 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDC13
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ              0.5767168 sec
RG                 197.72
DW                  4.400 usec
DE                  6.50 usec
TE                 298.2 K
D1             1.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
TD0                    1
SFO1          470.5453180 MHz
NUC1                  19F
P1                  15.00 usec
PLW1          34.86600113 W
SFO2          500.1320005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2          20.07200050 W
PLW12          0.31363001 W

F2 - Processing parameters
SI                  65536
SF            470.5923772 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.00
```

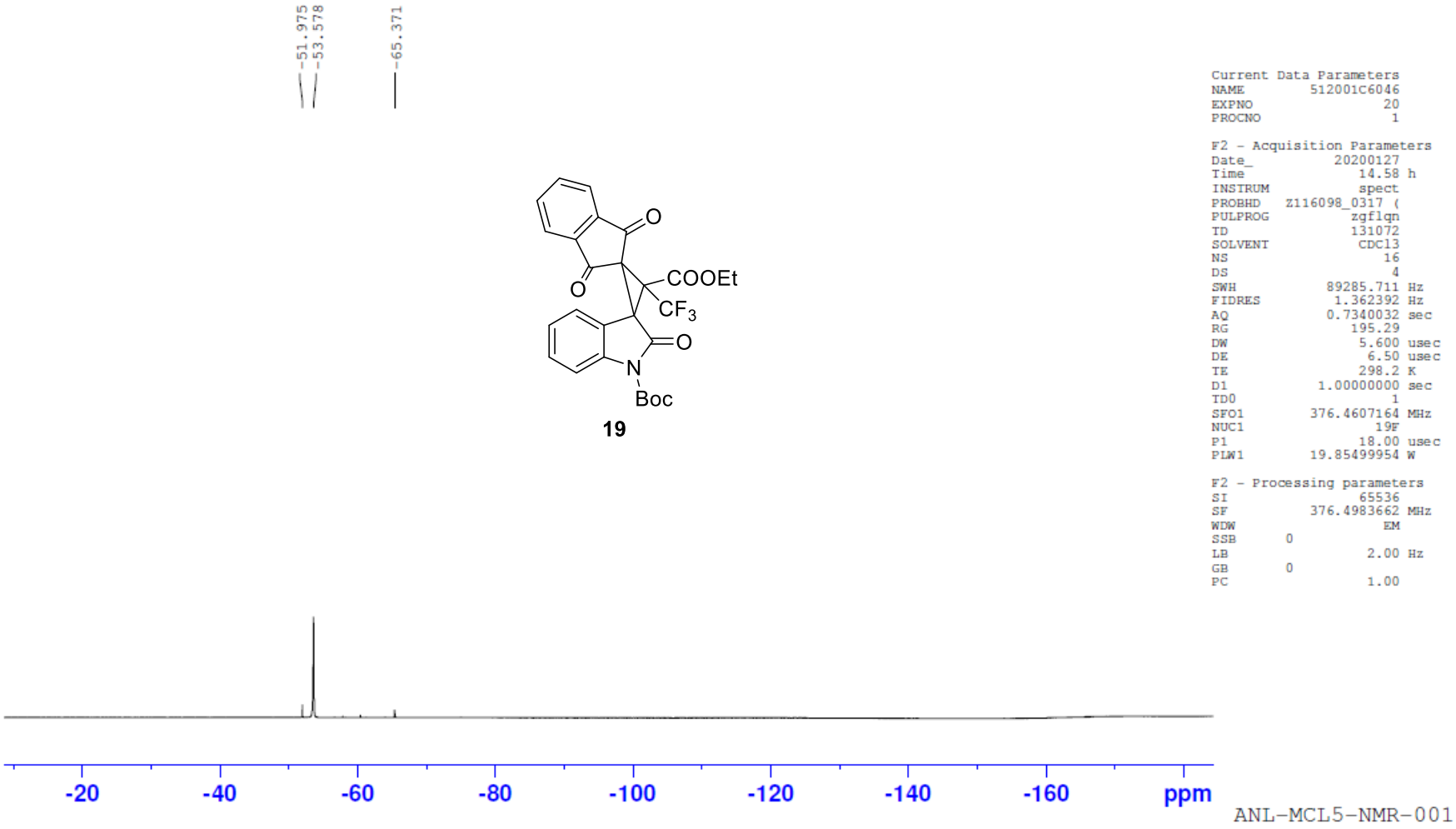

GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services
SAMPLE ID:TP-11580-013
06032020-120
ACQ.METHOD:FA-4-2 MIN
512003A7662A
Date of analysis: 06-Mar-202019:48:18
Instrument ID :ANL-MCL4-LCMS-SQD-1
4: Diode Array
Range: 3.383e+1
Time Height Area Area%
2.83 161455 4613.49 0.31
2.92 1168039 38640.82 2.56
2.97 1853253 61747.70 4.10
3.11 33823972 1401949.63 93.03
AU
3.0e+1
2.5e+1
2.0e+1
1.5e+1
1.0e+1
5.0
0.0
0.20 0.40 0.60 0.80 1.00 1.20 1.40 1.60 1.80 2.00 2.20 2.40 2.60 2.80 3.00 3.20 3.40 3.60 3.80 4.00 4.20
3.11
2.92 2.97
2.83
COOEt
CF3
Cl
F
O
N
Boc
17 (1:9.5, dr)

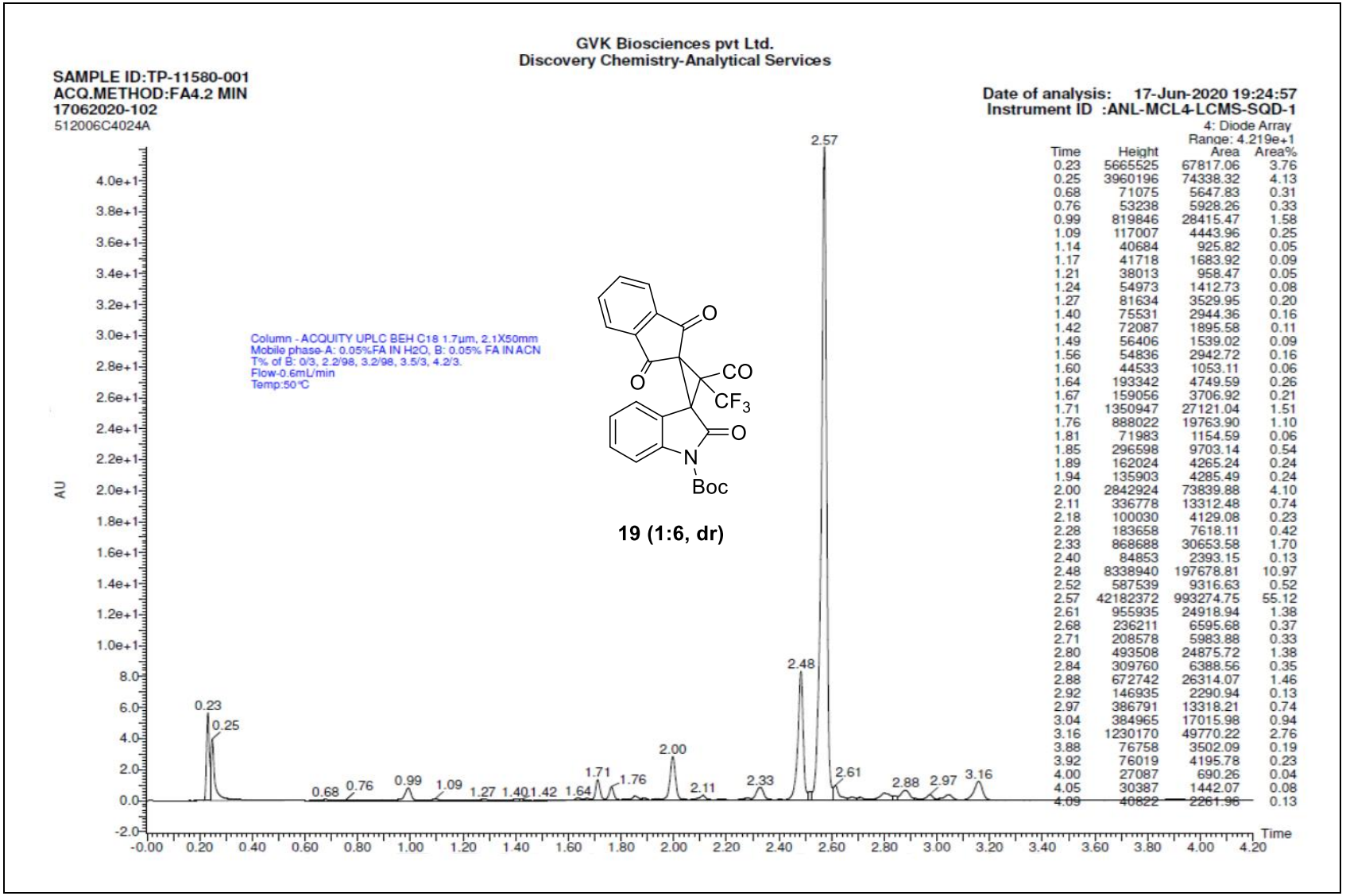


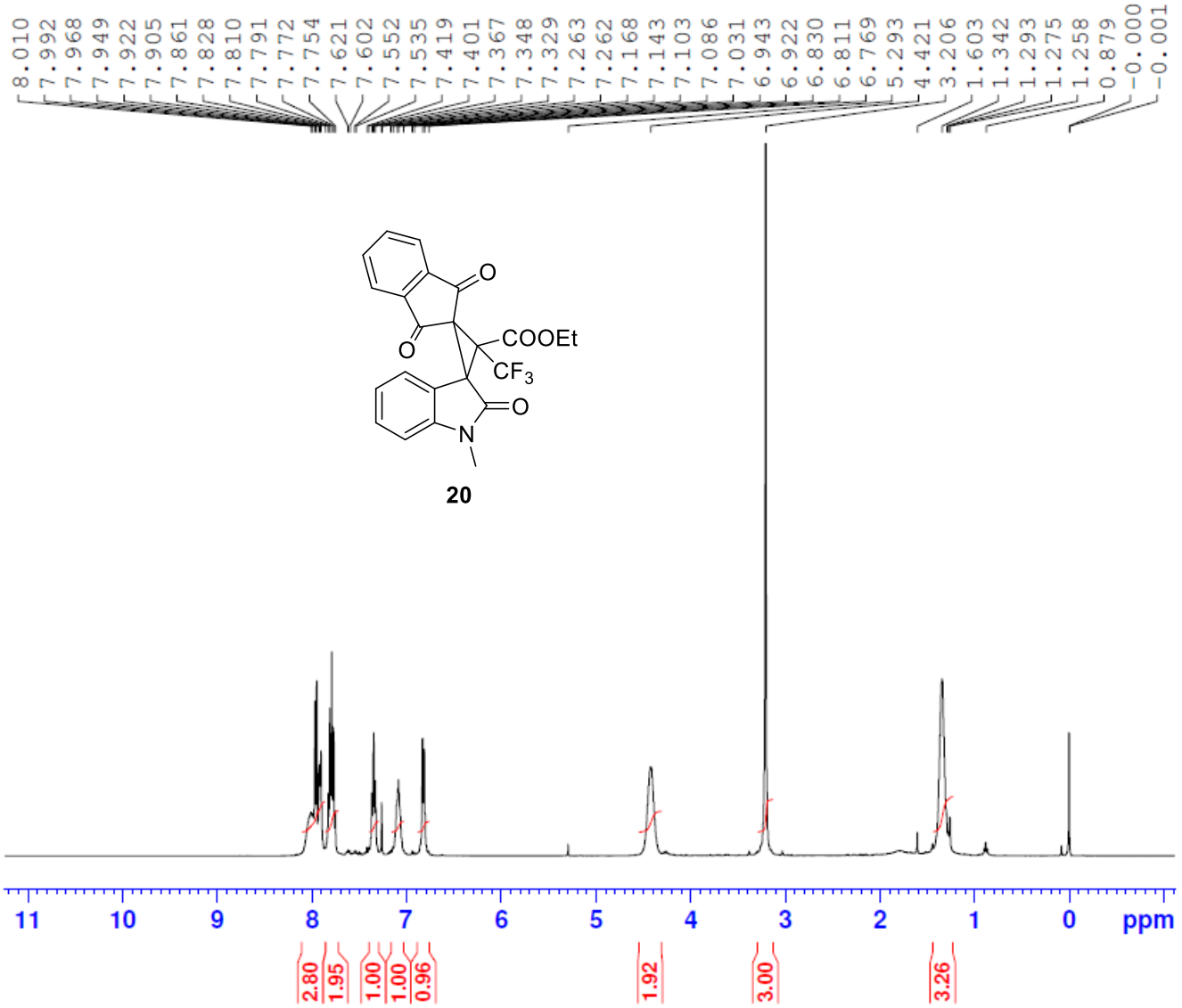

```
Current Data Parameters
NAME          512001c6046
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200125
Time                16.26 h
INSTRUM              spect
PROBHD    Z116098_0317 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ               4.0894465 sec
RG                  162.54
DW                  62.400 usec
DE                   6.50 usec
TE                  298.1 K
D1            1.00000000 sec
TD0                     1
SFO1          400.1324710 MHz
NUC1                   1H
P1                  10.00 usec
PLW1          16.43099976 W

F2 - Processing parameters
SI                  65536
SF            400.1300100 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```

ANL-MCL5-NMR-001


188.570
185.403
164.298
160.075
148.677
142.492
141.151
140.759
136.067
135.331
130.001
128.538
128.482
124.395
123.908
123.668
123.575
123.253
122.934
121.579
117.889
114.152
84.955
77.311
76.994
76.676
63.290
52.330
47.598
28.011
14.023
13.606
-0.041
O
O
COOEt
CF3
O
N
Boc
19
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME 512001c6046
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200125
Time 21.42 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 195.29
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 77.83999634 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.43099976 W
PLW12 0.20286000 W
PLW13 0.10204000 W
F2 - Processing parameters
SI 32768
SF 100.6127725 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
ANL-MCL5-NMR-001


-51.975
-53.578
-65.371
O
O
COOEt
CF3
O
N
Boc
19
Current Data Parameters
NAME 512001C6046
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200127
Time 14.58 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zgflqn
TD 131072
SOLVENT CDCl3
NS 16
DS 4
SWH 89285.711 Hz
FIDRES 1.362392 Hz
AQ 0.7340032 sec
RG 195.29
DW 5.600 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 376.4607164 MHz
NUC1 19F
P1 18.00 usec
PLW1 19.85499954 W
F2 - Processing parameters
SI 65536
SF 376.4983662 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00
-20
-40
-60
-80
-100
-120
-140
-160
ppm
ANL-MCL5-NMR-001

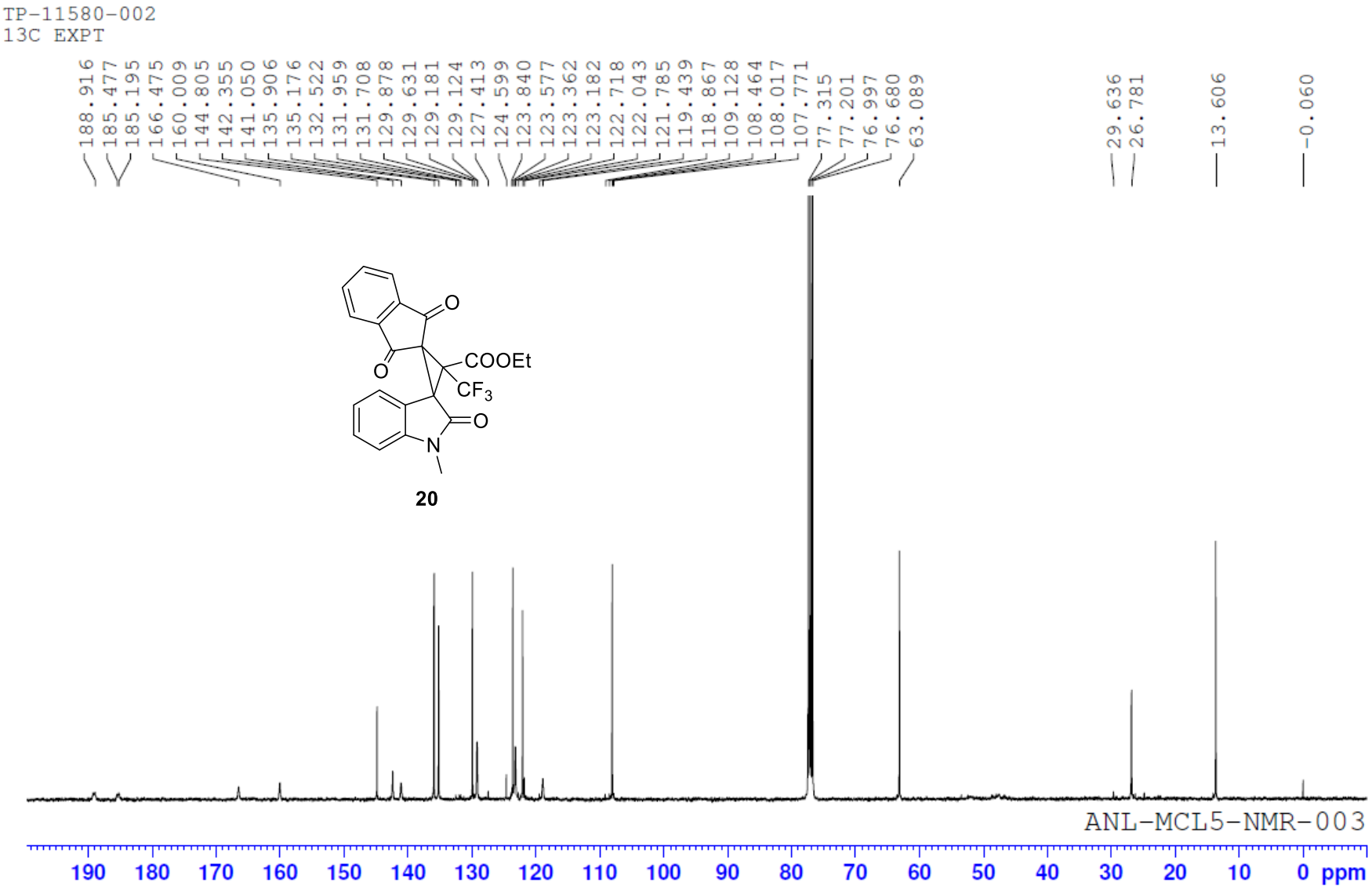

GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services
SAMPLE ID:TP-11580-001
ACQ.METHOD:FA4.2 MIN
17062020-102
512006C4024A
Date of analysis: 17-Jun-2020 19:24:57
Instrument ID :ANL-MCL4-LCMS-SQD-1
4: Diode Array
Range: 4.219e+1
Column - ACQUITY UPLC BEH C18 1.7µm, 2.1X50mm
Mobile phase-A: 0.05%FA IN H2O, B: 0.05% FA IN ACN
T% of B: 0/3, 2.2/98, 3.2/98, 3.5/3, 4.2/3.
Flow-0.6mL/min
Temp:50°C
19 (1:6, dr)
AU
Time
0.23 5665525 67817.06 3.76
0.25 3960196 74338.32 4.13
0.68 71075 5647.83 0.31
0.76 53238 5928.26 0.33
0.99 819846 28415.47 1.58
1.09 117007 4443.96 0.25
1.14 40684 925.82 0.05
1.17 41718 1683.92 0.09
1.21 38013 958.47 0.05
1.24 54973 1412.73 0.08
1.27 81634 3529.95 0.20
1.40 75531 2944.36 0.16
1.42 72087 1895.58 0.11
1.49 56406 1539.02 0.09
1.56 54836 2942.72 0.16
1.60 44533 1053.11 0.06
1.64 193342 4749.59 0.26
1.67 159056 3706.92 0.21
1.71 1350947 27121.04 1.51
1.76 888022 19763.90 1.10
1.81 71983 1154.59 0.06
1.85 296598 9703.14 0.54
1.89 162024 4265.24 0.24
1.94 135903 4285.49 0.24
2.00 2842924 73839.88 4.10
2.11 336778 13312.48 0.74
2.18 100030 4129.08 0.23
2.28 183658 7618.11 0.42
2.33 868688 30653.58 1.70
2.40 84853 2393.15 0.13
2.48 8338940 197678.81 10.97
2.52 587539 9316.63 0.52
2.57 42182372 993274.75 55.12
2.61 955935 24918.94 1.38
2.68 236211 6595.68 0.37
2.71 208578 5983.88 0.33
2.80 493508 24875.72 1.38
2.84 309760 6388.56 0.35
2.88 672742 26314.07 1.46
2.92 146935 2290.94 0.13
2.97 386791 13318.21 0.74
3.04 384965 17015.98 0.94
3.16 1230170 49770.22 2.76
3.88 76758 3502.09 0.19
3.92 76019 4195.78 0.23
4.00 27087 690.26 0.04
4.05 30387 1442.07 0.08
4.09 40822 2261.96 0.13

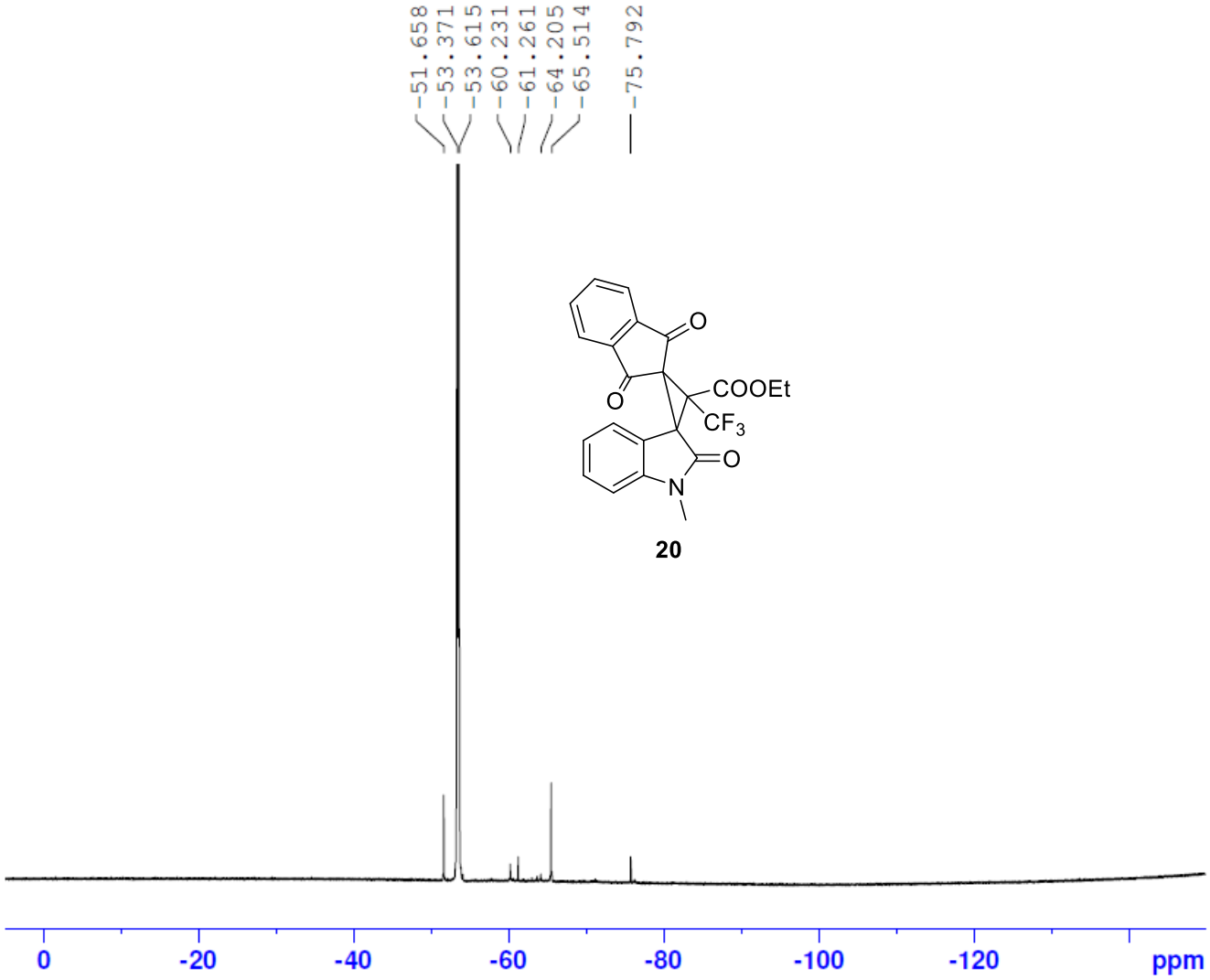


20

```
Current Data Parameters
NAME         512002C3919
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200221
Time                8.15 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zg30
TD                 65536
SOLVENT             CDC13
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                66.5803
DW                61.000 usec
DE                13.89 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.3024719 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                 65536
SF            400.3000091 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-003

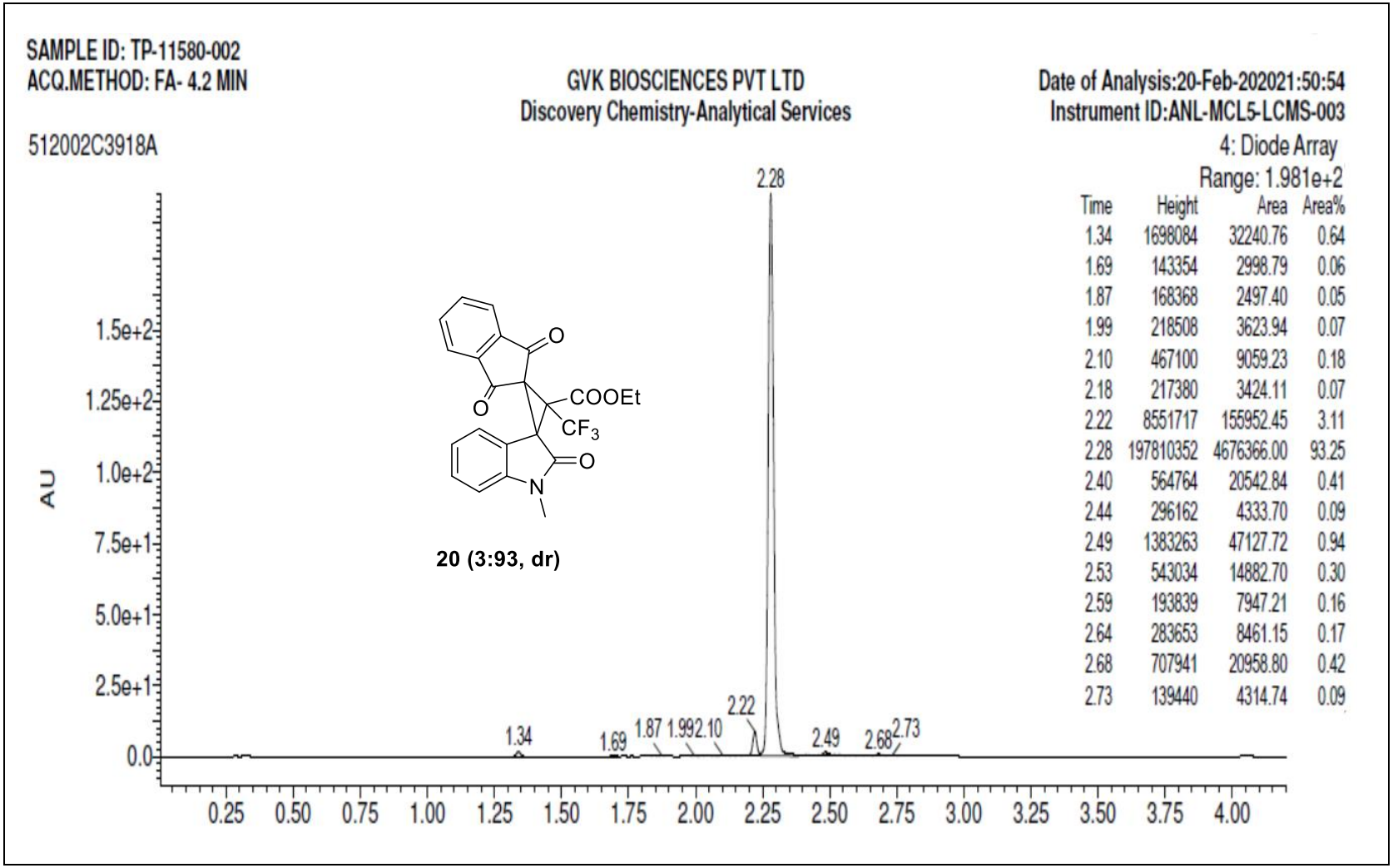

TP-11580-002
13C EXPT
188.916
185.477
185.195
166.475
160.009
144.805
142.355
141.050
135.906
135.176
132.522
131.959
131.708
129.878
129.631
129.181
129.124
127.413
124.599
123.840
123.577
123.362
123.182
122.718
122.043
121.785
119.439
118.867
109.128
108.464
108.017
107.771
77.315
77.201
76.997
76.680
63.089
29.636
26.781
13.606
-0.060
O
O
COOEt
CF3
O
N
20
ANL-MCL5-NMR-003
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

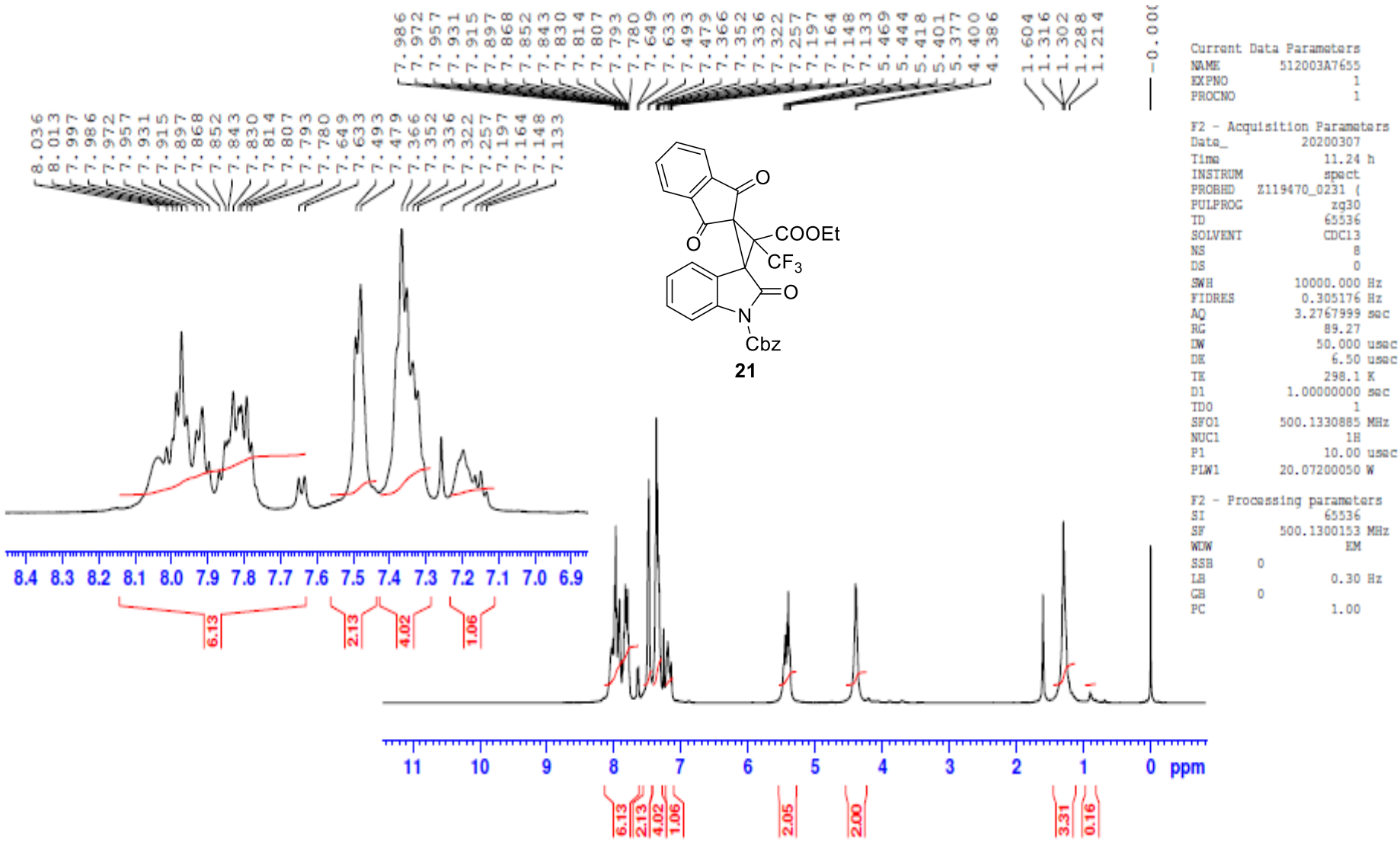


20

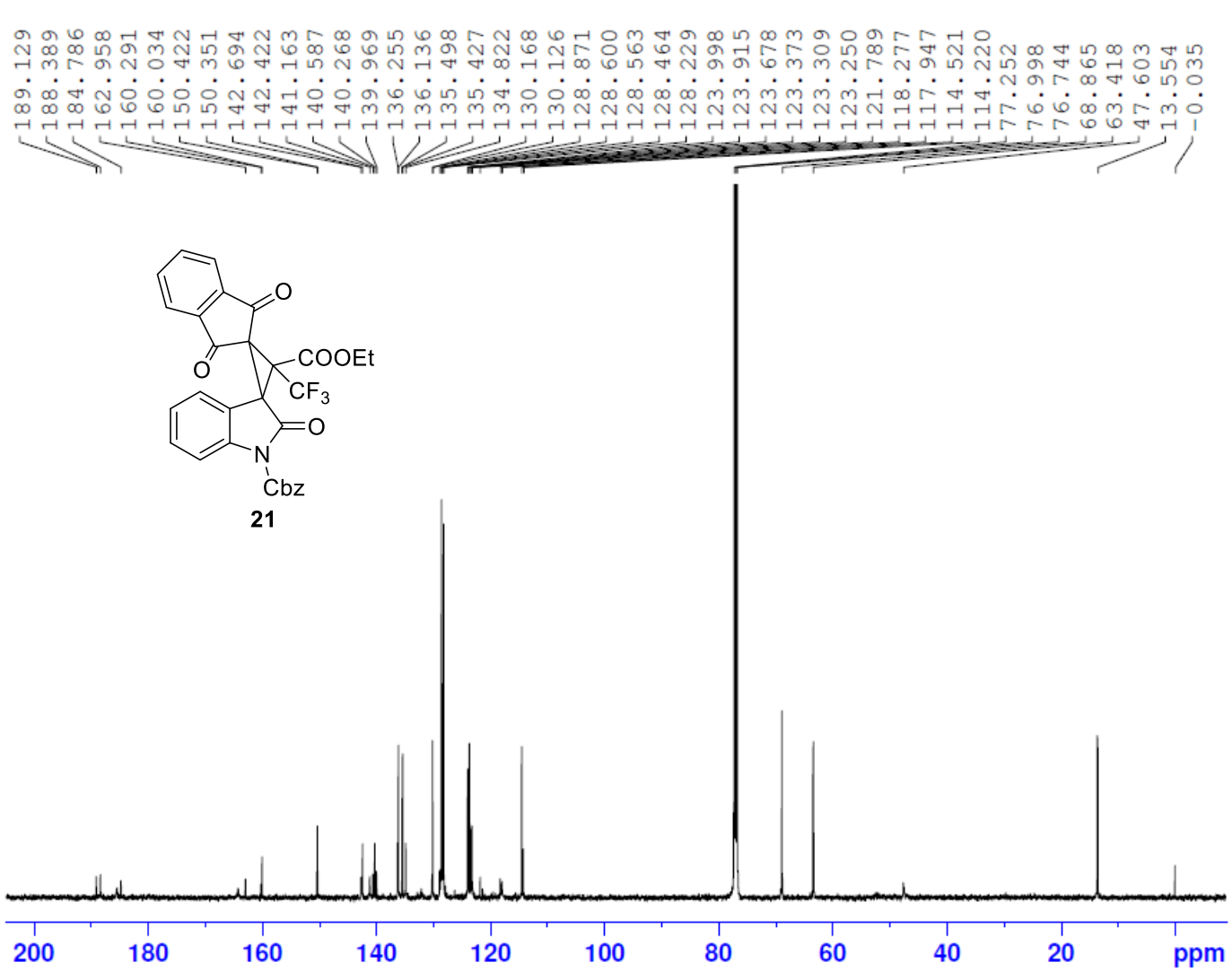
```
Current Data Parameters
NAME         512002C3919
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200221
Time               8.51 h
INSTRUM          Avance
PROBHD   Z163739_0135 (
PULPROG              zg
TD               131072
SOLVENT            CDC13
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1         37.49499893 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                   EM
SSB      0
LB                 2.00 Hz
GB       0
PC                 1.00
```

ANL-MCL5-NMR-003


SAMPLE ID: TP-11580-002
ACQ.METHOD: FA- 4.2 MIN
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:20-Feb-202021:50:54
Instrument ID:ANL-MCL5-LCMS-003
512002C3918A
4: Diode Array
Range: 1.981e+2
AU
1.5e+2
1.25e+2
1.0e+2
7.5e+1
5.0e+1
2.5e+1
0.0
0.25 0.50 0.75 1.00 1.25 1.50 1.75 2.00 2.25 2.50 2.75 3.00 3.25 3.50 3.75 4.00
1.34 1.69 1.87 1.99 2.10 2.22 2.28 2.49 2.68 2.73
O
O
COOEt
CF3
O
N
20 (3:93, dr)
Time Height Area Area%
1.34 1698084 32240.76 0.64
1.69 143354 2998.79 0.06
1.87 168368 2497.40 0.05
1.99 218508 3623.94 0.07
2.10 467100 9059.23 0.18
2.18 217380 3424.11 0.07
2.22 8551717 155952.45 3.11
2.28 197810352 4676366.00 93.25
2.40 564764 20542.84 0.41
2.44 296162 4333.70 0.09
2.49 1383263 47127.72 0.94
2.53 543034 14882.70 0.30
2.59 193839 7947.21 0.16
2.64 283653 8461.15 0.17
2.68 707941 20958.80 0.42
2.73 139440 4314.74 0.09


7.986 7.972 7.957 7.931 7.915 7.897 7.868 7.852 7.843 7.830 7.814 7.807 7.793 7.780 7.649 7.633 7.493 7.479 7.366 7.352 7.336 7.322 7.257 7.197 7.164 7.148 7.133 5.469 5.444 5.418 5.401 5.377 4.400 4.386
1.604 1.316 1.302 1.288 1.214
8.036 8.013 7.997 7.986 7.972 7.957 7.931 7.915 7.897 7.868 7.852 7.843 7.830 7.814 7.807 7.793 7.780 7.649 7.633 7.493 7.479 7.366 7.352 7.336 7.322 7.257 7.197 7.164 7.148 7.133
O
O
COOEt
CF3
O
N
Cbz
21
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
6.13 2.13 4.02 1.06
11 10 9 8 7 6 5 4 3 2 1 0 ppm
6.13 2.13 4.02 1.06 2.05 2.00 3.31 0.16
Current Data Parameters
NAME 512003A7655
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Data_ 20200307
Time 11.24 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 89.27
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 20.07200050 W
F2 - Processing parameters
SI 65536
SF 500.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TP-11580-004-F

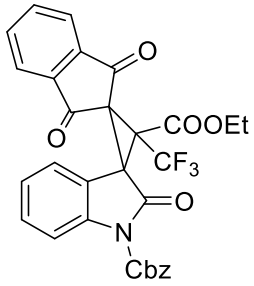


21

```
Current Data Parameters
NAME          512003A7655
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20200308
Time                3.47 h
INSTRUM            spect
PROBHD    Z119470_0231 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3072
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ             1.1010048 sec
RG                197.72
DW                16.800 usec
DE                 6.50 usec
TE                298.2 K
D1            3.00000000 sec
D11           0.03000000 sec
TD0                    1
SFO1        125.7703643 MHz
NUC1                13C
P1                10.00 usec
PLW1         86.78700256 W
SFO2        500.1320005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         20.07200050 W
PLW12         0.31363001 W
PLW13         0.15775000 W

F2 - Processing parameters
SI                 32768
SF          125.7577945 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

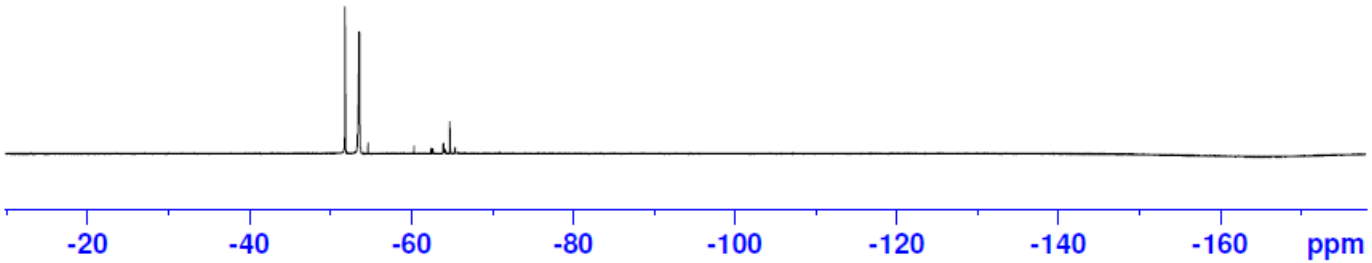
TP-11580-004-F

51.850
53.602
60.386
62.464
62.482
62.642
62.659
63.992
64.214
64.802
65.443



21

```
Current Data Parameters
NAME         512003A7655
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200308
Time                0.11 h
INSTRUM            spect
PROBHD   Z119470_0231 (
PULPROG            zgflqn
TD                 131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ             0.5767168 sec
RG                197.72
DW                 4.400 usec
DE                 6.50 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1
SFO1          470.5453180 MHz
NUC1                 19F
P1                15.00 usec
PLW1          34.86600113 W

F2 - Processing parameters
SI                 65536
SF            470.5923772 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



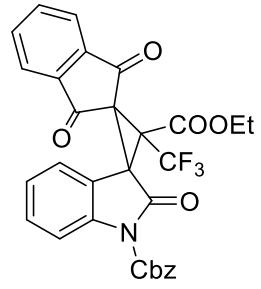
SAMPLE ID: TP-11580-004
ACQ.METHOD: FA- 4.2 MIN

GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services

Date of Analysis:20-Feb-202021:56:12
Instrument ID:ANL-MCL5-LCMS-003

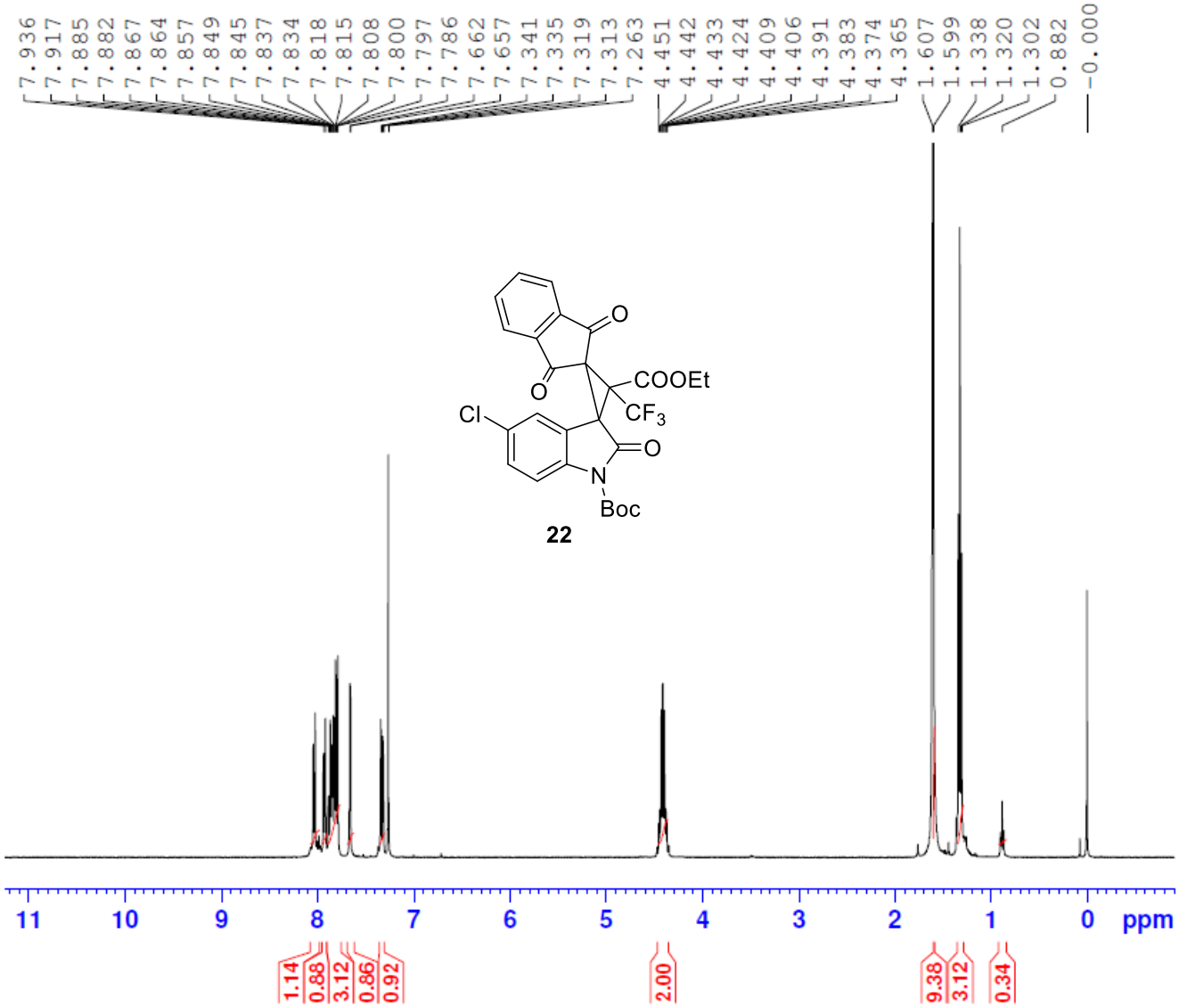
512002C3927A

4: Diode Array
Range: 1.659e+2



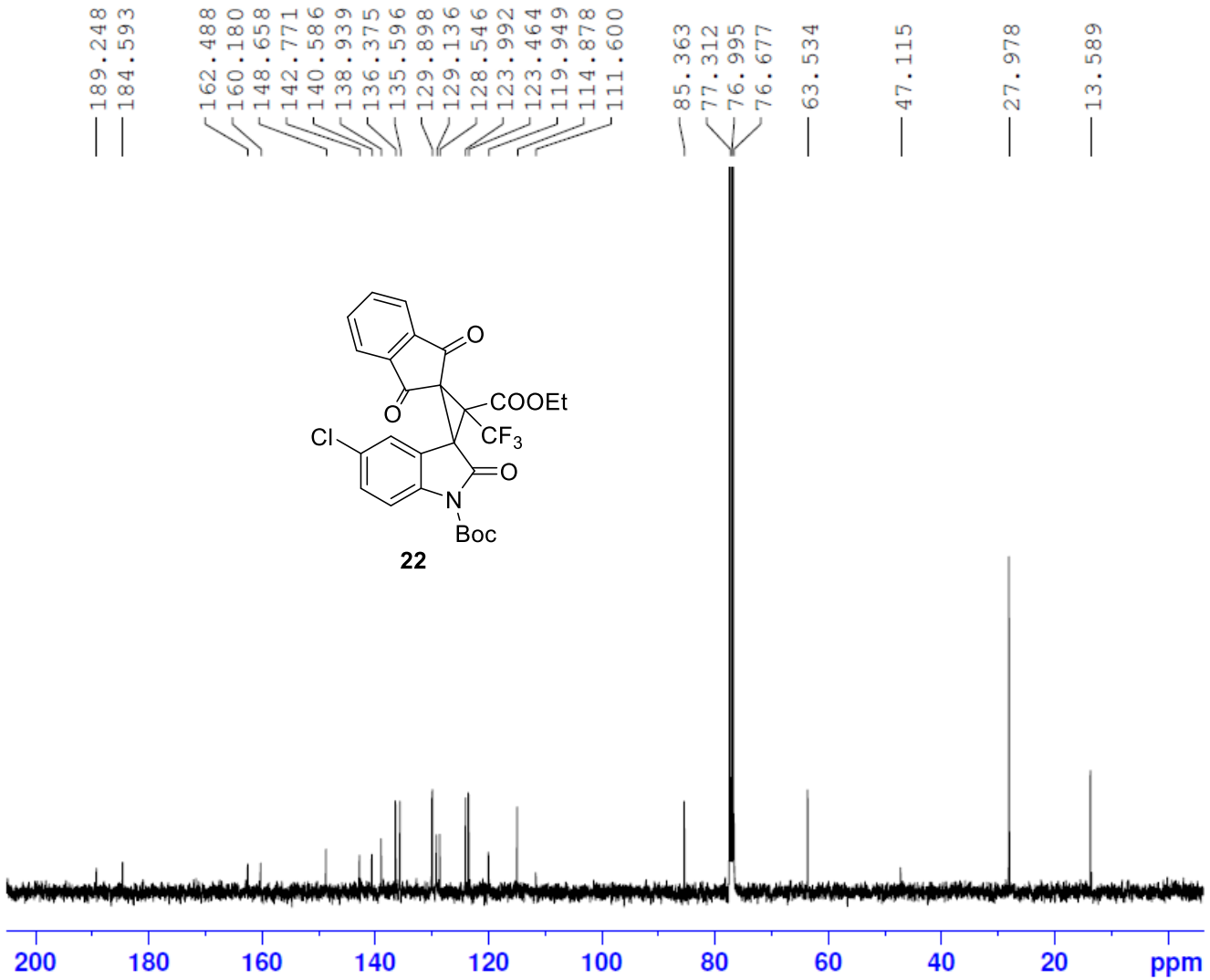
21 (1:99, dr)

Time	Height	Area	Area%
1.54	172277	2610.45	0.06
1.56	162485	3173.73	0.08
1.76	313386	7079.75	0.17
1.88	997858	16600.26	0.41
1.93	220294	3783.36	0.09
2.00	154843	2849.88	0.07
2.11	273393	5277.33	0.13
2.15	101590	2189.57	0.05
2.18	105397	3241.43	0.08
2.51	1889251	17460.24	0.43
2.54	165872448	3921427.25	96.13
2.60	1368061	37724.84	0.92
2.66	479065	19259.46	0.47
2.74	568766	20871.49	0.51
2.78	146927	5031.33	0.12
2.83	105808	3334.38	0.08
2.87	144689	3882.28	0.10
2.90	109808	3502.66	0.09




```
Current Data Parameters
NAME         512001B7992
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200120
Time                7.54 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                   101
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.3024719 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                 65536
SF            400.3000090 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

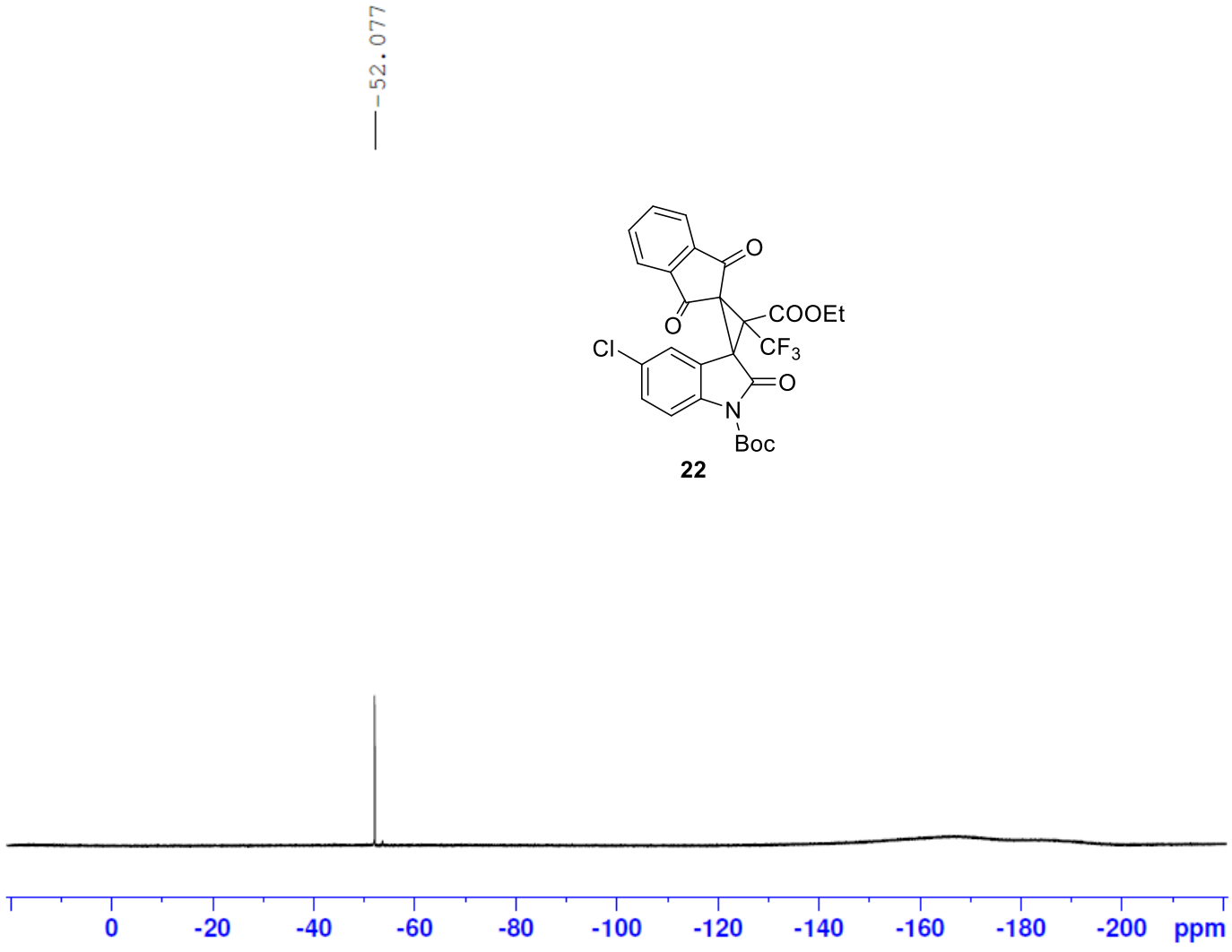
ANL-MCL5-NMR-003




```
Current Data Parameters
NAME          512001B7992
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200120
Time                8.48 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           23809.523 Hz
FIDRES         0.726609 Hz
AQ            1.3762560 sec
RG                  101
DW               21.000 usec
DE                6.50 usec
TE                298.1 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1        100.6655806 MHz
NUC1                13C
P0                 2.67 usec
P1                 8.00 usec
PLW1        92.65799713 W
SFO2        400.3016012 MHz
NUC2                1H
CPDPRG[2         waltz65
PCPD2             90.00 usec
PLW2        22.47400093 W
PLW12        0.17757000 W
PLW13        0.08931800 W

F2 - Processing parameters
SI                32768
SF          100.6555181 MHz
WDW                EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.40
```

ANL-MCL5-NMR-003

TP-11580-005



22

```
Current Data Parameters
NAME         512006A9192
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200608
Time               18.45 h
INSTRUM             spect
PROBHD   Z119470_0294 (
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH            113636.367 Hz
FIDRES           1.733953 Hz
AQ               0.5767168 sec
RG                 197.72
DW                   4.400 usec
DE                    6.50 usec
TE                   301.2 K
D1             1.00000000 sec
D11            0.03000000 sec
D12            0.00002000 sec
TD0                    1
SFO1           470.5453180 MHz
NUC1                  19F
P1                   15.00 usec
PLW1           36.26100159 W
SFO2           500.1320005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                80.00 usec
PLW2           21.30500031 W
PLW12           0.33287999 W

F2 - Processing parameters
SI                     65536
SF             470.5923772 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

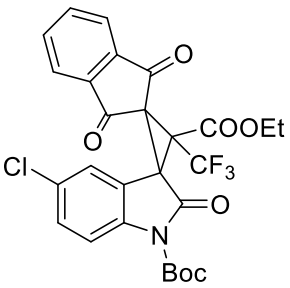
SAMPLE ID: TP-11580-005
ACQ.METHOD: 4.2 MIN
20012020-009
512001B7991A

GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services

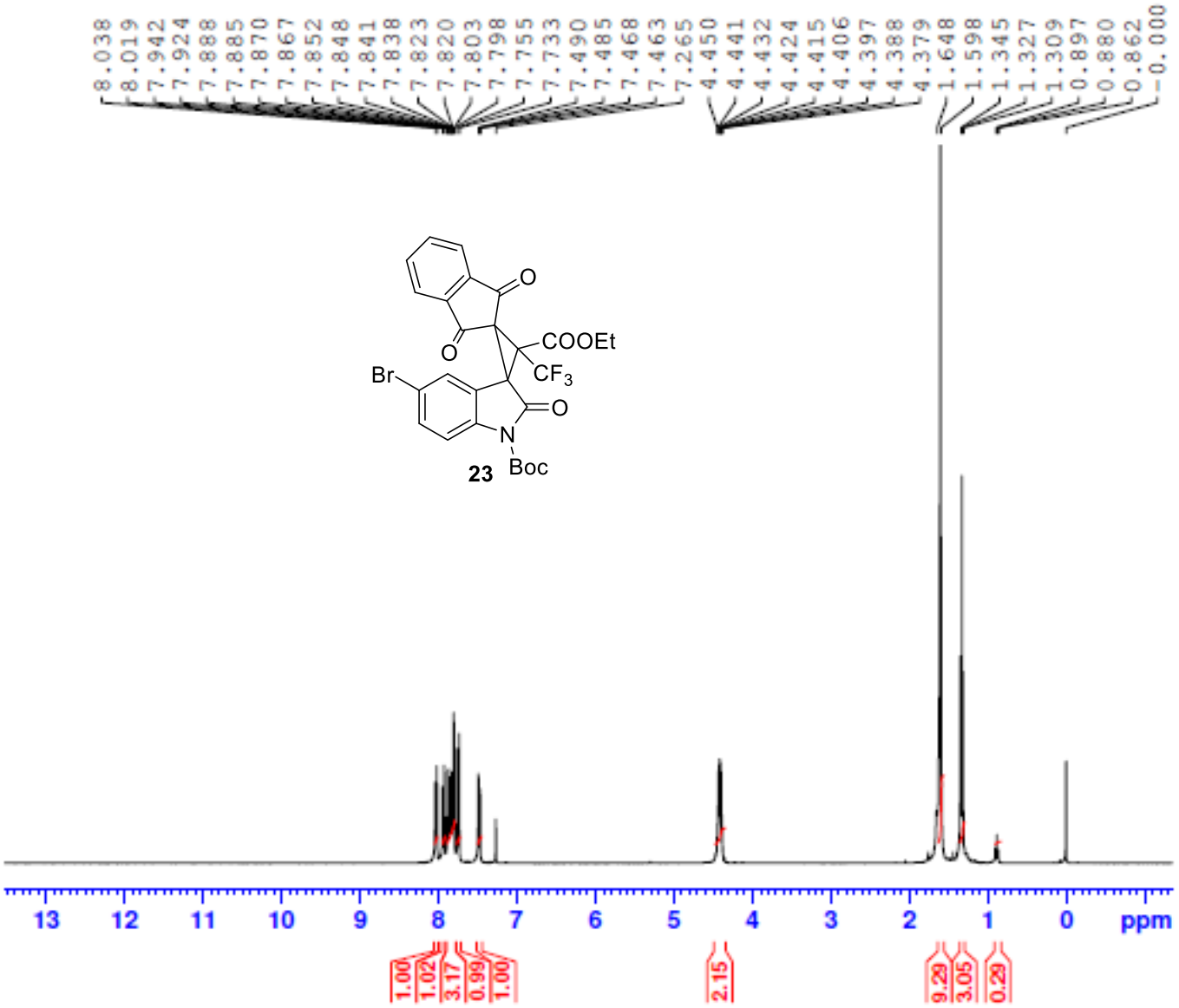
Date of Analysis:20-Jan-202008:38:02
Instrument ID:ANL-MCL5-LCMS-003

4: Diode Array
Range: 1.698e+2

Time	Height	Area	Area%
2.31	241907	7108.46	0.16
2.34	206819	6711.56	0.15
2.39	109550	3117.00	0.07
2.44	126182	5810.67	0.13
2.52	385585	15463.77	0.34
2.56	599602	14679.33	0.32
2.59	194298	4240.30	0.09
2.64	166462032	3845449.50	84.17
2.73	30916052	626347.63	13.71
2.79	579867	21486.41	0.47
2.83	247810	9413.53	0.21
2.89	214917	8651.71	0.19

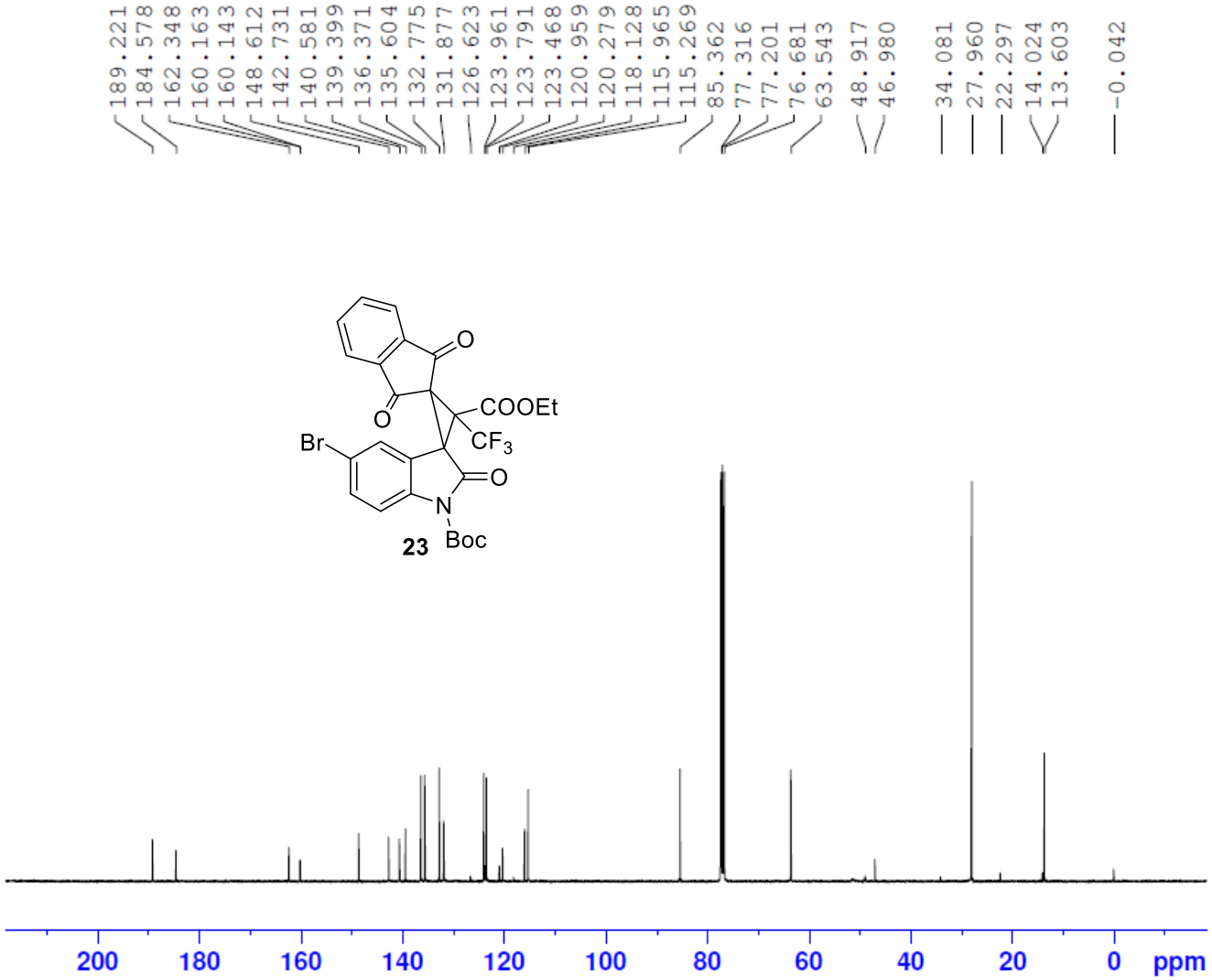


22 (8:1, dr)



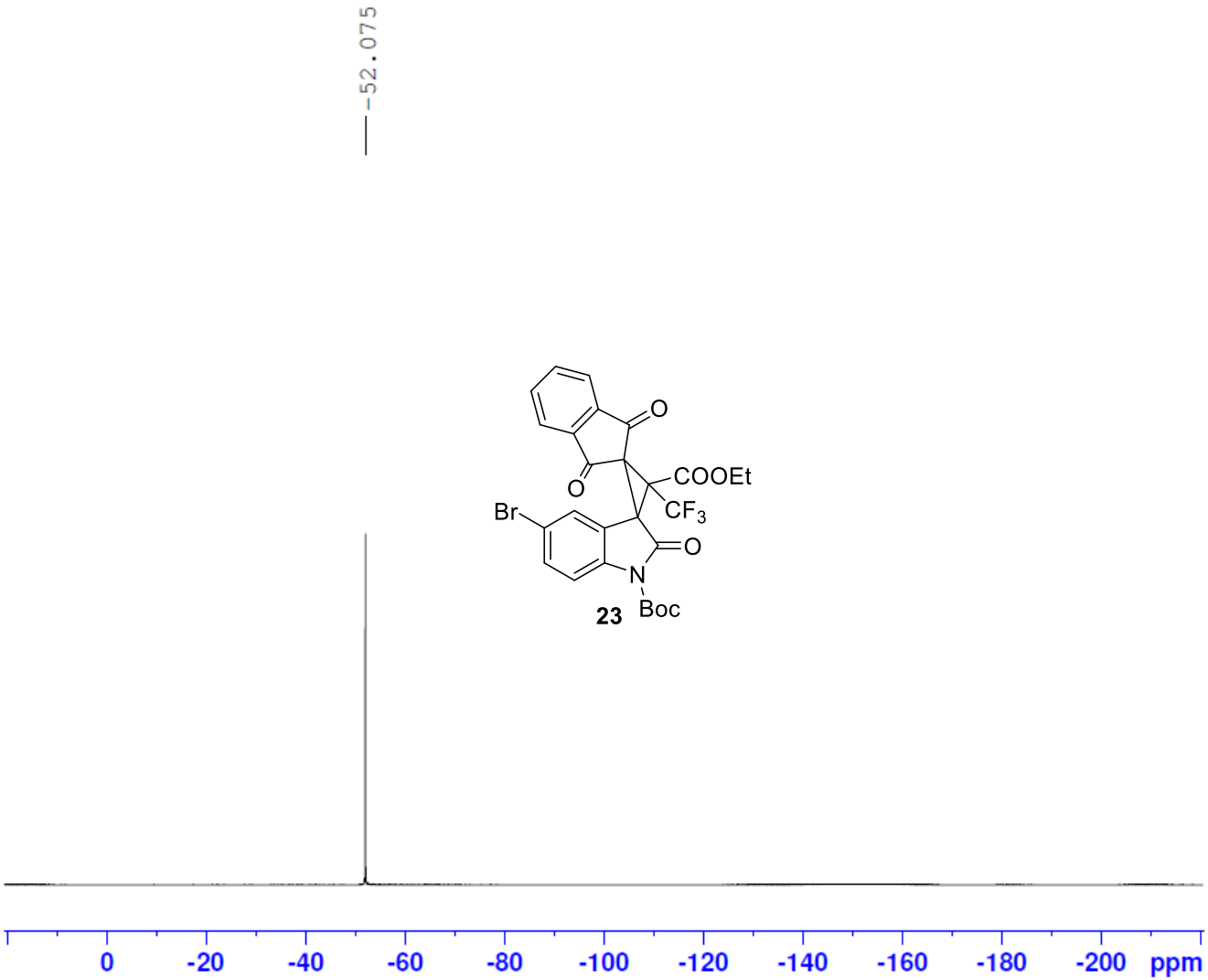

```
Current Data Parameters
NAME         512002C3929
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200221
Time                8.21 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     0
SWH             8196.722 Hz
FIDRES          0.250144 Hz
AQ              3.9976959 sec
RG                66.4296
DW                61.000 usec
DE                13.89 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.3024719 MHz
NUC1                  1H
P0                 2.67 usec
P1                 8.00 usec
PLW1          22.47400093 W

F2 - Processing parameters
SI                 65536
SF          400.3000080 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

ANL-MCL5-NMR-003




```
Current Data Parameters
NAME          512002C3929
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200221
Time                16.25 h
INSTRUM            Avance
PROBHD    Z163739_0135 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3000
DS                      4
SWH             23809.523 Hz
FIDRES          0.726609 Hz
AQ              1.3762560 sec
RG                    101
DW                 21.000 usec
DE                  6.50 usec
TE                 298.2 K
D1              3.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1          100.6655806 MHz
NUC1                  13C
P0                  2.67 usec
P1                  8.00 usec
PLW1          92.65799713 W
SFO2          400.3016012 MHz
NUC2                  1H
CPDPRG[2          waltz65
PCPD2               90.00 usec
PLW2          22.47400093 W
PLW12          0.17757000 W
PLW13          0.08931800 W

F2 - Processing parameters
SI                   32768
SF            100.6555196 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

ANL-MCL5-NMR-003




```
Current Data Parameters
NAME         512002C3929
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200221
Time               12.41 h
INSTRUM           Avance
PROBHD   Z163739_0135 (
PULPROG               zg
TD                131072
SOLVENT            CDC13
NS                    16
DS                     4
SWH            90909.094 Hz
FIDRES          1.387163 Hz
AQ             0.7208960 sec
RG                   101
DW                 5.500 usec
DE                 6.50 usec
TE                298.2 K
D1           1.00000000 sec
TD0                    1
SFO1         376.6206602 MHz
NUC1                 19F
P1                12.00 usec
PLW1         37.49499893 W

F2 - Processing parameters
SI                 65536
SF           376.6583260 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-003

SAMPLE ID: TP-11580-003
ACQ.METHOD: FA- 4.2 MIN

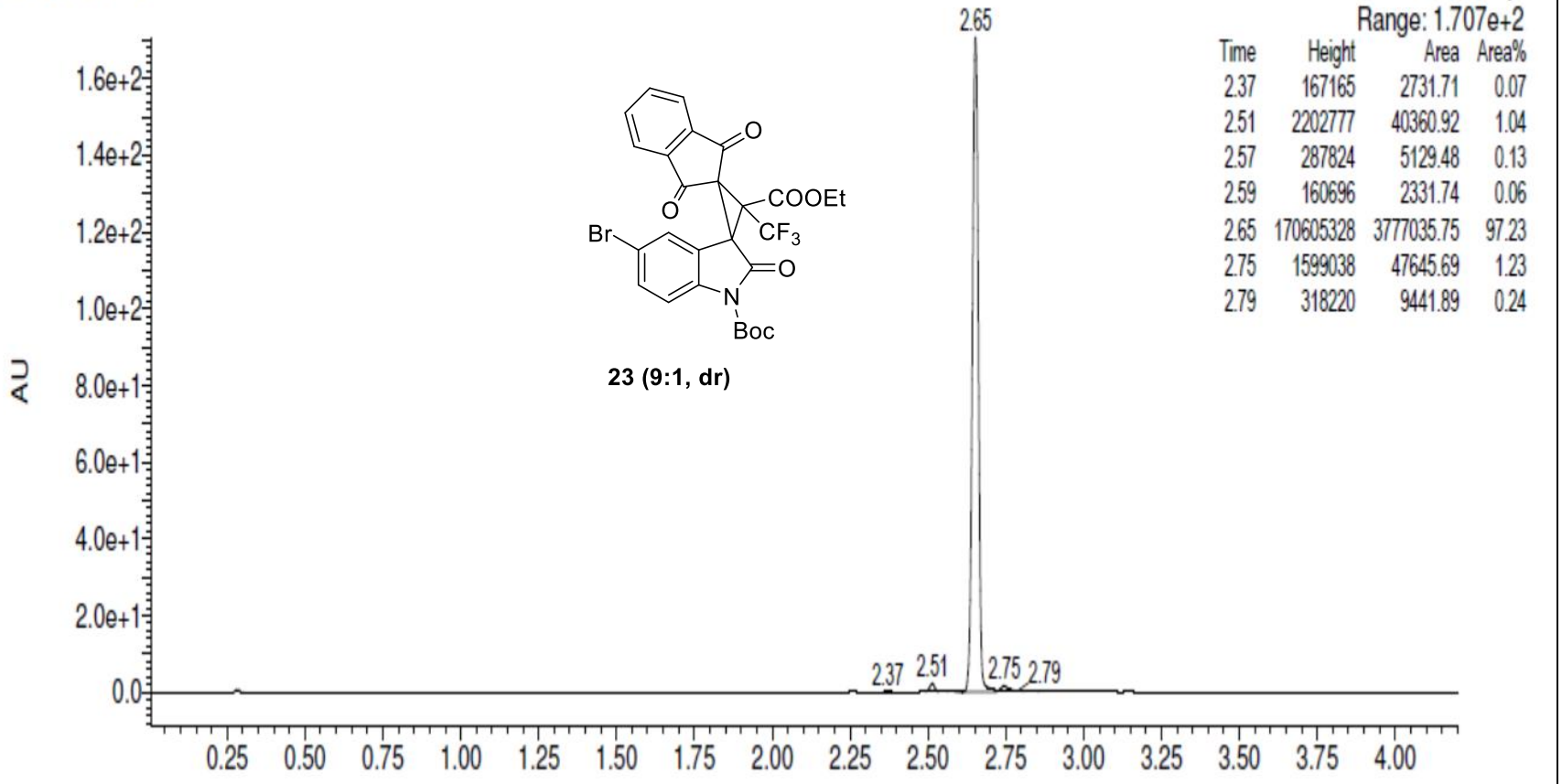
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services

Date of Analysis:20-Feb-202022:01:30
Instrument ID:ANL-MCL5-LCMS-003

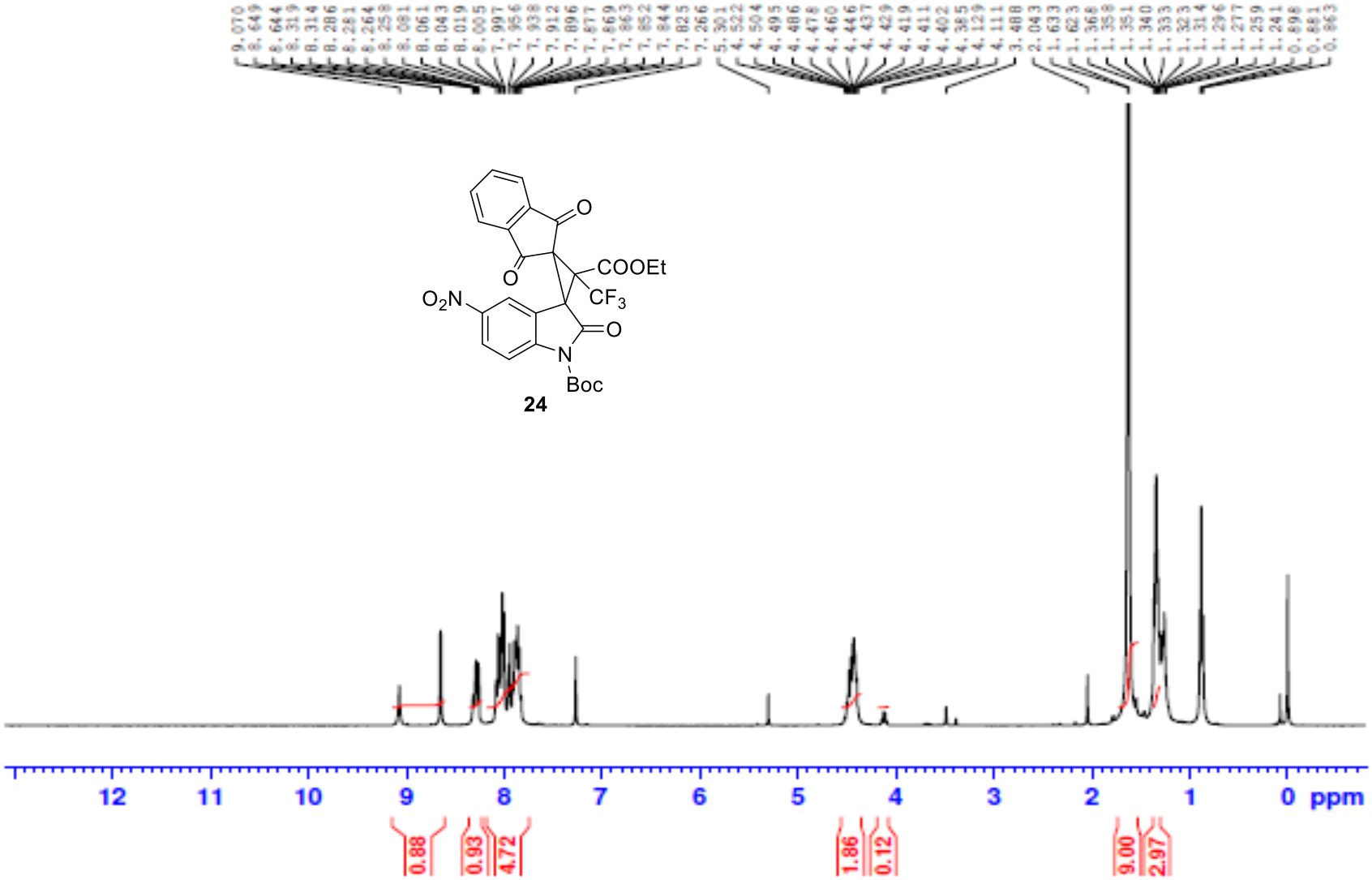
512002C3928A

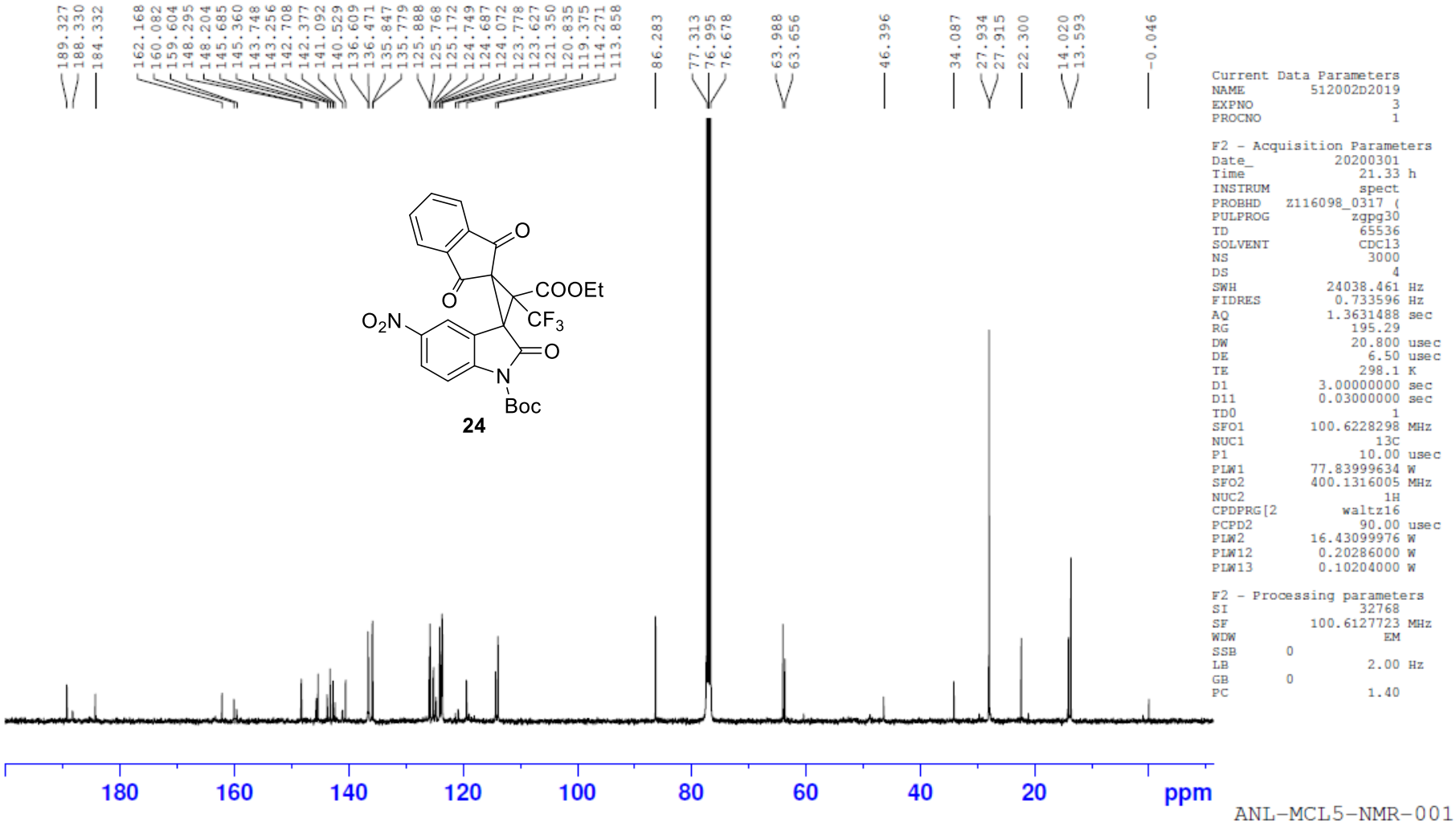
4: Diode Array
Range: 1.707e+2

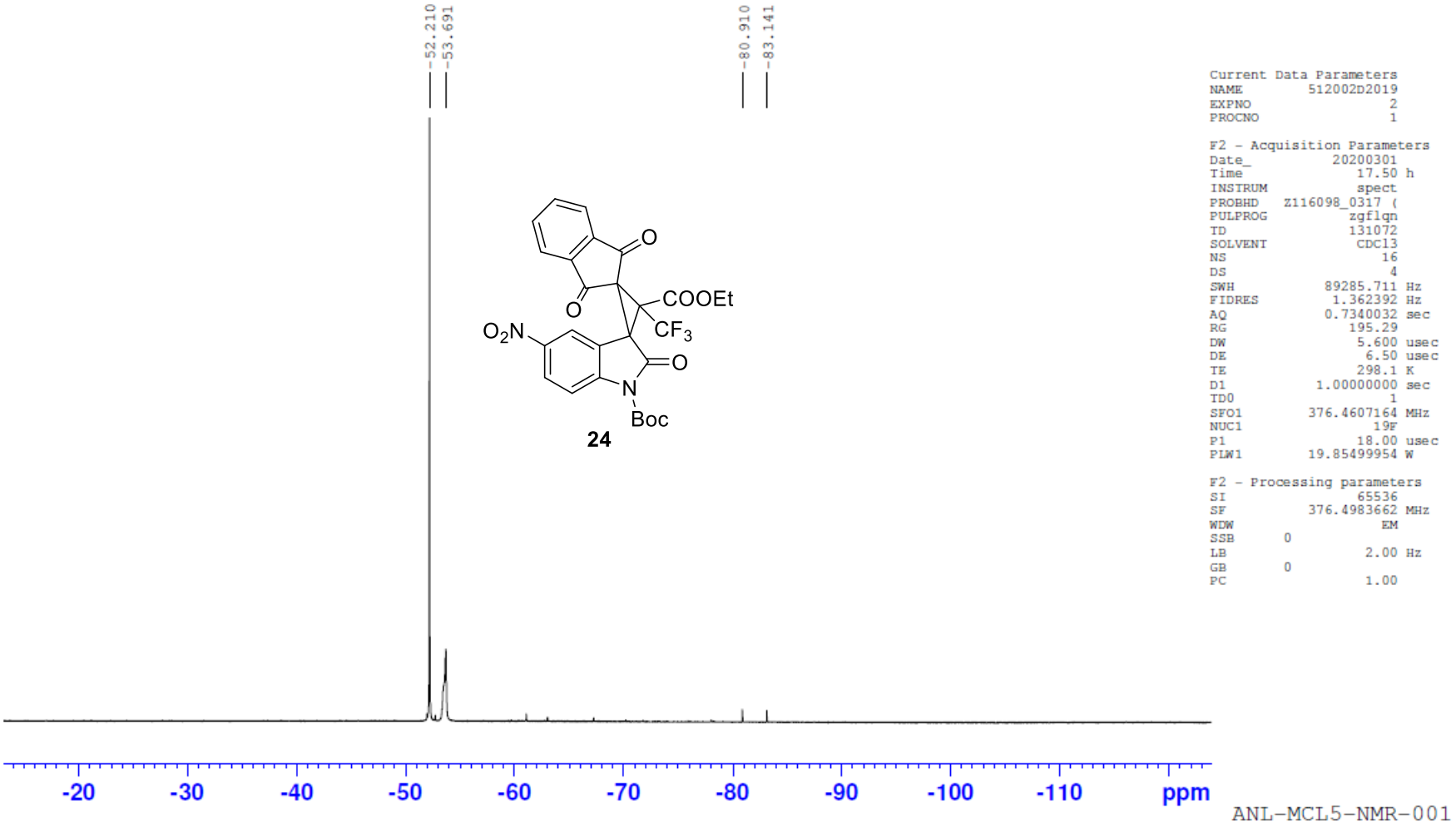
Time	Height	Area	Area%
2.37	167165	2731.71	0.07
2.51	2202777	40360.92	1.04
2.57	287824	5129.48	0.13
2.59	160696	2331.74	0.06
2.65	170605328	3777035.75	97.23
2.75	1599038	47645.69	1.23
2.79	318220	9441.89	0.24



23 (9:1, dr)

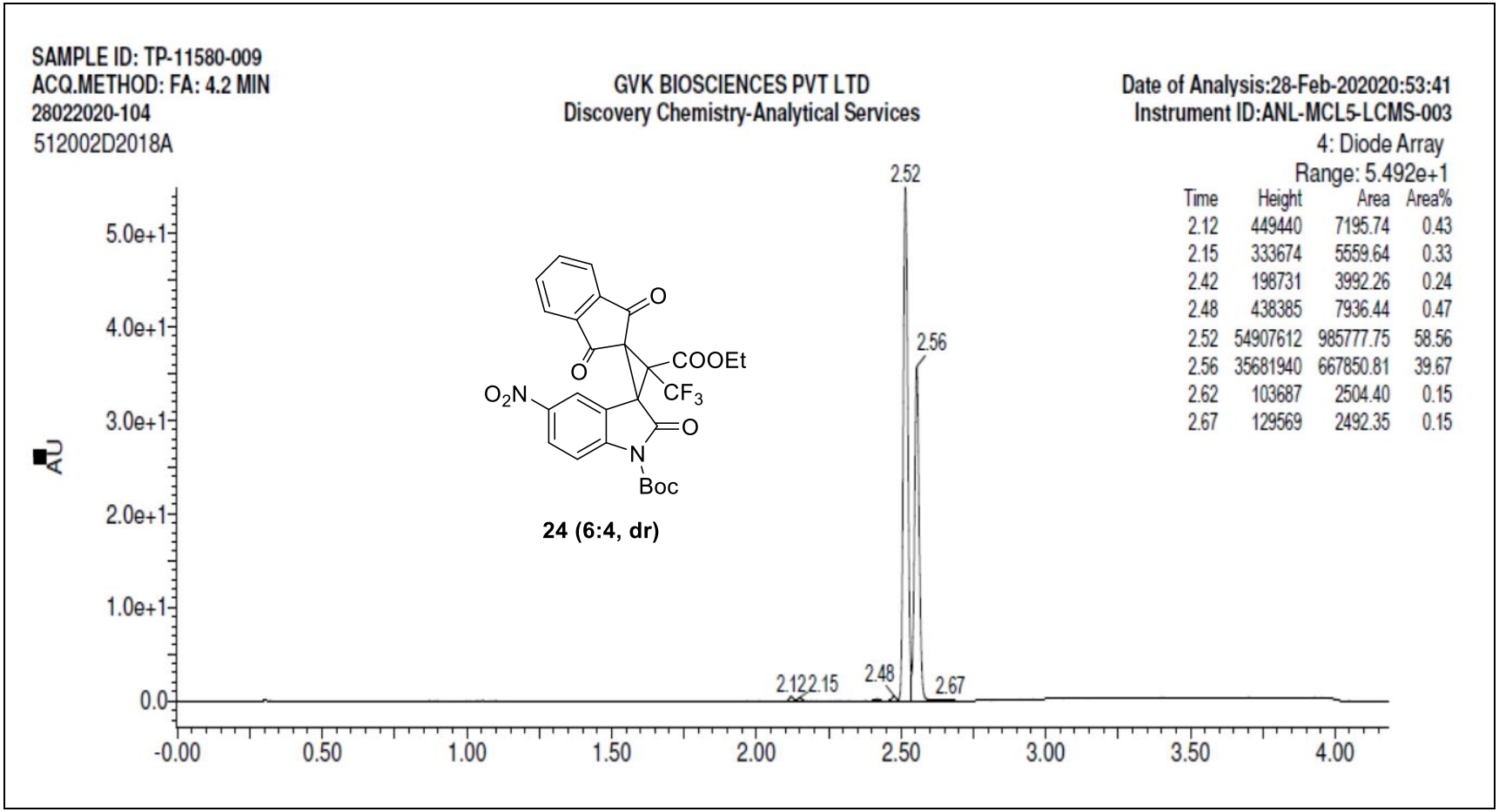

O
O
COOEt
CF3
O2N
O
N
Boc
24
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.88
0.93
4.72
1.86
0.12
9.00
2.97

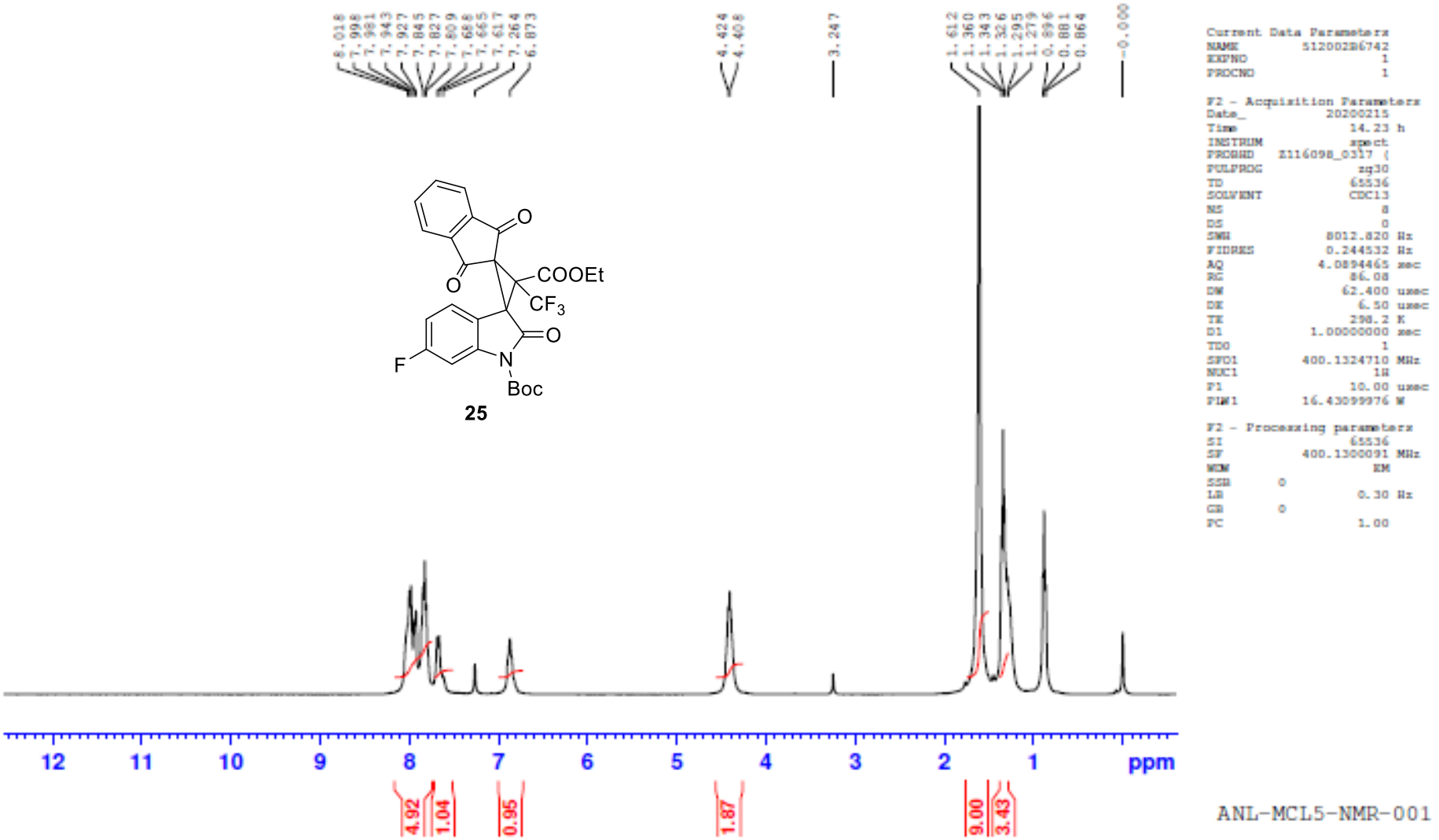

189.327
188.330
184.332
162.168
160.082
159.604
148.295
148.204
145.685
145.360
143.748
143.256
142.708
142.377
141.092
140.529
136.609
136.471
135.847
135.779
125.888
125.768
125.172
124.749
124.687
124.072
123.778
123.627
121.350
120.835
119.375
114.271
113.858
86.283
77.313
76.995
76.678
63.988
63.656
46.396
34.087
27.934
27.915
22.300
14.020
13.593
-0.046
O
O
COOEt
CF3
O2N
O
N
Boc
24
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME 512002D2019
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200301
Time 21.33 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3000
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 195.29
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6228298 MHz
NUC1 13C
P1 10.00 usec
PLW1 77.83999634 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.43099976 W
PLW12 0.20286000 W
PLW13 0.10204000 W
F2 - Processing parameters
SI 32768
SF 100.6127723 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40
ANL-MCL5-NMR-001

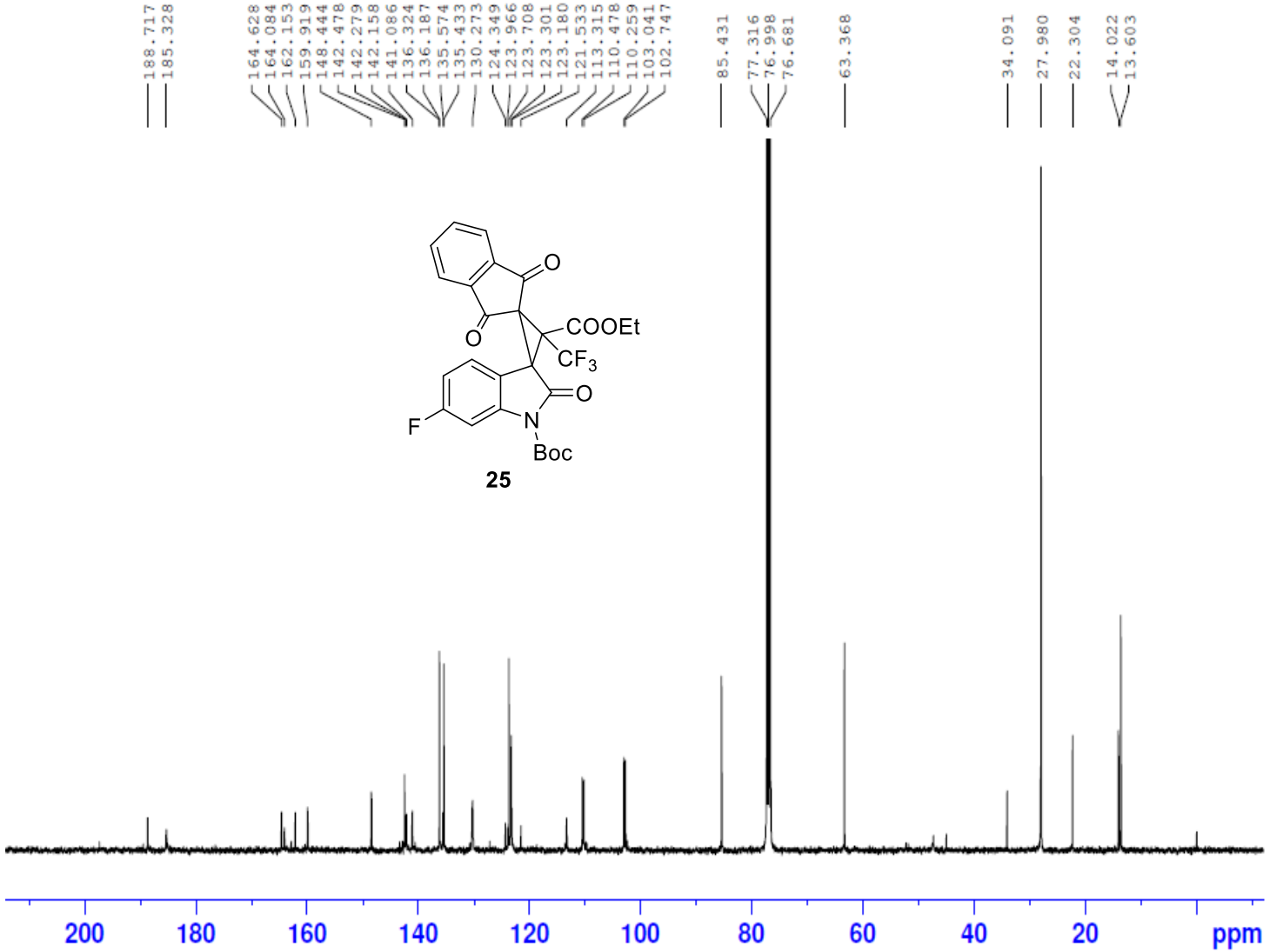



```
Current Data Parameters
NAME         512002D2019
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200301
Time               17.50 h
INSTRUM             spect
PROBHD   Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT             CDCl3
NS                    16
DS                     4
SWH            89285.711 Hz
FIDRES          1.362392 Hz
AQ              0.7340032 sec
RG                195.29
DW                  5.600 usec
DE                  6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1
SFO1          376.4607164 MHz
NUC1                 19F
P1                  18.00 usec
PLW1          19.85499954 W

F2 - Processing parameters
SI                 65536
SF            376.4983662 MHz
WDW                  EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.00
```

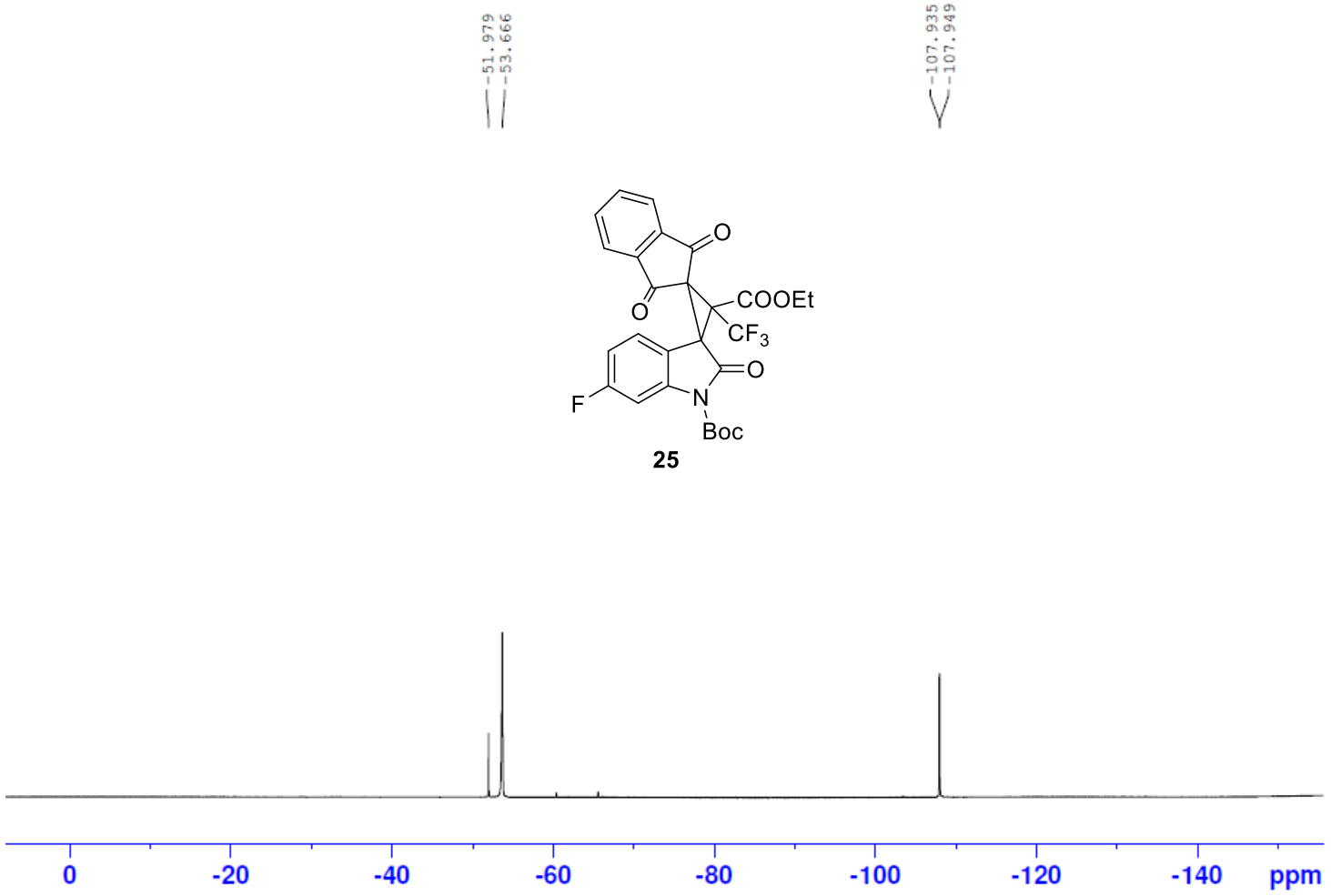
ANL-MCL5-NMR-001


SAMPLE ID: TP-11580-009
ACQ.METHOD: FA: 4.2 MIN
28022020-104
512002D2018A
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:28-Feb-202020:53:41
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 5.492e+1
Time Height Area Area%
2.12 449440 7195.74 0.43
2.15 333674 5559.64 0.33
2.42 198731 3992.26 0.24
2.48 438385 7936.44 0.47
2.52 54907612 985777.75 58.56
2.56 35681940 667850.81 39.67
2.62 103687 2504.40 0.15
2.67 129569 2492.35 0.15
24 (6:4, dr)
AU
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
-0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
2.52
2.56
2.12
2.15
2.48
2.67


8.018
7.998
7.981
7.943
7.927
7.845
7.827
7.809
7.688
7.665
7.617
7.264
6.873
4.424
4.408
3.247
1.612
1.360
1.343
1.326
1.295
1.279
0.896
0.881
0.864
-0.000
O
O
COOEt
CF3
O
F
N
Boc
25
12
11
10
9
8
7
6
5
4
3
2
1
ppm
4.92
1.04
0.95
1.87
9.00
3.43
Current Data Parameters
NAME 512002B6742
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200215
Time 14.23 h
INSTRUM spect
PROBHD Z116098_0317 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 4.0894465 sec
RG 86.08
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.43099976 W
F2 - Processing parameters
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ANL-MCL5-NMR-001




```
Current Data Parameters
NAME          512002B6742
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20200216
Time                0.48 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  3000
DS                     4
SWH           24038.461 Hz
FIDRES         0.733596 Hz
AQ             1.3631488 sec
RG                195.29
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6228298 MHz
NUC1                 13C
P1                10.00 usec
PLW1          77.83999634 W
SFO2          400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2          16.43099976 W
PLW12          0.20286000 W
PLW13          0.10204000 W

F2 - Processing parameters
SI                 32768
SF            100.6127719 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```




```
Current Data Parameters
NAME         512002B6742
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200215
Time              21.05 h
INSTRUM             spect
PROBHD    Z116098_0317 (
PULPROG           zgflqn
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH            89285.711 Hz
FIDRES          1.362392 Hz
AQ              0.7340032 sec
RG                 195.29
DW                  5.600 usec
DE                  6.50 usec
TE                 299.6 K
D1           1.00000000 sec
TD0                    1
SFO1         376.4607164 MHz
NUC1                  19F
P1                 18.00 usec
PLW1         19.85499954 W

F2 - Processing parameters
SI                 65536
SF           376.4983662 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.00
```

ANL-MCL5-NMR-001

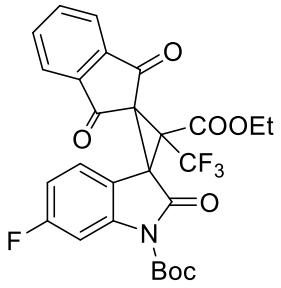
GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services

SAMPLE ID:TP-11580-008
14022020-128
ACQ.METHOD:FA- 4.2 MIN
512002B6741A

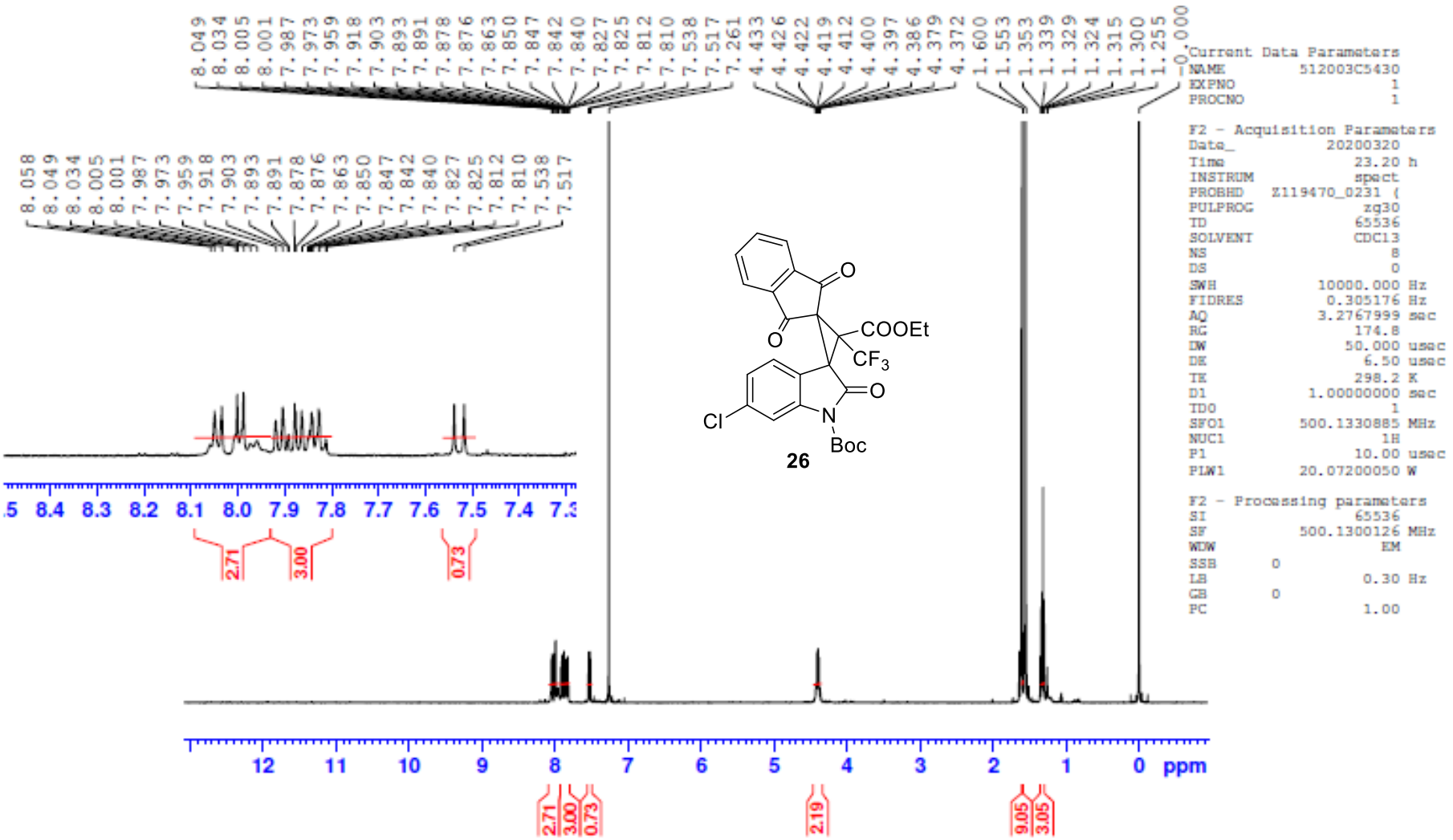
Date of analysis: 14-Feb-202021:37:03
Instrument ID :ANL-MCL4-LCMS-SQD-1

4: Diode Array
Range: 1.83e+2

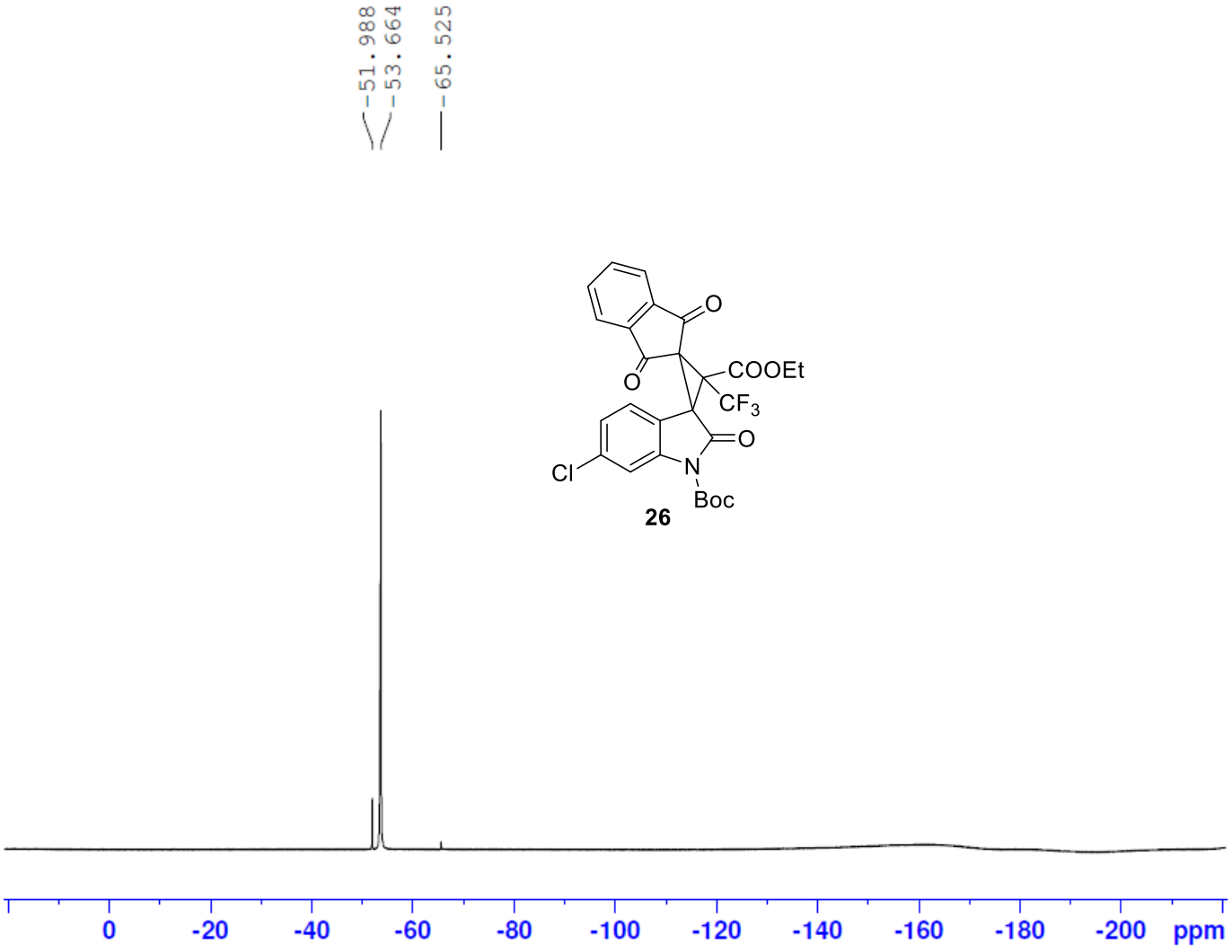
Time	Height	Area	Area%
1.31	12026183	257074.05	5.13
1.76	807245	15288.52	0.30
1.97	601508	8810.07	0.18
2.13	732567	14844.12	0.30
2.18	182142	2338.36	0.05
2.20	447708	7123.56	0.14
2.42	370921	5866.57	0.12
2.57	31183152	560817.88	11.19
2.66	182883664	4141689.50	82.60



25 (1:8, dr)

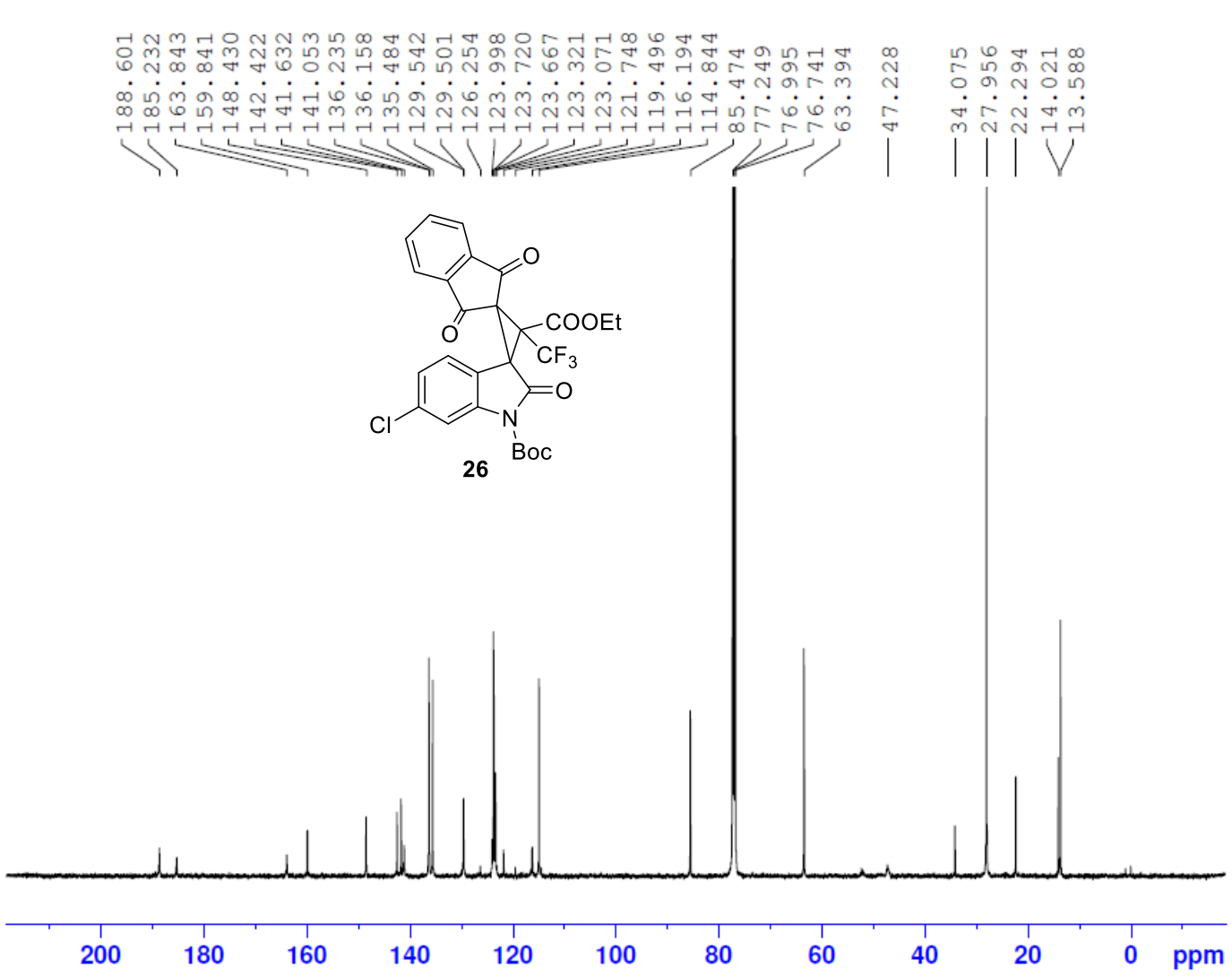

8.049 8.034 8.005 8.001 7.987 7.973 7.959 7.918 7.903 7.893 7.891 7.878 7.876 7.863 7.850 7.847 7.842 7.840 7.827 7.825 7.812 7.810 7.538 7.517 7.261 4.433 4.426 4.422 4.419 4.412 4.400 4.397 4.386 4.379 4.372 1.600 1.553 1.353 1.339 1.329 1.324 1.315 1.300 1.255 0.000
8.058 8.049 8.034 8.005 8.001 7.987 7.973 7.959 7.918 7.903 7.893 7.891 7.878 7.876 7.863 7.850 7.847 7.842 7.840 7.827 7.825 7.812 7.810 7.538 7.517
O
O
COOEt
CF3
O
Cl
N
Boc
26
2.71
3.00
0.73
2.19
9.05
3.05
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
Current Data Parameters
NAME 512003C5430
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Data_ 20200320
Time 23.20 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 174.8
DW 50.000 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 20.07200050 W
F2 - Processing parameters
SI 65536
SF 500.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

TP-11580-007

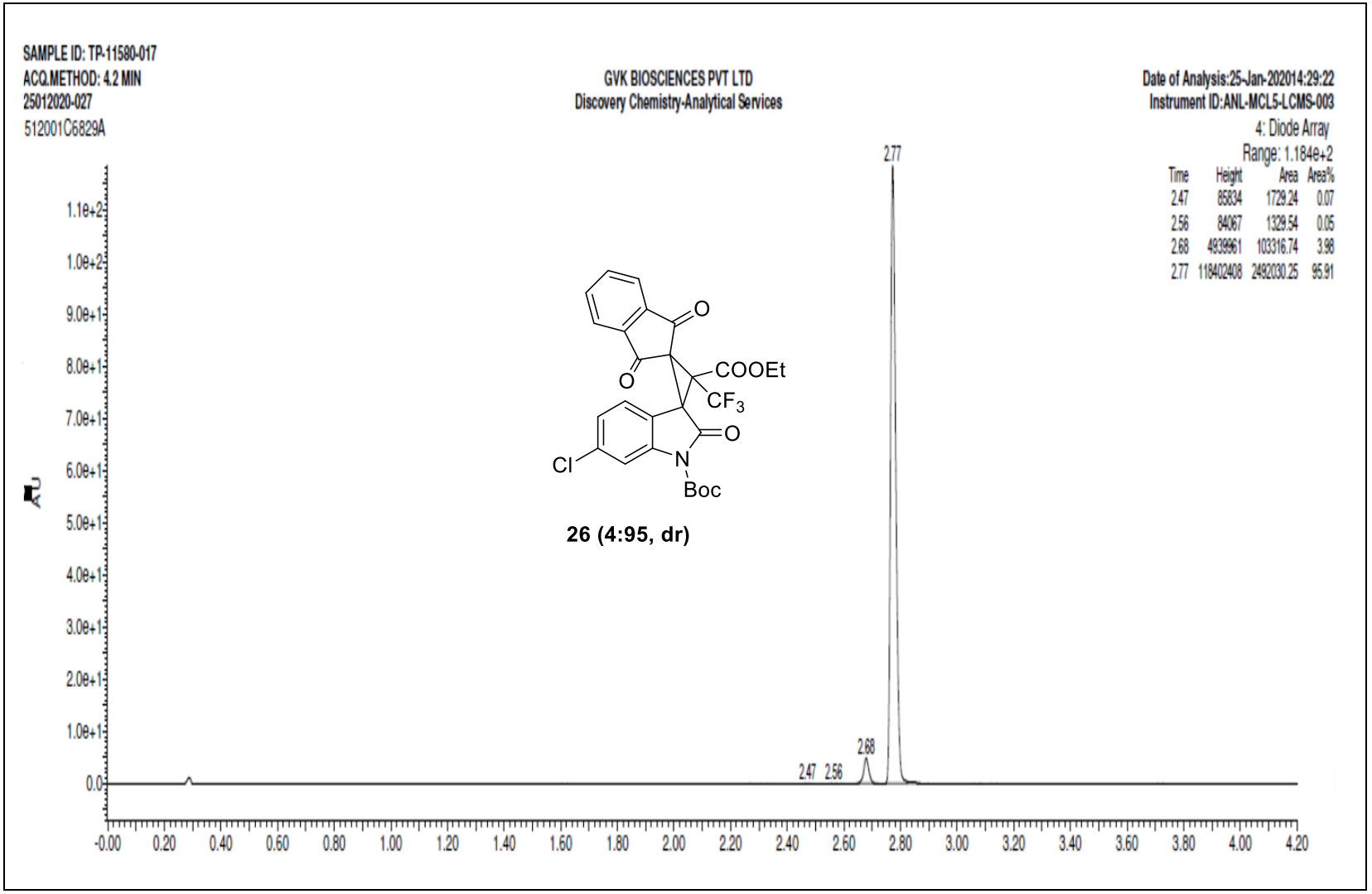



```
Current Data Parameters
NAME         512001C6921
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200126
Time                1.18 h
INSTRUM            spect
PROBHD   Z119470_0231 (
PULPROG           zgflqn
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ              0.5767168 sec
RG                 197.72
DW                  4.400 usec
DE                  6.50 usec
TE                 369.6 K
D1             1.00000000 sec
TD0                    1
SFO1          470.5453180 MHz
NUC1                  19F
P1                  15.00 usec
PLW1          34.86600113 W

F2 - Processing parameters
SI                 65536
SF            470.5923772 MHz
WDW                   EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.00
```

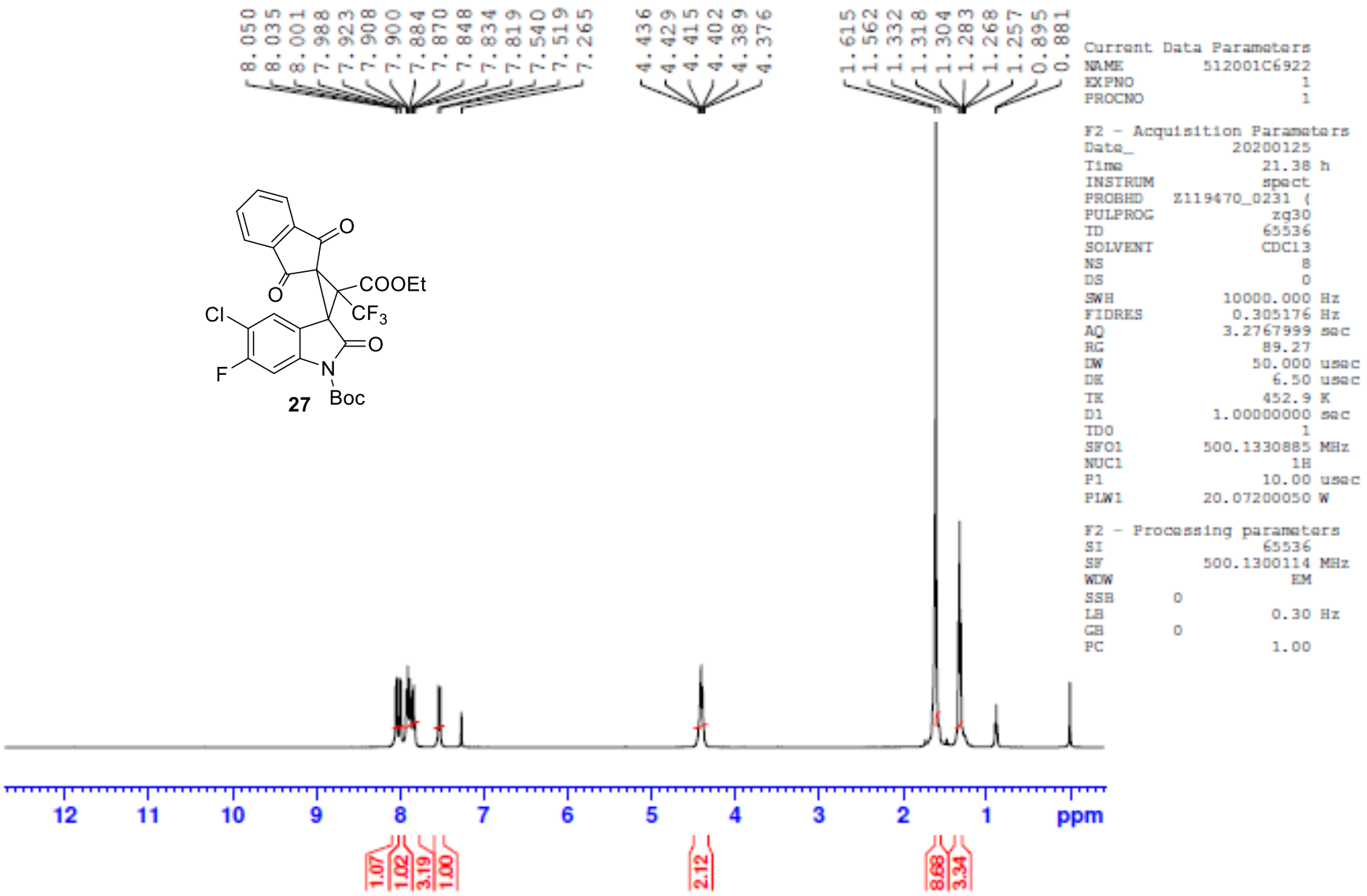
TP-11580-007



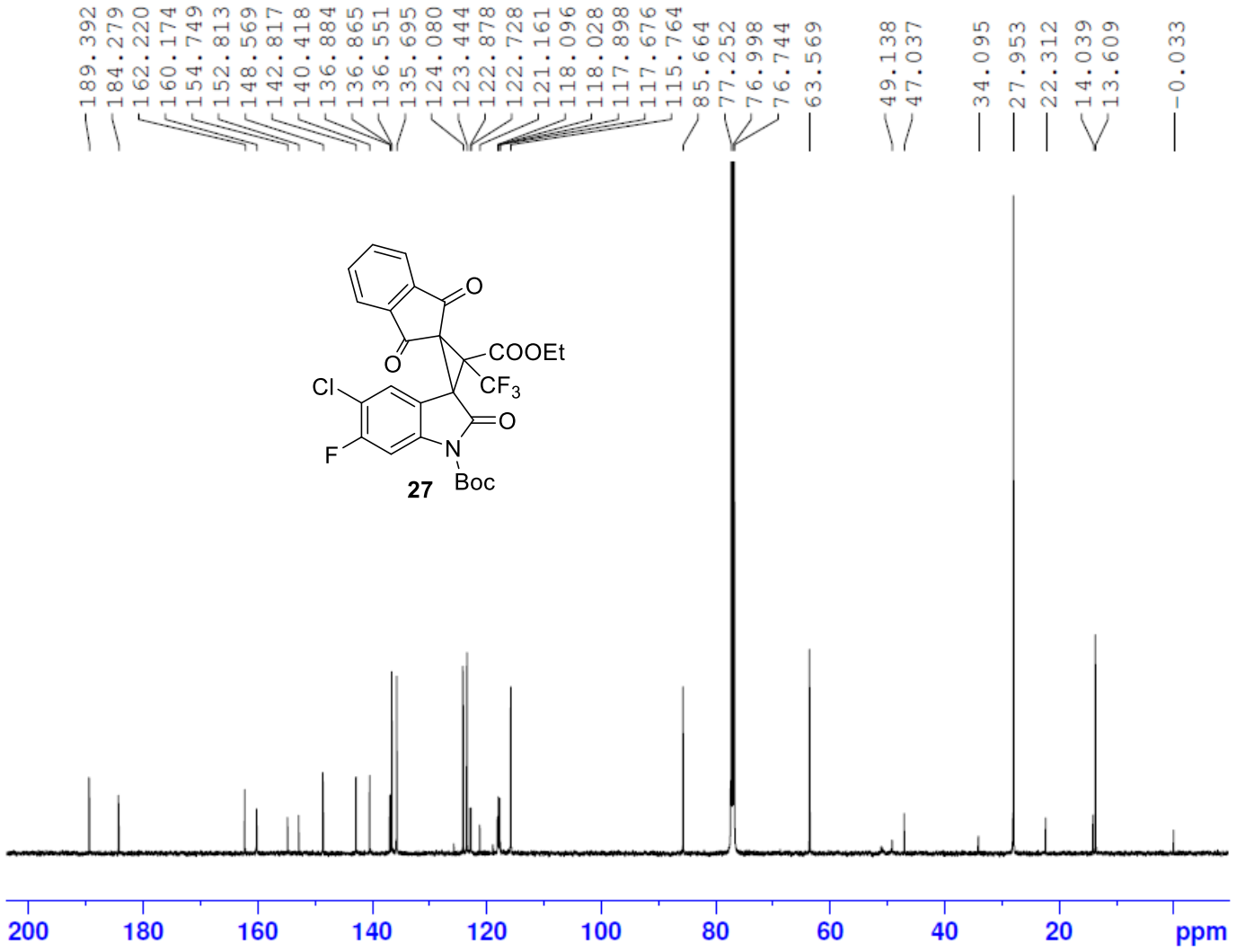

```
Current Data Parameters
NAME         512001C6921
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200126
Time                3.58 h
INSTRUM            spect
PROBHD   Z119470_0231 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3000
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ            1.1010048 sec
RG               197.72
DW               16.800 usec
DE                6.50 usec
TE               343.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
SFO1         125.7703643 MHz
NUC1                 13C
P1                10.00 usec
PLW1         86.78700256 W
SFO2         500.1320005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2             80.00 usec
PLW2         20.07200050 W
PLW12         0.31363001 W
PLW13         0.15775000 W

F2 - Processing parameters
SI                32768
SF           125.7577959 MHz
WDW                  EM
SSB       0
LB                2.00 Hz
GB        0
PC                1.40
```


SAMPLE ID: TP-11580-017
ACQ.METHOD: 4.2 MIN
25012020-027
512001C6829A
GVK BIOSCIENCES PVT LTD
Discovery Chemistry-Analytical Services
Date of Analysis:25-Jan-202014:29:22
Instrument ID:ANL-MCL5-LCMS-003
4: Diode Array
Range: 1.184e+2
Time Height Area Area%
2.47 85834 1729.24 0.07
2.56 84067 1329.54 0.05
2.68 4939961 103316.74 3.98
2.77 118402408 2492030.25 95.91
2.77
2.68
2.47 2.56
AU
1.1e+2
1.0e+2
9.0e+1
8.0e+1
7.0e+1
6.0e+1
5.0e+1
4.0e+1
3.0e+1
2.0e+1
1.0e+1
0.0
-0.00 0.20 0.40 0.60 0.80 1.00 1.20 1.40 1.60 1.80 2.00 2.20 2.40 2.60 2.80 3.00 3.20 3.40 3.60 3.80 4.00 4.20
O
O
COOEt
CF3
O
Cl
N
Boc

26 (4:95, dr)

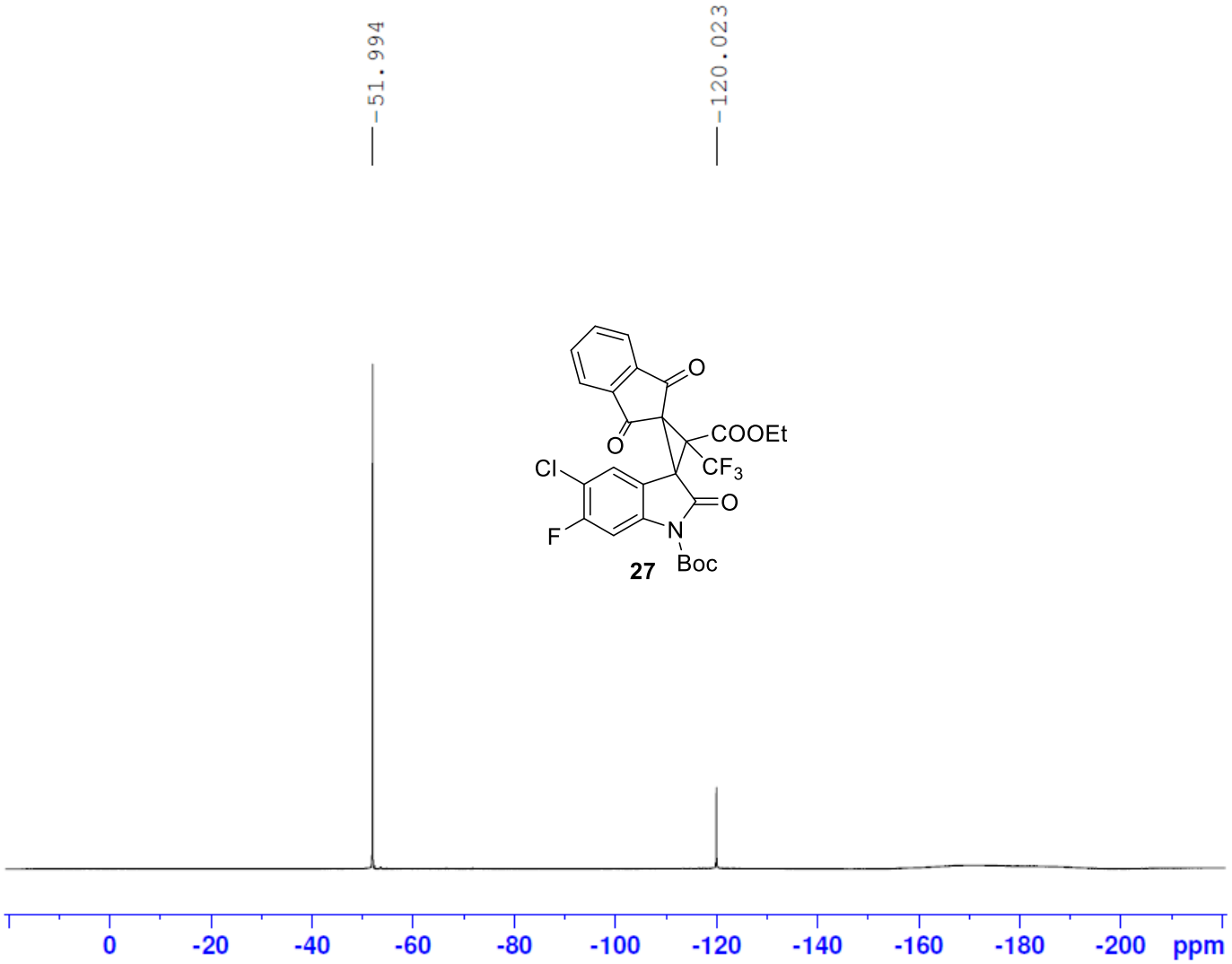

8.050
8.035
8.001
7.988
7.923
7.908
7.900
7.884
7.870
7.848
7.834
7.819
7.540
7.519
7.265
4.436
4.429
4.415
4.402
4.389
4.376
1.615
1.562
1.332
1.318
1.304
1.283
1.268
1.257
0.895
0.881
O
O
COOEt
Cl
CF3
O
F
N
Boc
27
Current Data Parameters
NAME 512001C6922
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200125
Time 21.38 h
INSTRUM spect
PROBHD Z119470_0231 (
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 89.27
DW 50.000 usec
DE 6.50 usec
TE 452.9 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 20.07200050 W
F2 - Processing parameters
SI 65536
SF 500.1300114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.07
1.02
3.19
1.00
2.12
8.68
3.34

TP-11580-006-001




```
Current Data Parameters
NAME         512001C6922
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200126
Time                1.11 h
INSTRUM            spect
PROBHD   Z119470_0231 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3000
DS                    4
SWH           29761.904 Hz
FIDRES         0.908261 Hz
AQ             1.1010048 sec
RG                197.72
DW                16.800 usec
DE                 6.50 usec
TE                371.7 K
D1             3.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          125.7703643 MHz
NUC1                  13C
P1                 10.00 usec
PLW1          86.78700256 W
SFO2          500.1320005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          20.07200050 W
PLW12          0.31363001 W
PLW13          0.15775000 W

F2 - Processing parameters
SI                 32768
SF            125.7577935 MHz
WDW                   EM
SSB       0
LB                 2.00 Hz
GB        0
PC                 1.40
```

TP-11580-006-001

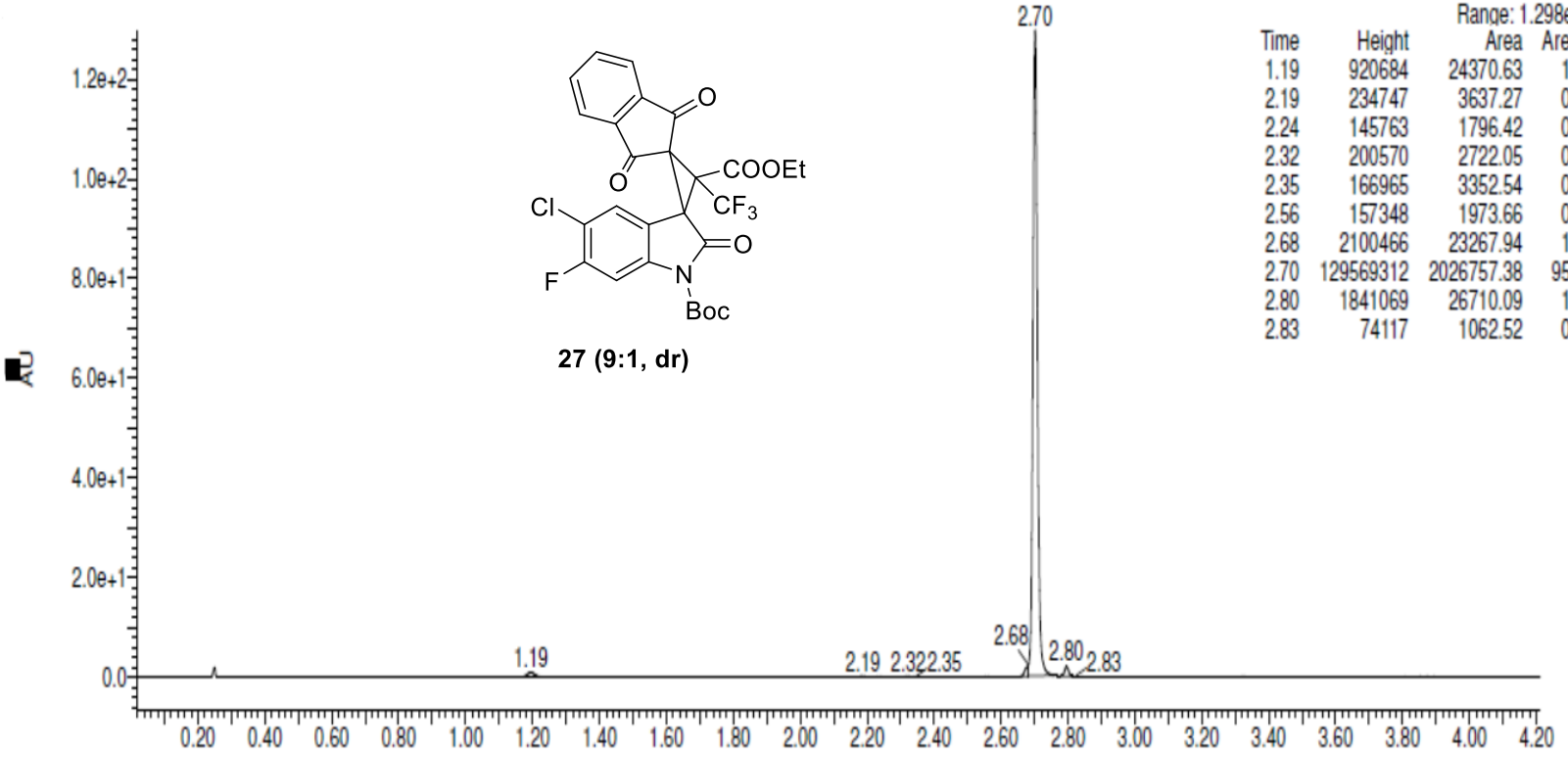



```
Current Data Parameters
NAME         512001C6922
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20200125
Time               21.40 h
INSTRUM             spect
PROBHD    Z119470_0231 (
PULPROG           zgflqn
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          1.733953 Hz
AQ              0.5767168 sec
RG                 197.72
DW                  4.400 usec
DE                  6.50 usec
TE                 452.9 K
D1            1.00000000 sec
TD0                    1
SFO1          470.5453180 MHz
NUC1                  19F
P1                 15.00 usec
PLW1          34.86600113 W

F2 - Processing parameters
SI                 65536
SF            470.5923772 MHz
WDW                   EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.00
```

GVK Biosciences pvt Ltd.
Discovery Chemistry-Analytical Services

SAMPLE ID:TP-11580-006-001
25012020-069
ACQ.METHOD:FA- 4.2 MIN
512001C6977A

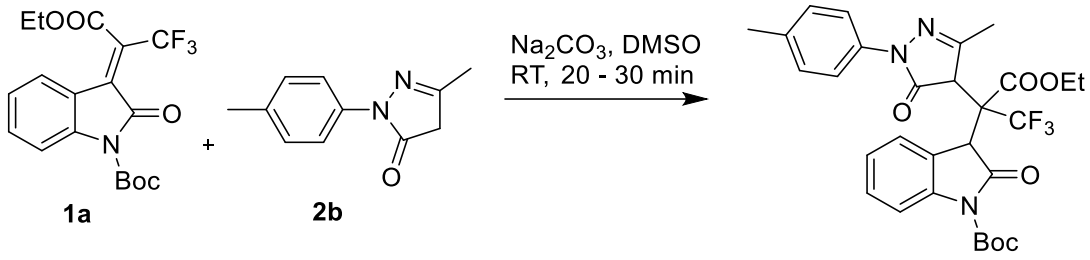
Date of analysis: 25-Jan-202019:10:13
Instrument ID :ANL-MCL4-LCMS-SQD-1



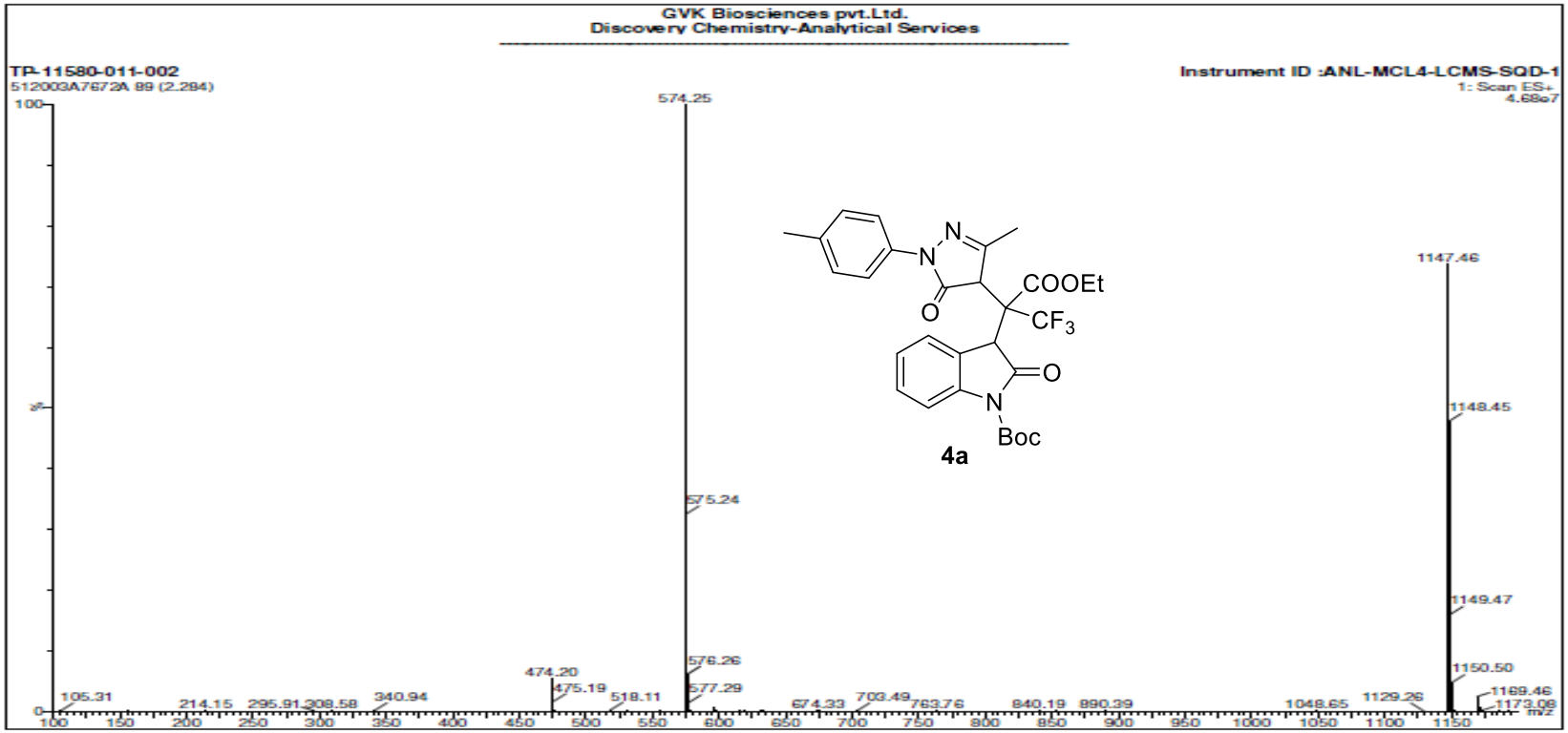
27 (9:1, dr)

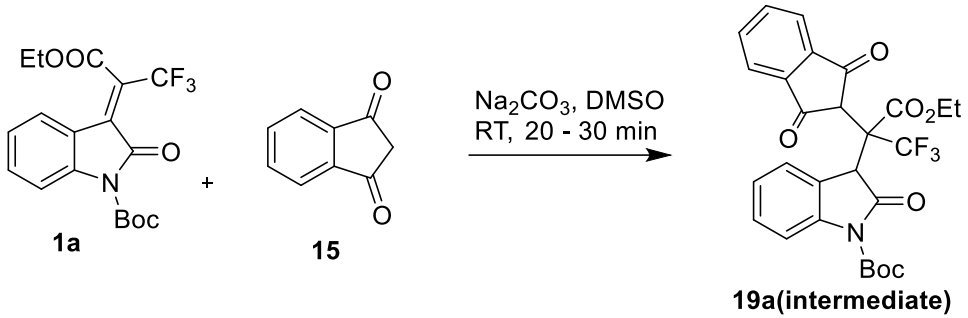
4: Diode Array
Range: 1.298e+2

Time	Height	Area	Area%
1.19	920684	24370.63	1.15
2.19	234747	3637.27	0.17
2.24	145763	1796.42	0.08
2.32	200570	2722.05	0.13
2.35	166965	3352.54	0.16
2.56	157348	1973.66	0.09
2.68	2100466	23267.94	1.10
2.70	129569312	2026757.38	95.80
2.80	1841069	26710.09	1.26
2.83	74117	1062.52	0.05

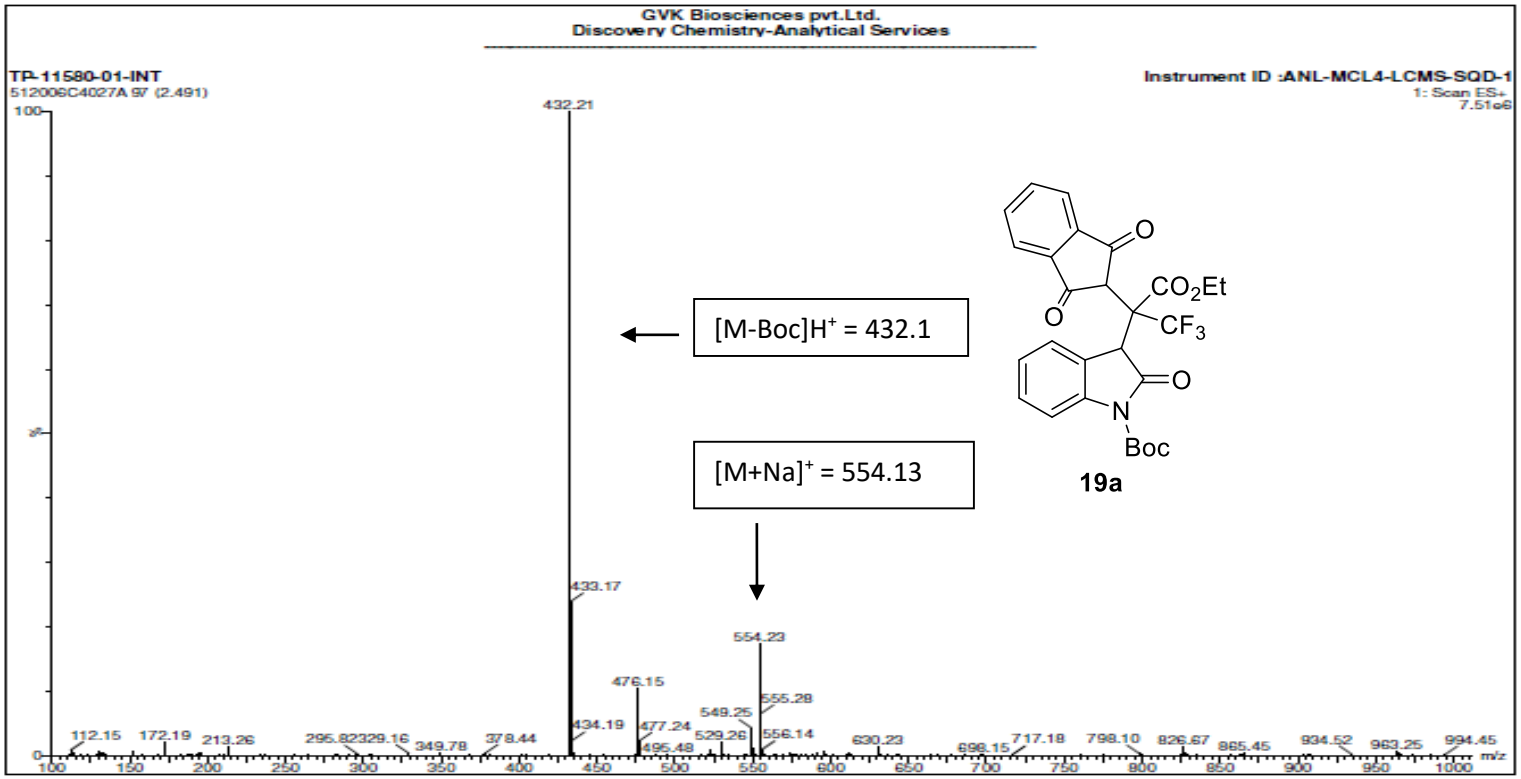


Scheme for pyrazolone derivative intermediate synthesis (4a).(ESI-MS calcd for $C_{29}H_{31}F_3N_3O_6$ [M+H]+, 574.21; found 574.25)





Scheme for indanone derivative intermediate synthesis (19a).(ESI-MS calcd for $C_{27}H_{24}F_3NNaO_7$ [M+Na]+, 554.13; found 554.23)



Single Crystal XRD Experiments:

The single crystal XRD data collection and data reduction were performed using CrysAlis PRO on a single crystal Rigaku Oxford XtaLab Pro diffractometer. The crystals were kept at 93(2) K during data collection using CuKα (λ = 1.54184 Å) radiation. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package using Least Squares minimisation.

Single Crystal structure, Cell parameters and structure data of the compound-3:

The crystals of the **compound-3** obtained as colourless plates from ethanol solution. The **compound-3** ($C_{28}H_{26}F_3N_3O_6$) crystallised in monoclinic $P2_1$ space group. The ORTEP crystal structure diagram of the compound is given in the **figure 1**.

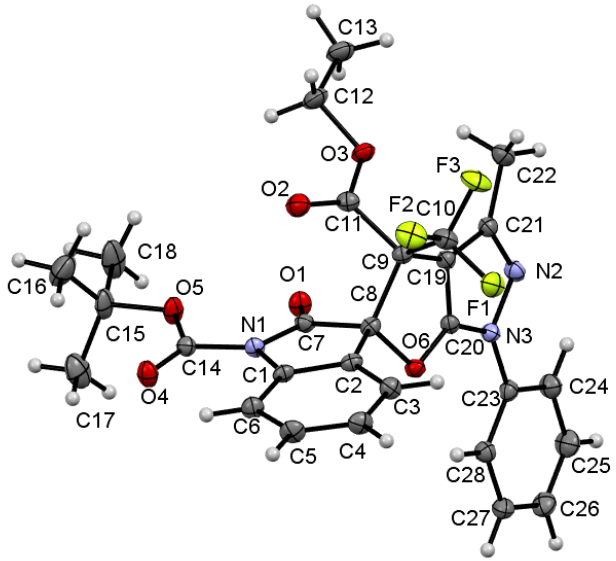


Figure 1: ORTEP diagram of **compound-3**. The thermal ellipsoids are drawn at 50 % probability level. **[CCDC 2011158]**

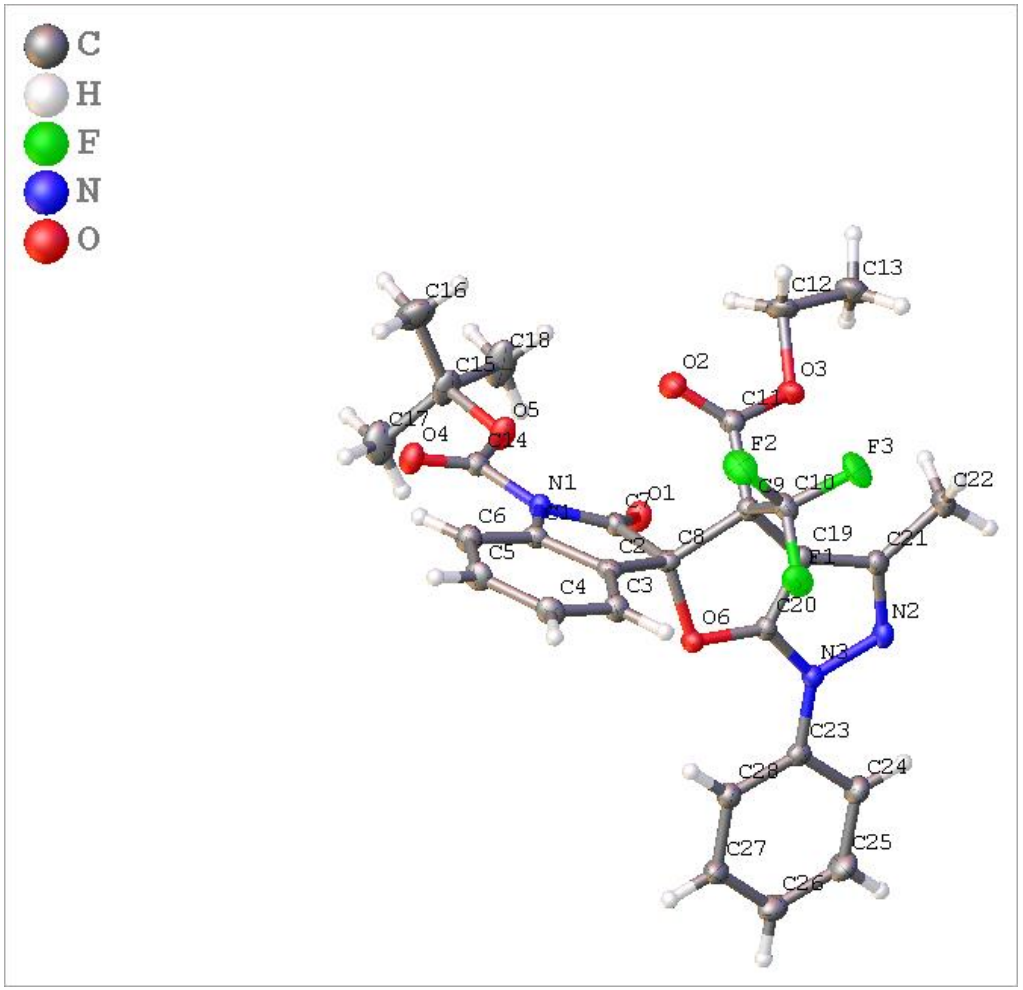

C
H
F
N
O
C16
C18
C15
C17
O4
O5
C14
N1
O1
C6
C5
C4
C3
C2
C8
C9
C10
F2
F3
C11
O2
O3
C12
C13
C22
C19
C21
F1
C20
O6
N2
N3
C23
C28
C24
C27
C25
C26

Table 1 Crystal data and structure refinement for compound-3.

Identification code	exp_586-11580-015 Repeat
Empirical formula	$C_{28}H_{26}F_3N_3O_6$
Formula weight	557.52
Temperature/K	93(2)
Crystal system	Monoclinic
Space group	$P2_1$
a/Å	8.85320(10)
b/Å	13.7373(2)
c/Å	11.5286(2)
α/°	90
β/°	107.761(2)
γ/°	90
Volume/Å^3	1335.27(4)
Z	2
ρ_{calc}g/cm^3	1.387
μ/mm^{-1}	0.954
F(000)	580.0
Crystal size/mm^3	0.15 × 0.1 × 0.03
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.052 to 159.162
Index ranges	$-8 \leq h \leq 10$, $-17 \leq k \leq 16$, $-14 \leq l \leq 13$
Reflections collected	8109
Independent reflections	4987 [R_{int} = 0.0277, R_{sigma} = 0.0419]
Data/restraints/parameters	4987/1/366
Goodness-of-fit on F^2	1.095
Final R indexes [I>=2σ (I)]	R_1 = 0.0330, wR_2 = 0.0885
Final R indexes [all data]	R_1 = 0.0341, wR_2 = 0.0893
Largest diff. peak/hole / e Å^{-3}	0.17/-0.22

Flack parameter -0.09(9)

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for compound-3. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
F1	5270.4(19)	6773.8(12)	7362.6(14)	27.0(3)
F3	6653(2)	7915.3(11)	6902.2(16)	29.9(4)
F2	4322.7(19)	7585.6(12)	5698.0(16)	30.3(4)
O6	6051.5(19)	4752.3(12)	6303.3(14)	16.7(3)
O4	2942(2)	4630.0(15)	1304.1(15)	23.7(4)
O1	7144(2)	4908.7(14)	4186.8(15)	21.6(4)
O3	8175(2)	7321.4(14)	4967.2(16)	21.9(4)
O5	5609(2)	4810.8(15)	1821.6(15)	24.1(4)
O2	5682(2)	7085.5(15)	3738.2(17)	26.5(4)
N3	8729(2)	4686.3(15)	7780.8(18)	16.7(4)
N1	4399(2)	5005.1(15)	3256.1(17)	16.4(4)
N2	9949(2)	5364.9(17)	8143.4(18)	19.9(4)
C2	3651(3)	5459.4(17)	4942(2)	16.4(4)
C20	7508(3)	5103.1(18)	6929(2)	15.6(4)
C23	8894(3)	3739.6(18)	8283(2)	17.9(5)
C11	6708(3)	6988.2(18)	4699(2)	19.3(5)
C8	5428(3)	5481.0(17)	5339(2)	15.7(4)
C28	7640(3)	3081.8(19)	7927(2)	20.2(5)
C14	4222(3)	4789.0(18)	2027(2)	16.9(4)
C19	7865(3)	6023.2(17)	6709(2)	16.4(5)
C7	5828(3)	5099.2(18)	4200(2)	16.7(4)
C21	9428(3)	6175.3(18)	7500(2)	18.6(5)

C1	3099(3)	5180.8(17)	3722(2)	16.1(4)
C10	5646(3)	7185.0(19)	6431(2)	21.9(5)
C4	991(3)	5475(2)	5028(2)	21.9(5)
C24	10324(3)	3469(2)	9139(2)	23.8(5)
C9	6434(3)	6454.7(18)	5792(2)	17.0(5)
C5	453(3)	5232(2)	3803(2)	22.5(5)
C6	1489(3)	5067.0(19)	3124(2)	21.1(5)
C3	2603(3)	5590.1(18)	5609(2)	20.0(5)
C22	10492(3)	7041(2)	7661(2)	25.1(5)
C27	7820(3)	2157.6(19)	8423(2)	23.0(5)
C15	5720(3)	4579(2)	592(2)	28.2(6)
C26	9239(3)	1875(2)	9264(2)	26.9(6)
C25	10473(4)	2537(2)	9618(3)	29.6(6)
C13	10286(4)	8119(2)	4473(3)	30.4(6)
C12	8554(3)	7870(2)	3999(3)	28.6(6)
C17	5207(4)	3535(3)	269(3)	38.4(7)
C16	4755(4)	5306(3)	-332(3)	41.3(8)
C18	7485(4)	4705(3)	788(3)	43.7(9)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound-3. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F1	30.3(8)	28.2(8)	28.4(8)	-6.2(7)	17.7(6)	-2.2(7)
F3	29.8(8)	18.4(7)	43.4(9)	-11.2(7)	14.1(7)	-4.3(6)
F2	23.5(8)	24.7(8)	42.6(10)	-0.8(7)	10.2(7)	8.0(6)
O6	14.1(7)	17.7(8)	17.5(7)	2.0(6)	3.7(6)	-1.6(6)
O4	16.4(8)	34.0(10)	19.4(8)	-3.5(8)	3.8(6)	-0.4(7)

O1	13.1(8)	30.8(10)	21.9(8)	-2.2(8)	7.0(6)	2.1(7)
O3	19.1(9)	25.0(9)	22.6(9)	5.1(7)	7.7(7)	-4.5(7)
O5	16.9(8)	38.8(11)	18.8(8)	-4.7(8)	9.0(6)	-1.6(8)
O2	23.7(9)	27.5(10)	24.7(9)	5.8(8)	2.1(7)	-2.8(8)
N3	16.4(9)	15.7(9)	17.0(9)	0.2(8)	3.7(7)	-1.6(7)
N1	12.4(9)	20.7(10)	17.0(9)	-2.2(8)	5.9(7)	-1.2(7)
N2	16.1(10)	23.7(10)	18.8(9)	-2.3(8)	3.7(8)	-3.6(8)
C2	14.9(11)	15.1(10)	20.1(11)	2.7(9)	6.5(9)	1.2(9)
C20	12.4(10)	19.1(11)	15.6(10)	-1.6(9)	4.9(8)	-1.3(8)
C23	20.3(12)	18.7(12)	15.5(10)	-0.6(9)	6.7(9)	0.9(9)
C11	17.6(11)	18.2(12)	22.2(11)	1.2(9)	6.3(9)	0.3(9)
C8	15.1(11)	15.7(11)	16.9(10)	2.6(9)	5.7(8)	2.8(9)
C28	19.6(12)	21.0(12)	19.5(11)	-0.2(10)	5.2(9)	-0.5(9)
C14	17.3(10)	16.4(11)	18.1(10)	-1.1(9)	7.0(8)	0.0(9)
C19	15.6(11)	17.7(11)	16.8(11)	-0.9(9)	6.3(9)	-1.1(9)
C7	15.2(11)	16.9(11)	17.9(11)	-1.1(9)	4.9(9)	-2.2(8)
C21	19.3(12)	19.0(11)	17.6(11)	-2.1(9)	5.9(9)	-2.7(9)
C1	14.1(11)	16.6(11)	19.8(11)	1.7(9)	8.5(9)	0.3(9)
C10	19.2(12)	18.5(12)	28.7(13)	-3.0(10)	8.4(10)	-2.2(9)
C4	18.6(12)	23.1(12)	28.1(12)	-2.5(10)	13.4(10)	0.4(10)
C24	19.5(12)	26.3(13)	22.6(12)	1.4(10)	1.8(10)	0.1(10)
C9	15.2(11)	17.5(11)	18.6(11)	0.4(9)	5.8(9)	-0.2(9)
C5	13.3(11)	28.1(13)	27.8(13)	-0.9(10)	8.9(9)	-0.7(9)
C6	17.0(11)	25.4(13)	21.9(11)	-1.2(10)	7.4(9)	-0.5(10)
C3	18.0(11)	21.3(12)	22.4(12)	-2.4(10)	8.6(9)	0.2(9)
C22	21.9(13)	26.4(13)	24.6(12)	-1.3(11)	3.6(10)	-7.3(10)
C27	28.3(13)	18.5(12)	20.8(11)	0.0(10)	5.2(10)	-2.5(10)
C15	25.3(13)	45.5(17)	17.4(12)	-7.9(12)	11.9(10)	-1.4(12)
C26	33.1(15)	22.4(13)	23.7(12)	3.2(10)	6.5(11)	3.6(11)

C25	26.3(14)	31.1(15)	26.0(13)	5.8(11)	0.1(11)	3.8(11)
C13	31.8(16)	29.5(14)	35.0(15)	4.9(12)	17.7(12)	-7.5(12)
C12	30.4(14)	31.0(14)	26.2(13)	9.0(12)	11.2(11)	-7.1(12)
C17	33.8(16)	48.7(19)	36.1(16)	-16.7(14)	15.5(14)	1.3(14)
C16	43.1(18)	56(2)	29.0(14)	10.2(15)	16.4(14)	-1.6(16)
C18	26.3(15)	81(3)	29.9(14)	-17.0(17)	17.3(12)	-7.7(17)

Table 4 Bond Lengths for compound-3.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
F1	C10	1.342(3)	C20	C19	1.346(3)
F3	C10	1.342(3)	C23	C28	1.393(4)
F2	C10	1.336(3)	C23	C24	1.397(4)
O6	C20	1.359(3)	C11	C9	1.540(3)
O6	C8	1.472(3)	C8	C7	1.552(3)
O4	C14	1.204(3)	C8	C9	1.603(3)
O1	C7	1.198(3)	C28	C27	1.382(4)
O3	C11	1.322(3)	C19	C21	1.422(3)
O3	C12	1.468(3)	C19	C9	1.502(3)
O5	C14	1.320(3)	C21	C22	1.493(4)
O5	C15	1.484(3)	C1	C6	1.390(3)
O2	C11	1.206(3)	C10	C9	1.532(3)
N3	N2	1.391(3)	C4	C5	1.387(4)
N3	C20	1.346(3)	C4	C3	1.388(4)
N3	C23	1.413(3)	C24	C25	1.385(4)
N1	C14	1.409(3)	C5	C6	1.393(3)

N1	C7	1.401(3)	C27	C26	1.388(4)
N1	C1	1.432(3)	C15	C17	1.517(5)
N2	C21	1.339(3)	C15	C16	1.519(5)
C2	C8	1.499(3)	C15	C18	1.519(4)
C2	C1	1.394(3)	C26	C25	1.384(4)
C2	C3	1.385(3)	C13	C12	1.502(4)

Table 5 Bond Angles for compound-3.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
C20	O6	C8	103.64(17)	O1	C7	C8	123.9(2)
C11	O3	C12	115.8(2)	N1	C7	C8	107.65(18)
C14	O5	C15	120.16(19)	N2	C21	C19	109.6(2)
N2	N3	C23	121.3(2)	N2	C21	C22	119.7(2)
C20	N3	N2	108.13(19)	C19	C21	C22	130.6(2)
C20	N3	C23	130.6(2)	C2	C1	N1	110.5(2)
C14	N1	C1	123.91(19)	C6	C1	N1	128.0(2)
C7	N1	C14	126.67(19)	C6	C1	C2	121.5(2)
C7	N1	C1	109.41(18)	F1	C10	F3	106.7(2)
C21	N2	N3	106.64(19)	F1	C10	C9	111.7(2)
C1	C2	C8	109.0(2)	F3	C10	C9	109.9(2)
C3	C2	C8	130.2(2)	F2	C10	F1	107.2(2)
C3	C2	C1	120.5(2)	F2	C10	F3	107.1(2)
N3	C20	O6	131.1(2)	F2	C10	C9	113.9(2)
C19	C20	O6	118.2(2)	C5	C4	C3	120.2(2)
C19	C20	N3	110.8(2)	C25	C24	C23	118.9(3)
C28	C23	N3	120.2(2)	C11	C9	C8	109.92(19)
C28	C23	C24	120.3(2)	C19	C9	C11	116.98(19)

C24	C23	N3	119.5(2)	C19	C9	C8	99.52(19)
O3	C11	C9	111.2(2)	C19	C9	C10	109.29(19)
O2	C11	O3	126.0(2)	C10	C9	C11	106.9(2)
O2	C11	C9	122.8(2)	C10	C9	C8	114.45(19)
O6	C8	C2	109.50(18)	C4	C5	C6	122.0(2)
O6	C8	C7	106.22(18)	C1	C6	C5	117.0(2)
O6	C8	C9	106.16(18)	C2	C3	C4	118.7(2)
C2	C8	C7	102.94(18)	C28	C27	C26	120.8(3)
C2	C8	C9	122.88(19)	O5	C15	C17	109.6(2)
C7	C8	C9	108.07(18)	O5	C15	C16	110.0(2)
C27	C28	C23	119.5(2)	O5	C15	C18	101.4(2)
O4	C14	O5	127.6(2)	C17	C15	C16	112.8(3)
O4	C14	N1	121.8(2)	C17	C15	C18	111.2(3)
O5	C14	N1	110.61(19)	C18	C15	C16	111.2(3)
C20	C19	C21	104.8(2)	C25	C26	C27	119.1(3)
C20	C19	C9	107.9(2)	C26	C25	C24	121.3(3)
C21	C19	C9	147.2(2)	O3	C12	C13	106.5(2)
O1	C7	N1	128.4(2)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound-3.

Atom	***x***	***y***	***z***	**U(eq)**
H28	6667.44	3267.28	7346.42	24
H4	255.21	5562.93	5470.86	26
H24	11179.52	3917.49	9387.64	29
H5	-656.38	5177.38	3413.72	27
H6	1111.32	4884.58	2290.69	25
H3	2981.03	5755.35	6448.89	24

H22A	11433.85	6937.95	8365.27	38
H22B	9925.04	7621.31	7796.67	38
H22C	10816.82	7131.17	6928.33	38
H27	6961.91	1710.44	8186.37	28
H26	9362.27	1234.97	9592.64	32
H25	11442.25	2348.2	10200.14	36
H13A	10561.94	8563.48	3904.38	46
H13B	10917.31	7522.52	4554.01	46
H13C	10509.67	8433.31	5271.05	46
H12A	7906.78	8470.19	3804.83	34
H12B	8336.63	7470.52	3251.25	34
H17A	5789.49	3103.65	932.23	58
H17B	5434.42	3351.61	-482.19	58
H17C	4066.59	3474.18	148.66	58
H16A	3623.63	5209.5	-437.91	62
H16B	4956.94	5207.95	-1113.57	62
H16C	5060.33	5969.14	-41.24	62
H18A	7802.5	5368.84	1070.91	66
H18B	7712.09	4590.61	18.89	66
H18C	8077.98	4237.84	1400.22	66

Crystal structure determination of compound-3

Crystal Data for $C_{28}H_{26}F_3N_3O_6$ (*M* =557.52 g/mol): monoclinic, space group $P2_1$ (no. 4), *a* = 8.85320(10) Å, *b* = 13.7373(2) Å, *c* = 11.5286(2) Å, *β* = 107.761(2)°, *V* = 1335.27(4) Å^3, *Z* = 2, *T* = 93(2) K, μ(CuKα) = 0.954 mm^{-1}, *Dcalc* = 1.387 g/cm^3, 8109 reflections measured (8.052° ≤ 2Θ ≤ 159.162°), 4987 unique (R_{int} = 0.0277, R_{sigma} = 0.0419) which were used in all calculations. The final R_1 was 0.0330 (I > 2σ(I)) and wR_2 was 0.0893 (all data).

Refinement model description

Number of restraints - 1, number of constraints - unknown.

Details:
1. Fixed Uiso
At 1.2 times of:
All C(H) groups, All C(H,H) groups
At 1.5 times of:
All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
C12(H12A,H12B)
2.b Aromatic/amide H refined with riding coordinates:
C28(H28), C4(H4), C24(H24), C5(H5), C6(H6), C3(H3), C27(H27), C26(H26),
C25(H25)
2.c Idealised Me refined as rotating group:
C22(H22A,H22B,H22C), C13(H13A,H13B,H13C), C17(H17A,H17B,H17C), C16(H16A,H16B,
H16C), C18(H18A,H18B,H18C)
This report has been created with Olex2, compiled on 2018.05.29 svn.r3508 for OlexSys.

Single Crystal XRD Experiments:

The single crystal XRD data collection and data reduction were performed using CrysAlis PRO on a single crystal Rigaku Oxford XtaLab Pro diffractometer. The crystals were kept at 93(2) K during data collection using CuKα (λ = 1.54184 Å) radiation. Using Olex2 [1], the structure was solved with the ShelXT [2] structure solution program using Intrinsic Phasing and refined with the ShelXL [3] refinement package using Least Squares minimisation.

Single Crystal structure, Cell parameters and structure data of compound 22:

The crystals of the **compound-22** obtained as colourless plates from ethanol solution. ($C_{27}H_{21}ClF_3NO_7$) crystallised in monoclinic $P2_1/n$ space group. The ORTEP crystal structure diagram of the compound is given in the **figure 2**.

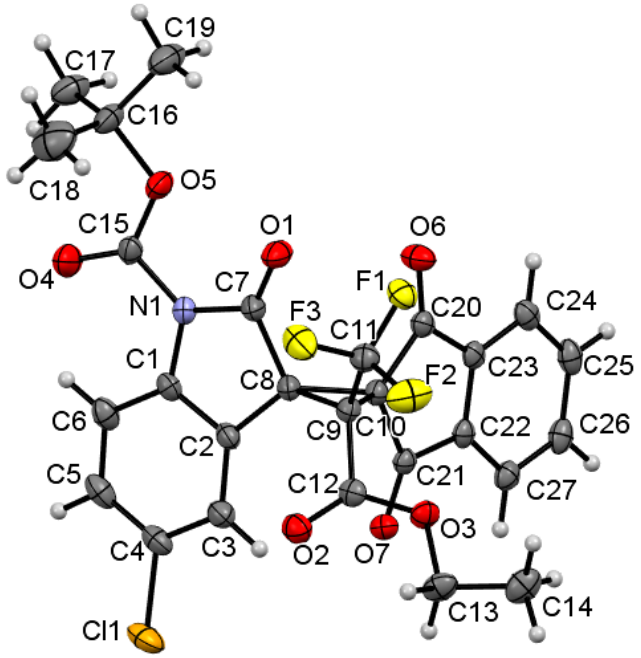
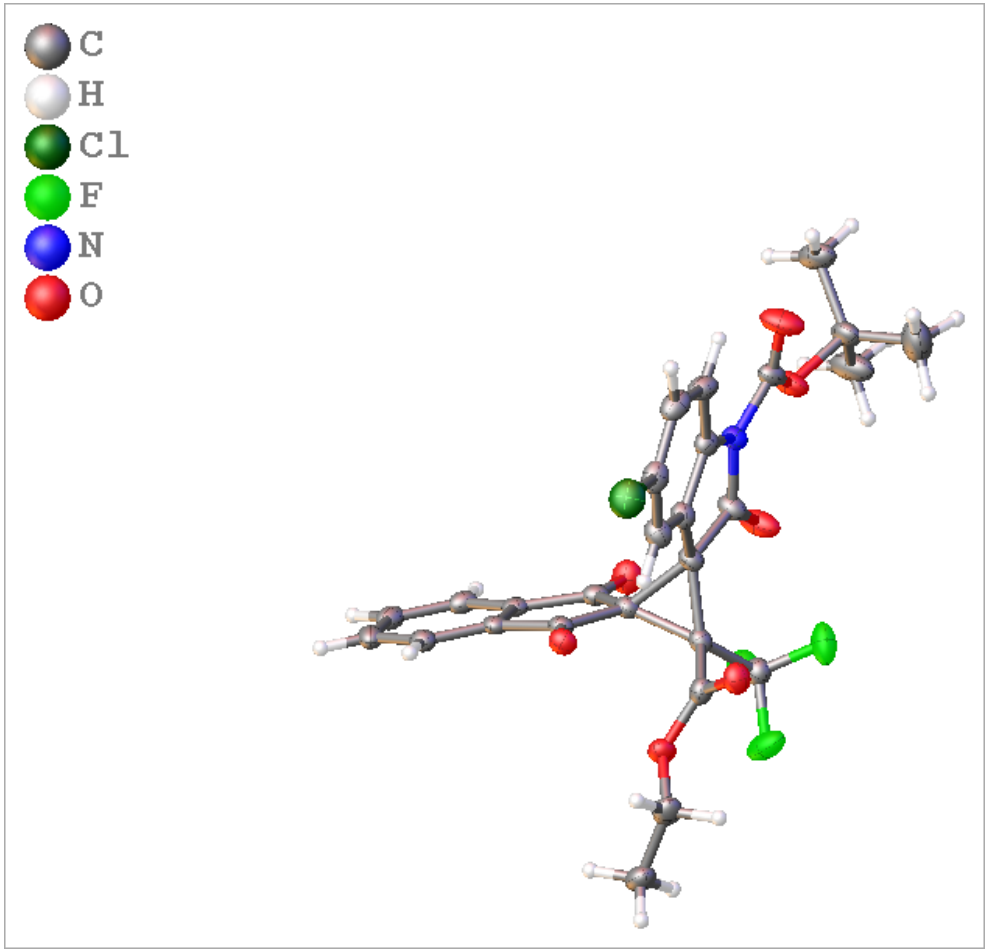


Figure 2: ORTEP diagram of compound **compound-22**. The thermal ellipsoids are drawn at 50 % probability level. **[CCDC 2011157]**


C
H
Cl
F
N
O

Cyclopropane ring Bond angles: C8-C9-C10, C9-C10-C8, C10-C8-C9 are 62.78(13), 58.11(13), 59.12(13)º respectively.

Cyclopropane bond lengths: The C8-C9, C9-C10 and C8-C10 bond lengths are 1.509(3), 1.526(3)and 1.581(3) Å respectively.

C=O bond lengths: C7=O1, C12=O2, C15=O4, C20=O6, C21=O7 are 1.197(3), 1.202(3), 1.196(3), 1.205(3), 1.210(3) Å respectively.

F-C-F bond angles: F1-C11-F2, F1-C11-F3 and F2-C11-F3 are 105.11(18), 110.14(18) and 104.52(19)º respectively.

C-F bond distance: C11-F1, C11-F2 and C11-F3 are 1.313(3), 1.363(3), and 1.331(3)Å respectively.

Table 1 Crystal data and structure refinement for compound-22.

Identification code	exp_576_THIRUPATHI
Empirical formula	$C_{27}H_{21}ClF_3NO_7$
Formula weight	563.90
Temperature/K	93(2)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	13.5978(2)
b/Å	14.6190(2)
c/Å	13.9308(2)
α/°	90
β/°	117.095(2)
γ/°	90
Volume/Å^3	2465.33(7)
Z	4
ρ_{calc}g/cm^3	1.519
μ/mm^{-1}	2.024
F(000)	1160.0
Crystal size/mm^3	0.1 × 0.05 × 0.05
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	7.532 to 159.27

Index ranges	$-16 \le h \le 9$, $-16 \le k \le 18$, $-17 \le l \le 17$
Reflections collected	16651
Independent reflections	5206 [R_{int} = 0.0374, R_{sigma} = 0.0354]
Data/restraints/parameters	5206/0/356
Goodness-of-fit on F^2	1.101
Final R indexes [I>=2σ (I)]	R_1 = 0.0542, wR_2 = 0.1559
Final R indexes [all data]	R_1 = 0.0584, wR_2 = 0.1594
Largest diff. peak/hole / e $Å^{-3}$	0.68/-0.53

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters ($Å^2$×10^3) for compound-22. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl1	5628.3(6)	8106.2(4)	6969.1(5)	41.18(19)
F1	6690.5(11)	6023.8(9)	1899.6(10)	30.0(3)
F3	5430.1(12)	7040.4(11)	1630.3(11)	41.1(4)
O7	8106.5(12)	6944.9(10)	5829.1(12)	25.2(3)
F2	7091.5(15)	7438.8(11)	2086.8(12)	44.6(4)
O3	8145.4(12)	7863.3(10)	4118.7(13)	26.2(3)
O2	6479.1(13)	8340.1(11)	3910.9(13)	30.2(4)
O6	7154.0(14)	4569.1(11)	3219.2(13)	32.7(4)
O5	3321.3(13)	4496.0(12)	2067.3(14)	33.3(4)
O4	2624.6(15)	5001.9(14)	3160.9(17)	44.4(5)
O1	5048.7(14)	5352.4(14)	2185.1(14)	39.5(4)
N1	4325.7(14)	5479.4(12)	3406.0(14)	22.2(4)
C22	8877.3(16)	5478.3(14)	5783.1(16)	21.4(4)
C23	8596.2(16)	4795.4(14)	5007.4(16)	21.3(4)
C21	8078.8(16)	6236.8(14)	5367.4(16)	20.7(4)
C20	7592.5(16)	5045.3(14)	4008.4(17)	22.5(4)

C1	4570.2(17)	6043.2(14)	4316.0(16)	22.3(4)		
C10	7232.4(16)	5990.3(14)	4219.4(16)	21.3(4)		
C12	7092.6(17)	7760.9(15)	3882.1(16)	24.0(4)		
C15	3330.2(17)	4971.7(14)	2874.4(18)	24.6(4)		
C24	9229.1(18)	4003.1(15)	5203.8(18)	25.5(4)		
C2	5523.8(17)	6561.7(15)	4568.6(16)	23.2(4)		
C9	6709.9(16)	6790.3(14)	3450.0(16)	21.9(4)		
C8	5984.1(16)	6271.4(14)	3819.8(16)	22.3(4)		
C27	9809.2(17)	5407.9(15)	6778.2(17)	24.9(4)		
C7	5104.2(17)	5634.9(16)	3014.7(17)	25.7(4)		
C3	5858.1(18)	7205.4(16)	5393.5(18)	28.2(5)		
C6	3975.5(19)	6122.1(16)	4902.4(19)	28.4(5)		
C25	10158.1(18)	3929.5(16)	6197.2(19)	28.7(5)		
C26	10449.0(17)	4623.2(17)	6970.7(18)	28.1(5)		
C11	6469.8(18)	6788.7(16)	2257.3(18)	27.1(4)		
C4	5251.0(19)	7281.3(16)	5964.7(18)	28.9(5)		
C5	4338(2)	6740.8(16)	5747.4(19)	31.5(5)		
C13	8593(2)	8791.8(16)	4353(2)	31.9(5)		
C16	2271.3(18)	4100.6(17)	1226.0(19)	31.9(5)		
C14	9628(2)	8775.9(18)	4225(2)	36.7(5)		
C17	1828(2)	3382(2)	1707(2)	40.8(6)		
C19	2661(2)	3654(2)	464(2)	48.3(7)		
C18	1483(3)	4861(2)	679(3)	57.5(8)		

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for compound-22. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**

Cl1	53.3(4)	39.9(3)	35.5(3)	-12.1(2)	24.8(3)	5.3(3)
F1	33.7(7)	33.2(7)	27.1(6)	-2.5(5)	17.4(5)	1.5(5)
F3	37.0(8)	52.6(9)	24.3(7)	-1.0(6)	5.7(6)	15.4(7)
O7	24.9(7)	24.1(8)	26.2(7)	-4.8(6)	11.4(6)	-2.9(6)
F2	63.6(10)	41.6(9)	31.5(7)	-0.2(6)	24.1(7)	-18.3(7)
O3	26.3(7)	20.6(7)	31.4(8)	0.7(6)	12.9(6)	-2.7(6)
O2	29.6(8)	27.9(8)	30.7(8)	-0.7(6)	11.6(6)	4.3(6)
O6	38.6(9)	28.4(8)	27.5(8)	-6.8(6)	11.9(7)	0.3(7)
O5	25.0(8)	41.8(10)	35.2(8)	-12.5(7)	15.5(7)	-12.6(7)
O4	37.6(10)	52.3(12)	57.4(12)	-19.9(9)	33.8(9)	-16.2(8)
O1	33.8(9)	54.9(11)	37.6(9)	-23.0(8)	23.0(8)	-19.0(8)
N1	18.4(8)	24.4(9)	25.3(8)	-1.2(7)	11.2(7)	0.2(6)
C22	19.1(9)	24.1(10)	25.2(10)	1.9(8)	13.7(8)	-2.6(7)
C23	21.5(9)	22.4(10)	25.6(9)	1.2(8)	15.7(8)	-2.6(7)
C21	19.0(9)	23.0(10)	22.5(9)	-1.3(7)	11.6(7)	-3.8(7)
C20	22.8(9)	23.0(10)	26.2(10)	-1.4(8)	15.0(8)	-3.3(7)
C1	23.6(9)	21.2(10)	24.3(9)	1.7(7)	12.8(8)	4.9(7)
C10	18.9(9)	23.5(10)	22.6(9)	-1.5(7)	10.4(7)	-1.8(7)
C12	25.1(10)	23.4(10)	21.1(9)	1.9(8)	8.5(8)	2.4(8)
C15	21.0(9)	22.0(10)	33.2(11)	0.5(8)	14.5(8)	-0.6(7)
C24	29.5(10)	22.6(10)	32.2(11)	2.6(8)	20.8(9)	0.2(8)
C2	22.1(9)	26.2(10)	22.5(9)	-1.6(8)	11.3(8)	4.2(8)
C9	18.7(9)	24.3(10)	21.7(9)	-1.4(8)	8.4(7)	0.8(7)
C8	18.3(9)	26.0(10)	23.6(9)	-3.5(8)	10.4(7)	-0.2(7)
C27	22.7(10)	29.2(11)	25.0(10)	1.2(8)	12.9(8)	-3.1(8)
C7	20.8(9)	31.8(11)	26.8(10)	-7.1(8)	12.9(8)	-2.5(8)
C3	27.1(10)	29.8(11)	28.1(10)	-4.1(9)	12.8(9)	2.5(8)
C6	33.2(11)	26.7(11)	34.3(11)	3.8(9)	23.0(10)	3.0(8)
C25	27.6(10)	29.9(11)	36.2(11)	8.6(9)	21.2(9)	4.9(8)

C26	21.6(10)	37.7(12)	27.5(10)	8.1(9)	13.1(8)	0.9(8)
C11	27.1(10)	29.1(11)	24.6(10)	-0.5(8)	11.2(8)	-0.2(8)
C4	35.2(11)	28.5(11)	25.2(10)	-0.7(8)	15.7(9)	9.8(9)
C5	41.1(13)	30.9(12)	32.5(11)	4.7(9)	25.4(10)	10.5(9)
C13	35.0(12)	22.1(10)	34.3(11)	0.7(9)	12.0(9)	-5.0(9)
C16	24.2(10)	39.0(13)	29.1(11)	-3.3(9)	9.3(9)	-11.2(9)
C14	36.9(13)	35.7(13)	35.6(12)	7.0(10)	14.8(10)	-8.1(10)
C17	44.1(14)	44.9(15)	35.4(13)	-8.8(11)	19.8(11)	-20.3(11)
C19	44.2(15)	64.4(19)	42.4(14)	-22.1(13)	25.1(12)	-26.9(14)
C18	46.6(16)	56.5(19)	47.7(17)	3.3(14)	2.6(13)	2.7(14)

Table 4 Bond Lengths for compound-22.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	C4	1.738(2)	C20	C10	1.538(3)
F1	C11	1.313(3)	C1	C2	1.402(3)
F3	C11	1.331(3)	C1	C6	1.391(3)
O7	C21	1.210(3)	C10	C9	1.526(3)
F2	C11	1.363(3)	C10	C8	1.581(3)
O3	C12	1.323(3)	C12	C9	1.537(3)
O3	C13	1.462(3)	C24	C25	1.389(3)
O2	C12	1.202(3)	C2	C8	1.502(3)
O6	C20	1.205(3)	C2	C3	1.392(3)
O5	C15	1.317(3)	C9	C8	1.509(3)
O5	C16	1.491(3)	C9	C11	1.541(3)
O4	C15	1.196(3)	C8	C7	1.527(3)
O1	C7	1.197(3)	C27	C26	1.390(3)
N1	C1	1.419(3)	C3	C4	1.387(3)

N1	C15	1.421(3)	C6	C5	1.385(3)
N1	C7	1.412(3)	C25	C26	1.399(3)
C22	C23	1.391(3)	C4	C5	1.384(4)
C22	C21	1.473(3)	C13	C14	1.497(4)
C22	C27	1.392(3)	C16	C17	1.511(3)
C23	C20	1.484(3)	C16	C19	1.531(4)
C23	C24	1.394(3)	C16	C18	1.490(4)
C21	C10	1.528(3)			

Table 5 Bond Angles for compound-22.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C12	O3	C13	116.98(17)	C10	C9	C11	123.96(18)
C15	O5	C16	120.60(17)	C12	C9	C11	106.95(17)
C1	N1	C15	123.31(17)	C8	C9	C10	62.78(13)
C7	N1	C1	109.85(16)	C8	C9	C12	119.20(17)
C7	N1	C15	125.90(18)	C8	C9	C11	121.14(17)
C23	C22	C21	110.01(17)	C2	C8	C10	123.32(17)
C23	C22	C27	121.6(2)	C2	C8	C9	129.74(19)
C27	C22	C21	128.37(19)	C2	C8	C7	104.32(16)
C22	C23	C20	111.21(18)	C9	C8	C10	59.12(13)
C22	C23	C24	121.16(19)	C9	C8	C7	118.09(17)
C24	C23	C20	127.62(19)	C7	C8	C10	117.17(17)
O7	C21	C22	126.57(19)	C26	C27	C22	117.3(2)
O7	C21	C10	125.74(19)	O1	C7	N1	126.0(2)
C22	C21	C10	107.63(17)	O1	C7	C8	127.09(19)
O6	C20	C23	125.1(2)	N1	C7	C8	106.79(16)
O6	C20	C10	128.68(19)	C4	C3	C2	118.2(2)

C23	C20	C10	106.18(17)	C5	C6	C1	118.5(2)
C2	C1	N1	110.61(17)	C24	C25	C26	121.4(2)
C6	C1	N1	127.6(2)	C27	C26	C25	121.1(2)
C6	C1	C2	121.8(2)	F1	C11	F3	110.14(18)
C21	C10	C20	104.96(16)	F1	C11	F2	105.11(18)
C21	C10	C8	118.90(16)	F1	C11	C9	115.96(18)
C20	C10	C8	123.39(17)	F3	C11	F2	104.52(19)
C9	C10	C21	116.25(17)	F3	C11	C9	110.32(18)
C9	C10	C20	129.88(17)	F2	C11	C9	110.08(17)
C9	C10	C8	58.11(13)	C3	C4	Cl1	118.86(19)
O3	C12	C9	109.69(17)	C5	C4	Cl1	118.59(17)
O2	C12	O3	126.9(2)	C5	C4	C3	122.5(2)
O2	C12	C9	123.17(19)	C4	C5	C6	119.7(2)
O5	C15	N1	110.92(17)	O3	C13	C14	106.09(19)
O4	C15	O5	127.4(2)	O5	C16	C17	110.70(19)
O4	C15	N1	121.7(2)	O5	C16	C19	101.55(18)
C25	C24	C23	117.4(2)	C17	C16	C19	110.2(2)
C1	C2	C8	107.67(18)	C18	C16	O5	108.7(2)
C3	C2	C1	119.33(19)	C18	C16	C17	113.9(2)
C3	C2	C8	133.0(2)	C18	C16	C19	111.0(3)
C10	C9	C12	117.80(16)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for compound-22.

Atom	*x*	*y*	*z*	U(eq)
H24	9032.93	3531.97	4678.95	31
H27	10000.27	5877.24	7304.59	30
H3	6484.45	7582.3	5560.92	34

H6	3336.03	5760.12	4727.25	34
H25	10605.31	3397	6354.61	34
H26	11094.86	4556.81	7638.94	34
H5	3961.27	6793.84	6175.81	38
H13A	8759.15	8969.36	5097.46	38
H13B	8057.22	9233.99	3844.92	38
H14A	9445.03	8622.95	3477.68	55
H14B	10134.64	8315.34	4709.67	55
H14C	9980.29	9378.86	4404.2	55
H17A	2406.21	2930.29	2096.64	61
H17B	1192.19	3077.25	1129.15	61
H17C	1598.92	3673.28	2206.38	61
H19A	2991.63	4120.22	194.88	72
H19B	2029.35	3374.41	-143.92	72
H19C	3211.76	3182.96	854.62	72
H18A	1197.3	5099.51	1160.78	86
H18B	869.09	4632.09	16	86
H18C	1865.43	5350.4	502.17	86

Crystal structure determination of compound-22

Crystal Data for $C_{27}H_{21}ClF_3NO_7$ (M =563.90 g/mol): monoclinic, space group $P2_1/n$ (no. 14), a = 13.5978(2) Å, b = 14.6190(2) Å, c = 13.9308(2) Å, β = 117.095(2)°, V = 2465.33(7) Å^3, Z = 4, T = 93(2) K, μ(CuKα) = 2.024 mm^{-1}, *Dcalc* = 1.519 g/cm^3, 16651 reflections measured (7.532° ≤ 2Θ ≤ 159.27°), 5206 unique (R_{int} = 0.0374, R_{sigma} = 0.0354) which were used in all calculations. The final R_1 was 0.0542 (I > 2σ(I)) and wR_2 was 0.1594 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.
Details:
1. Fixed Uiso
At 1.2 times of:

All C(H) groups, All C(H,H) groups
At 1.5 times of:
All C(H,H,H) groups
2.a Secondary CH2 refined with riding coordinates:
C13(H13A,H13B)
2.b Aromatic/amide H refined with riding coordinates:
C24(H24), C27(H27), C3(H3), C6(H6), C25(H25), C26(H26), C5(H5)
2.c Idealised Me refined as rotating group:
C14(H14A,H14B,H14C), C17(H17A,H17B,H17C), C19(H19A,H19B,H19C), C18(H18A,H18B, H18C)

This report has been created with Olex2, compiled on 2018.05.29 svn.r3508 for OlexSys.

References

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8.