

Supplementary Material

Synthesis and structure-activity relationships for the anti-mycobacterial activity of 3-phenyl-*N*-(pyridin-2-ylmethyl)pyrazolo[1,5-*a*]pyrimidin-7-amines

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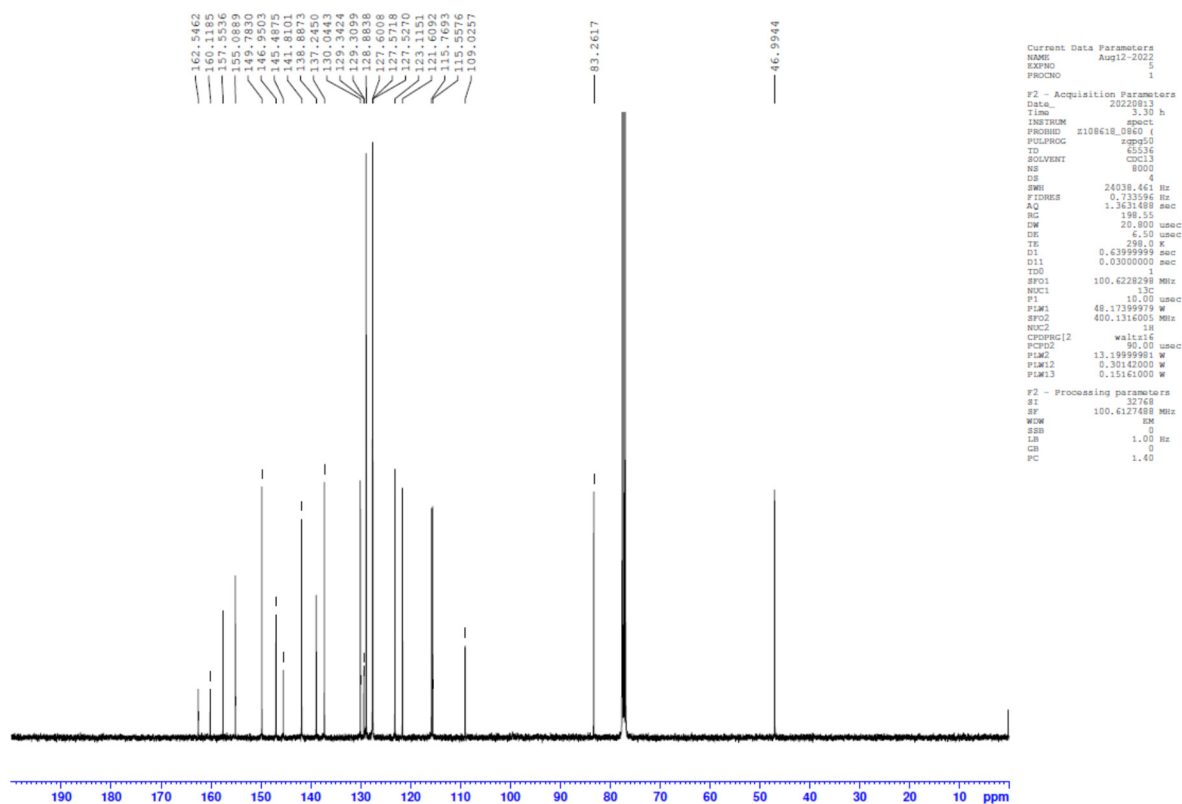
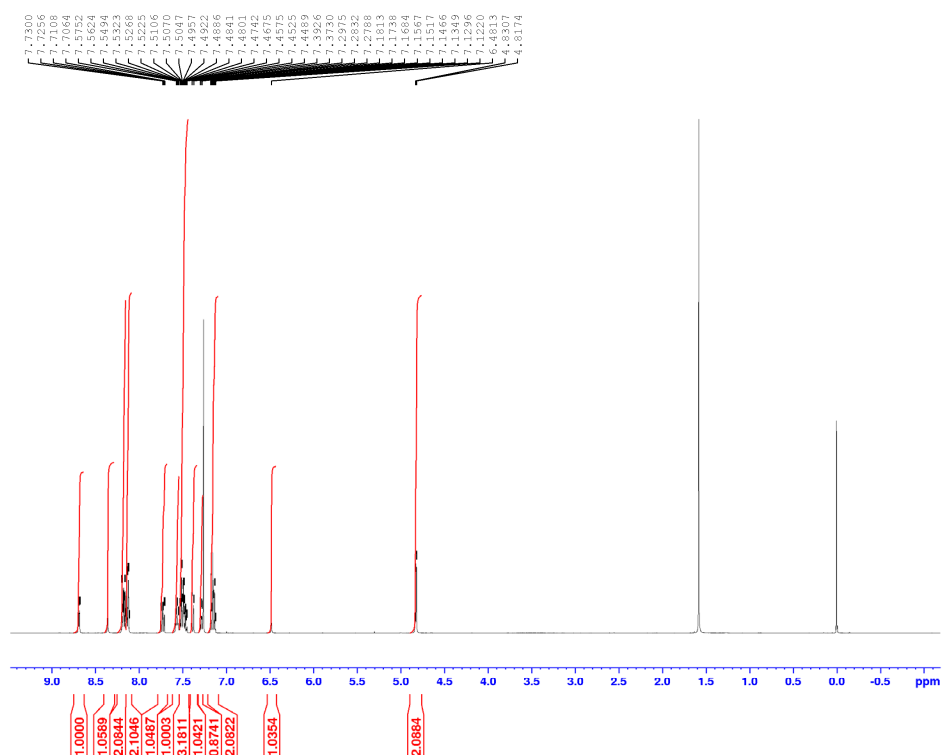
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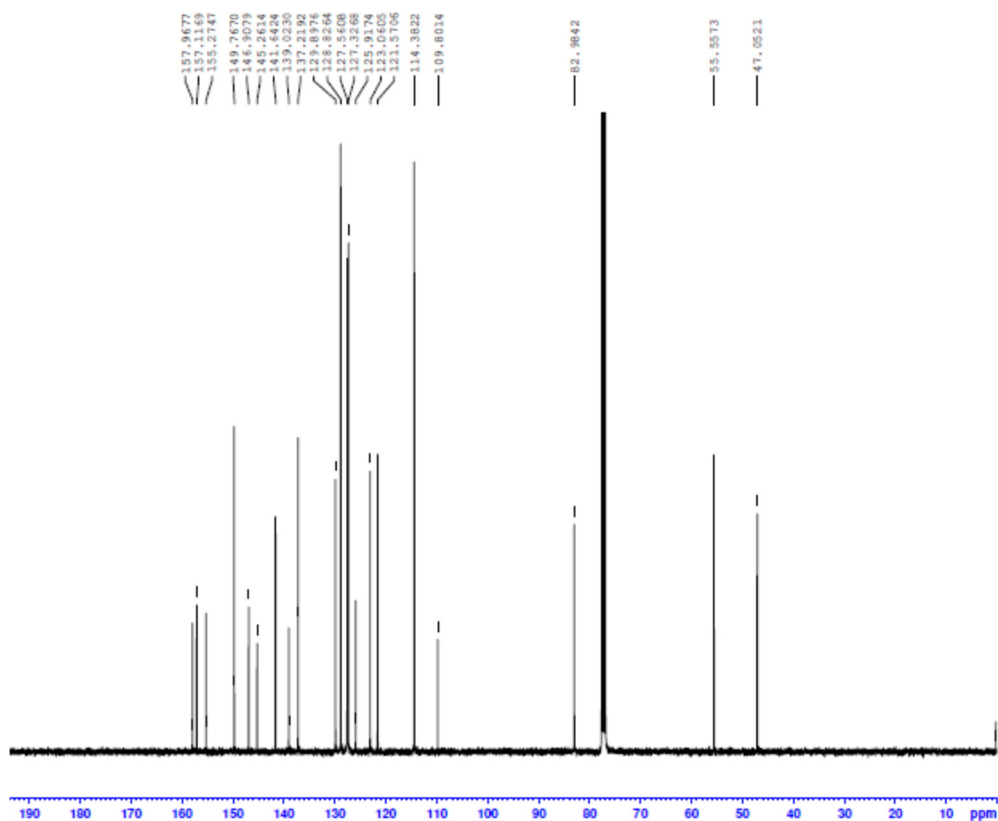
General information

Final products were analysed by reverse-phase HPLC (Alltima C18 5 μ m column, 15 \times 3.2 mm; Alltech Associated, Inc., Deerfield, IL) using an Agilent HP1100 equipped with a diode-array detector. It was run using mobile phases with 80% CH₃CN/20% H₂O (v/v) in 45 mM NH₄HCO₂ at pH 3.5 and 0.5 mL/min. Purity level was determined by monitoring at 330 \pm 50 nm and was \geq 95% for all final products. NMR spectra were obtained on a Bruker Avance 400 spectrometer at 400 MHz for ¹H and ¹³C. Low-resolution mass spectra (LRMS), using atmospheric pressure chemical ionization (APCI) were measured on a ThermoFinnigan Surveyor MSQ mass spectrometer, connected to a Gilson autosampler. High resolution mass spectra (HRMS) were obtained using an Agilent G6530B Q-TOF spectrometer and are reported as M+H. Melting points were determined on an Electrothermal 9100 melting point apparatus.

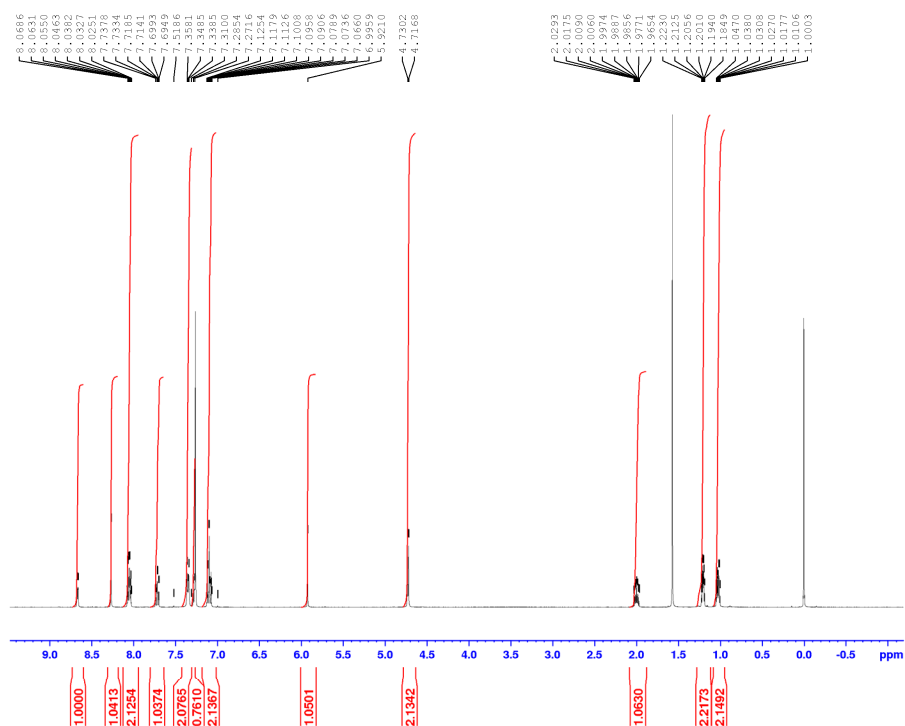
¹H NMR and ¹³C NMR Spectra for compounds that progressed to advanced testing

Compound 18



[illegible]

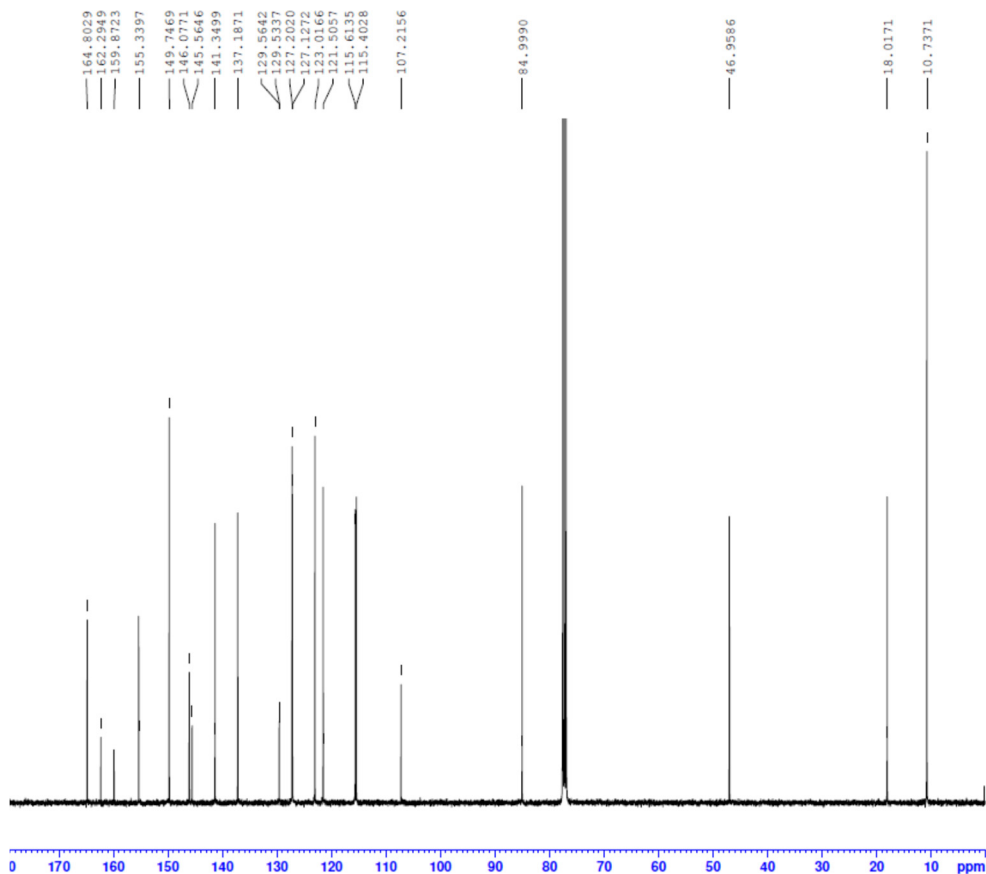
Compound 25



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 TE 298.0 K
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F2 - Processing parameters
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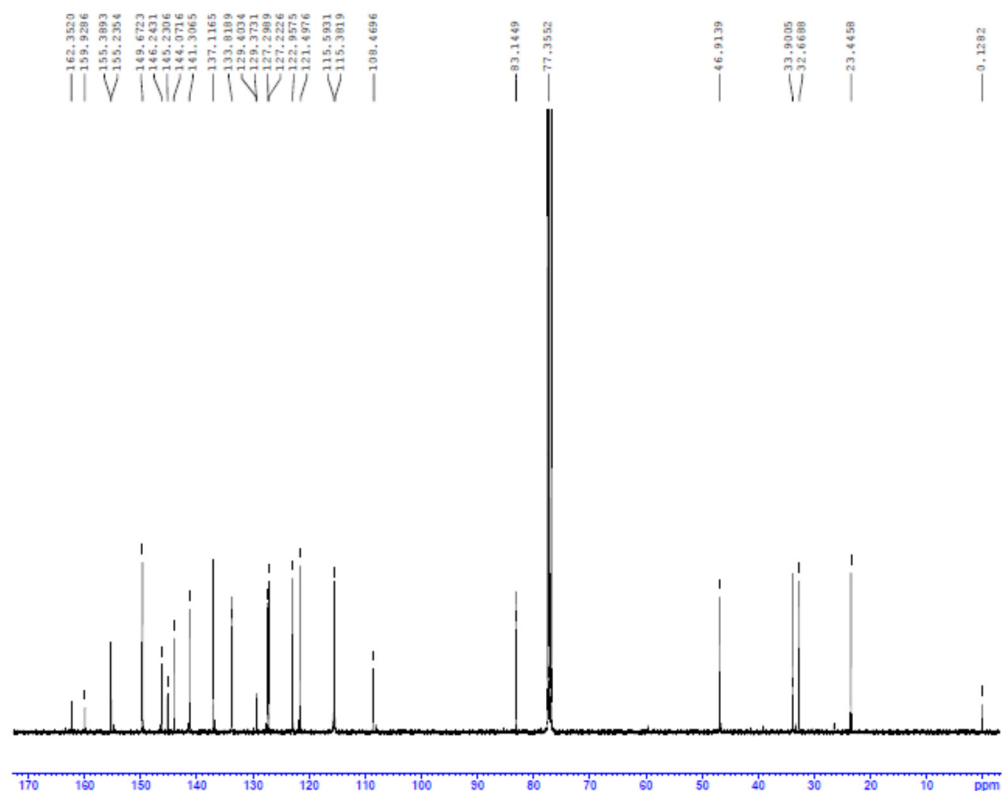
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 NUC2 1H
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 PCPD2 90.00 usec
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 PLW12 0.30142000 W
 PLW13 0.15161000 W

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¹H NMR spectrum (CDCl₃) of compound 10. The x-axis represents the chemical shift in ppm, ranging from 0.0 to 9.0. The spectrum shows several sharp singlet peaks. Integration values are indicated below the baseline. The chemical shifts (δ) are listed on the right side of the spectrum.

Chemical shifts (δ): 8.3079, 8.1459, 8.1404, 8.1383, 8.1322, 8.1252, 8.1155, 8.1099, 7.7831, 7.7199, 7.7155, 7.7007, 7.6922, 7.4022, 7.3891, 7.3692, 7.3650, 7.3583, 7.3528, 7.2728, 7.1346, 7.1121, 7.1073, 7.0956, 6.9762, 6.9704, 6.6640, 6.6591, 6.6542, 6.1707, 4.7548, 4.7416, 2.9289, 2.9233, 2.9171, 2.9117, 2.9042, 2.8996, 2.8953, 2.8900, 2.8844, 2.6411, 2.6349, 2.6288, 2.6238, 2.6167, 2.6108, 2.5994, 2.5943, 2.5513, 2.5811, 2.1042, 2.0980, 2.0933, 2.0833, 2.0462, 2.0278, 1.9586, 0.8818, 0.8643.

Integration values (from left to right): 1.0000, 1.0362, 2.1165, 1.0383, 2.0669, 1.0173, 2.1319, 1.0377, 1.0471, 2.1382, 2.1247, 2.1486, 2.1276, 0.2289, 0.1083.



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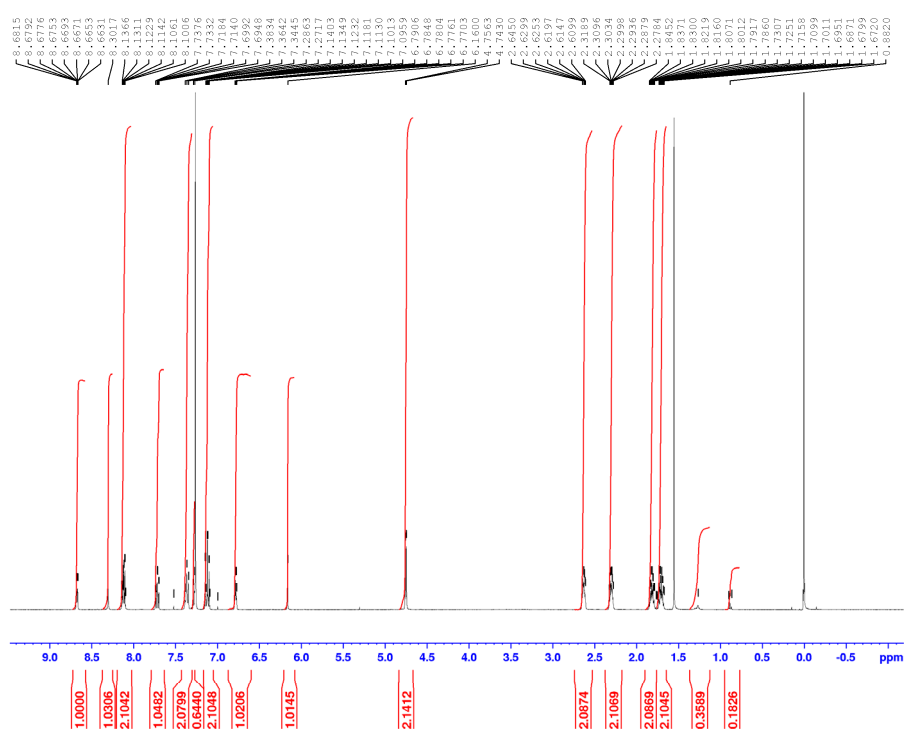
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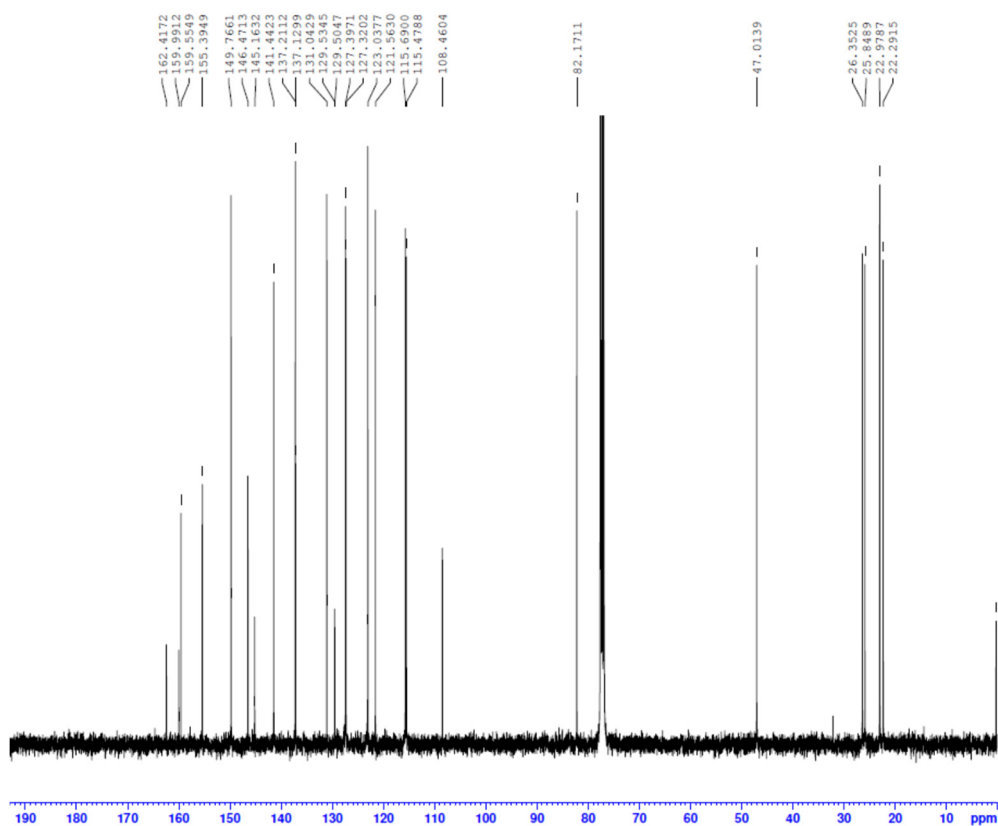
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Compound 27

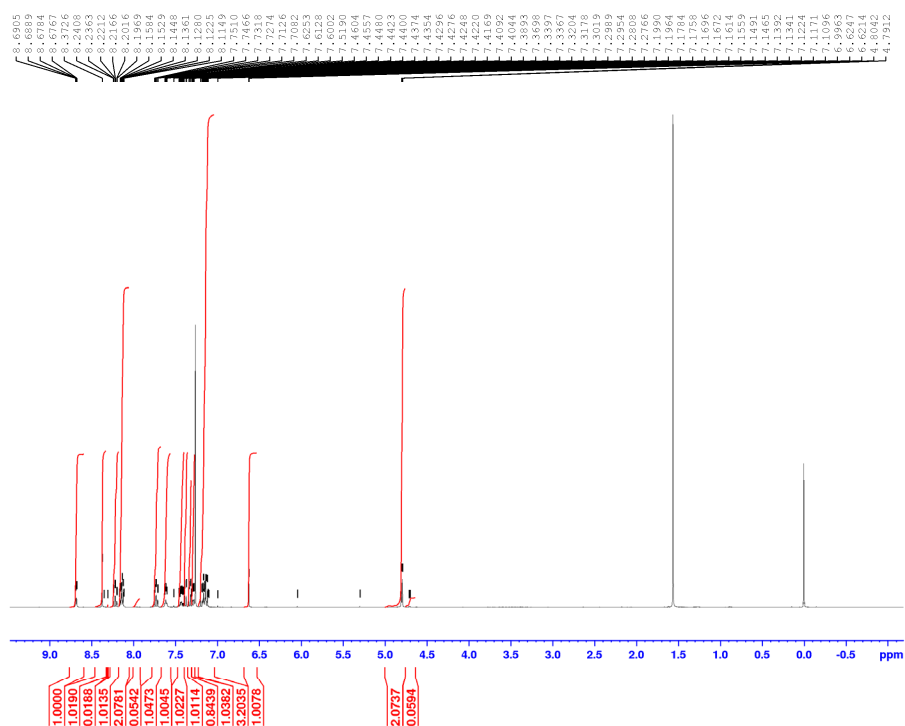


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 FIDRES: 0.244532 Hz
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 P1: 13.60 usec
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 PULPROG: zgpg50
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 SOLVENT: CDCl3
 NS: 16000
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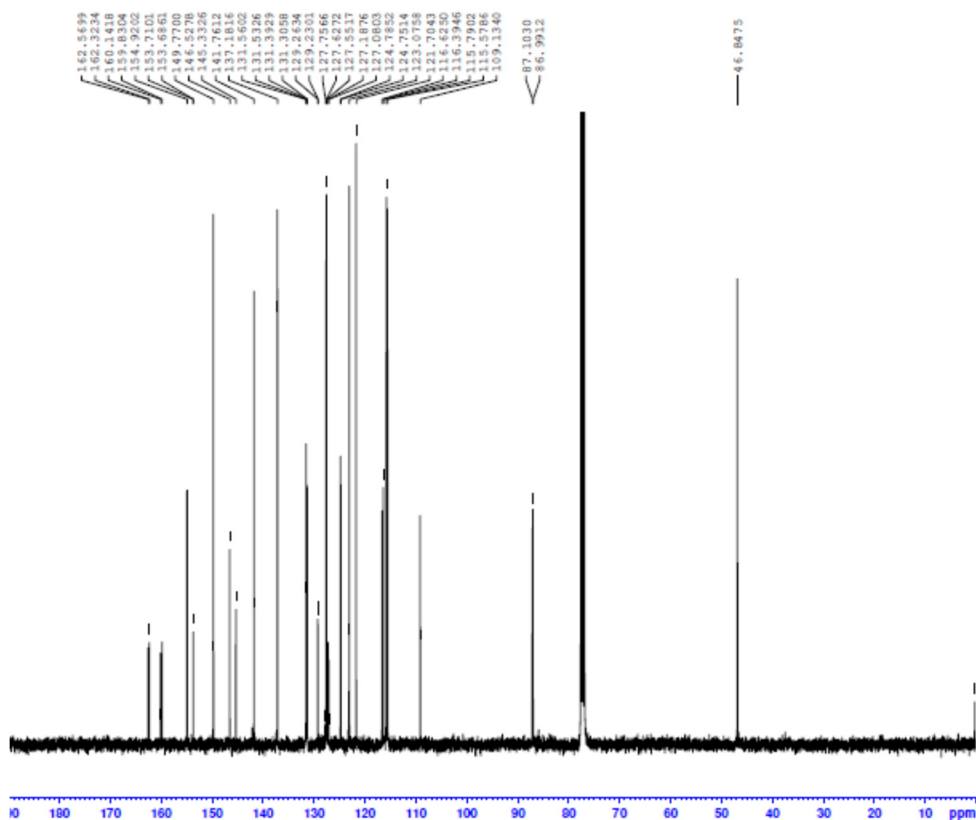
Compound 28



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SOLVENT CDCl3
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RG 198.50
DW 62.400 usec
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NUC1 1H
P1 13.60 usec
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DB 8
PC 1.00

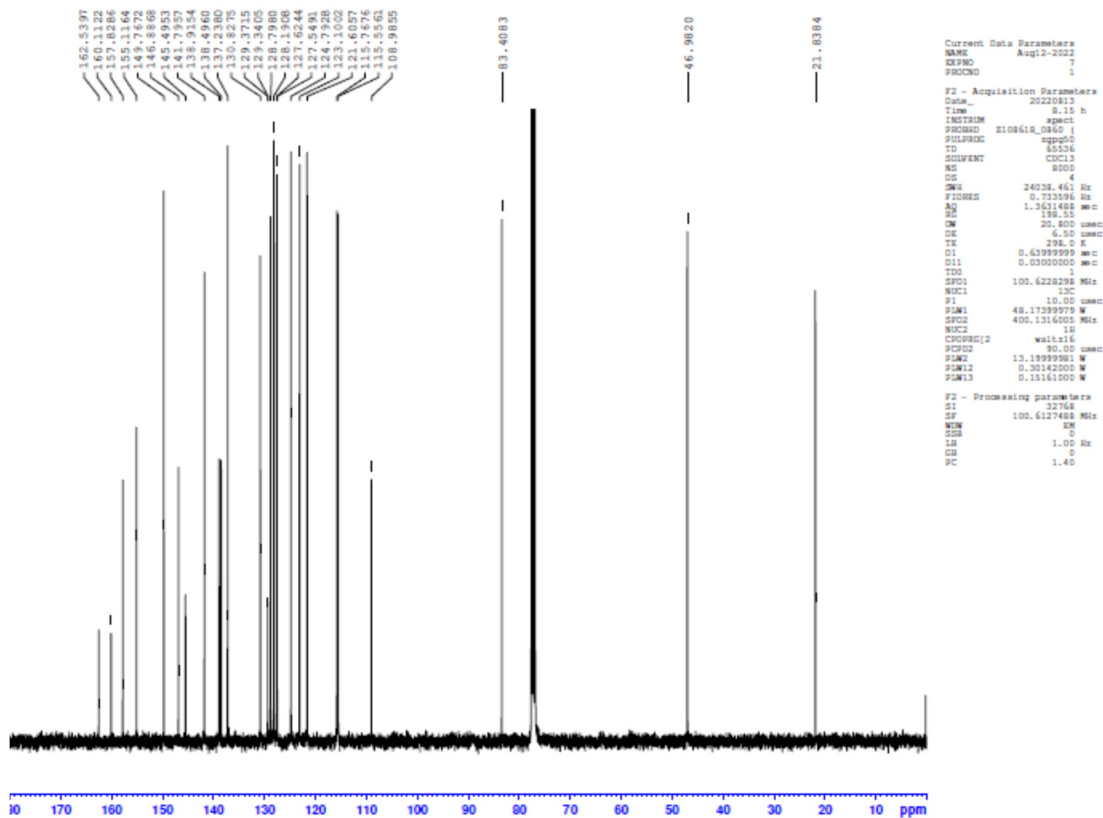
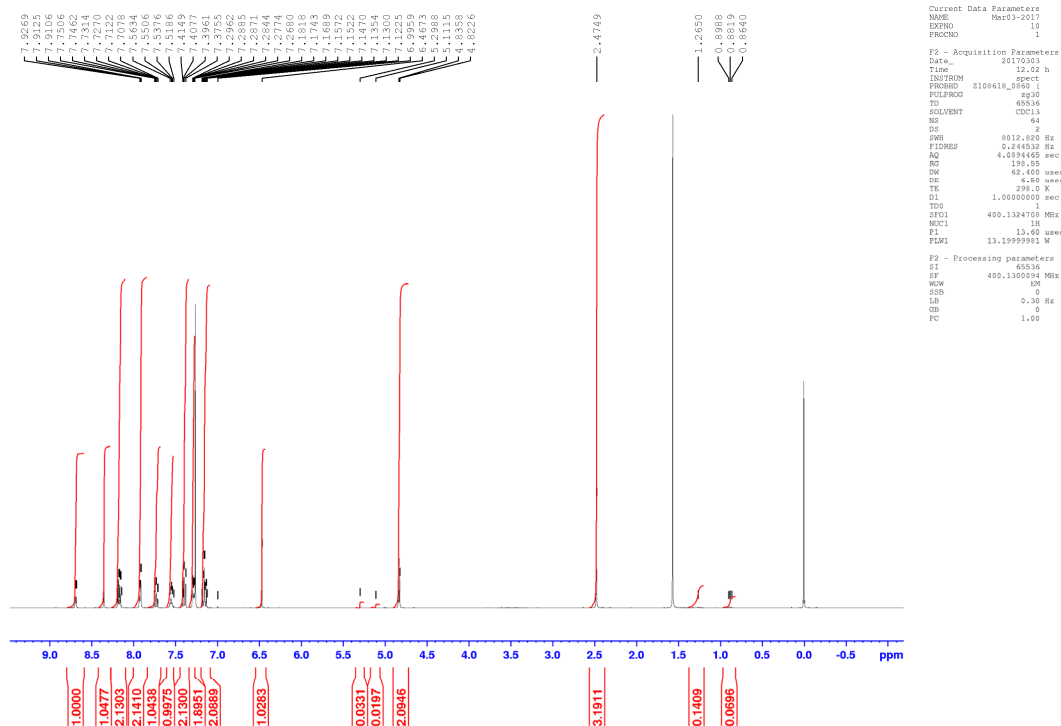


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PROCNO 1

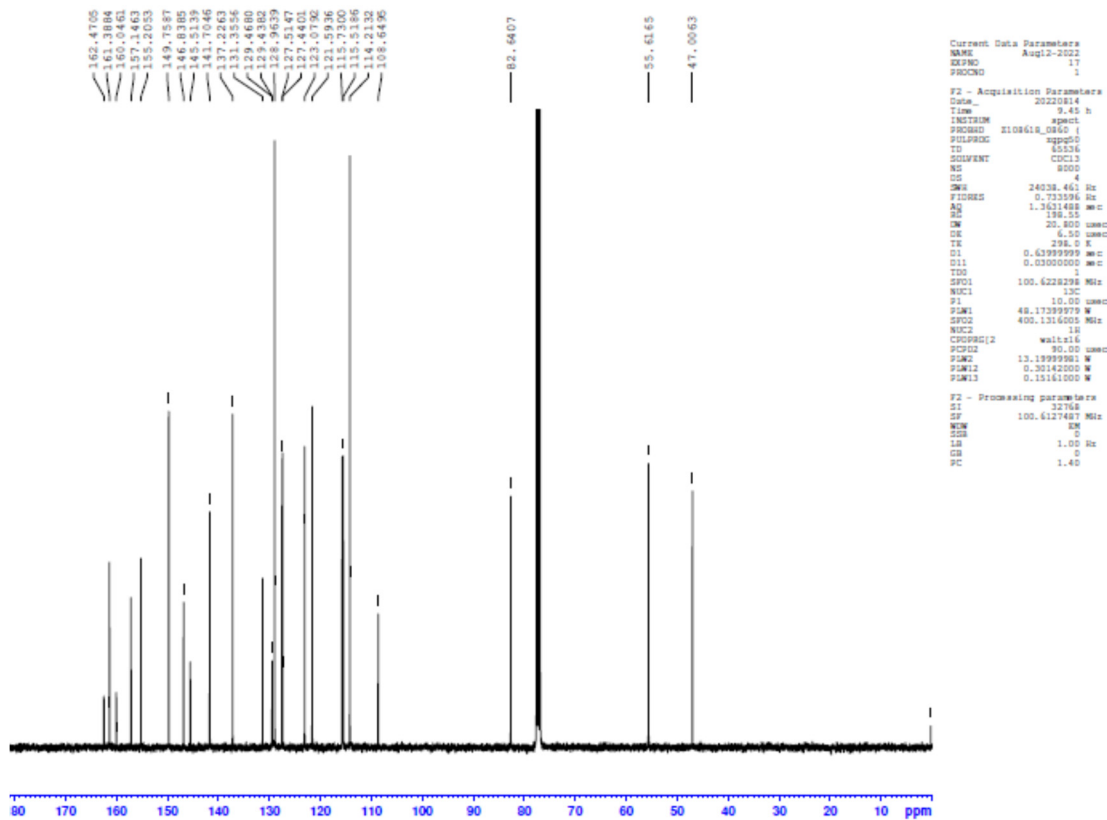
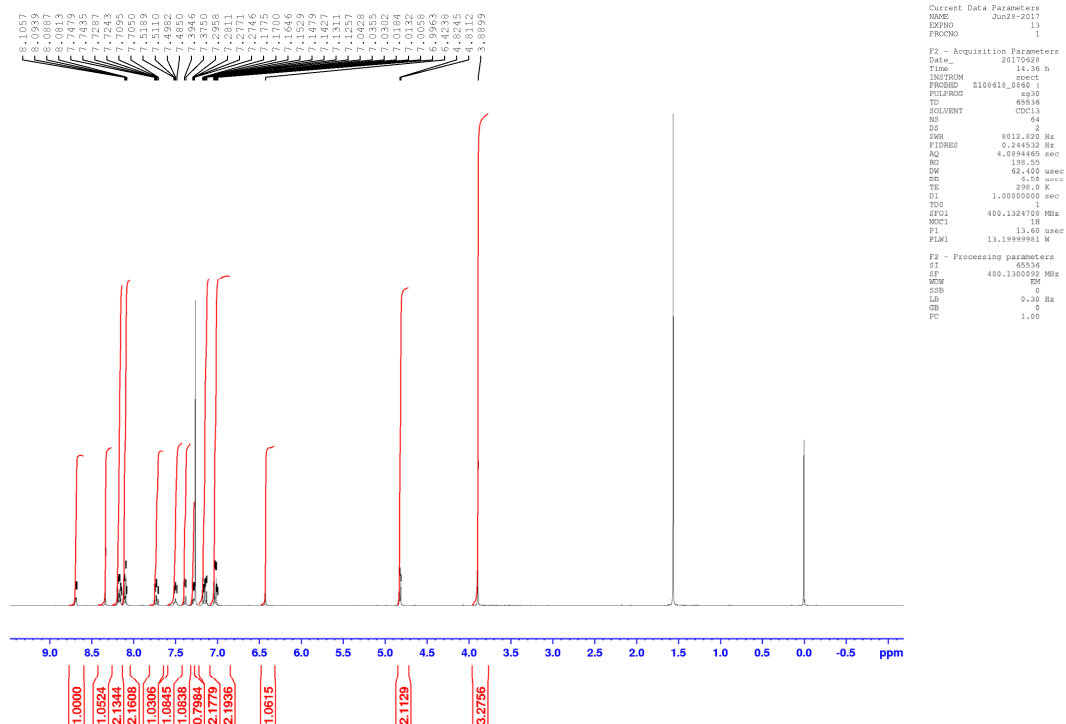
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NS 6400
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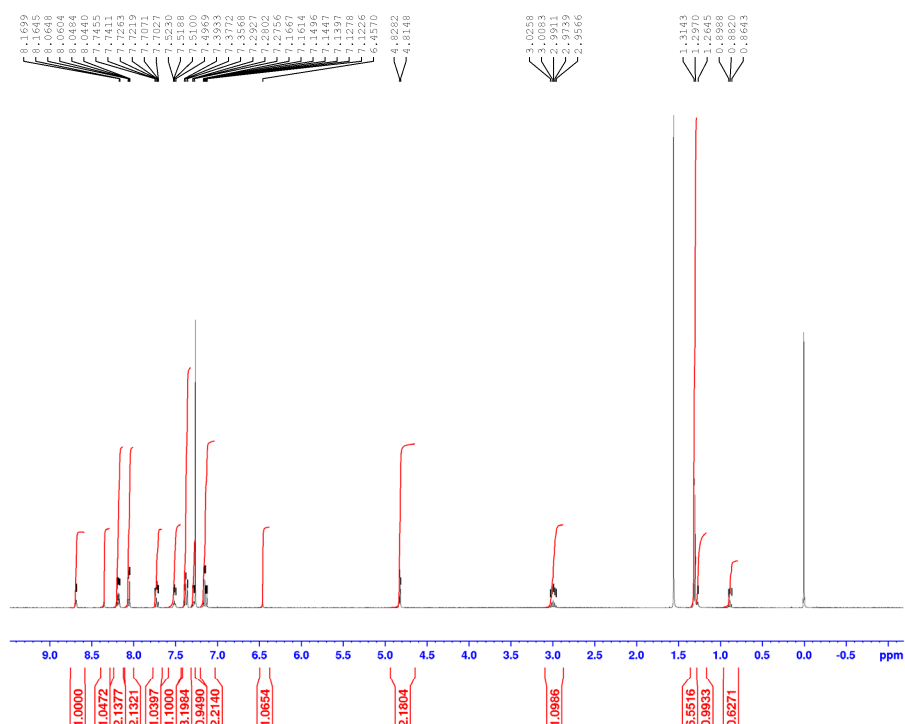
Compound 30



Compound 34



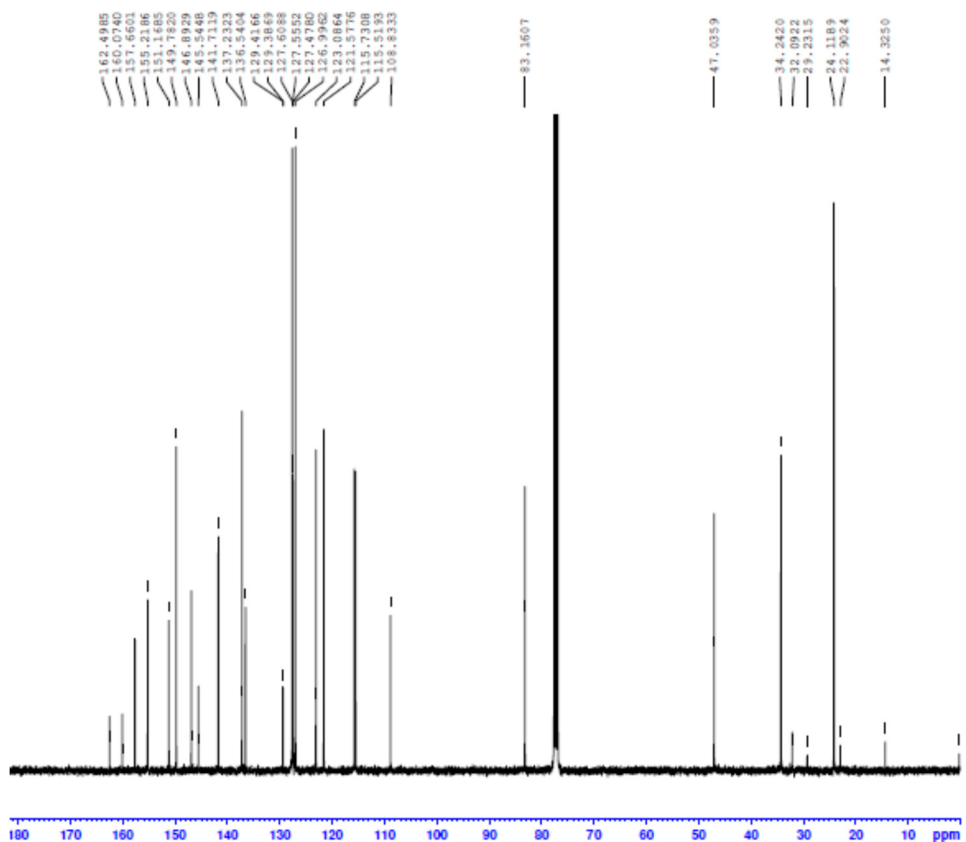
Compound 35



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 NS 64
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 D1 1.00000000 sec
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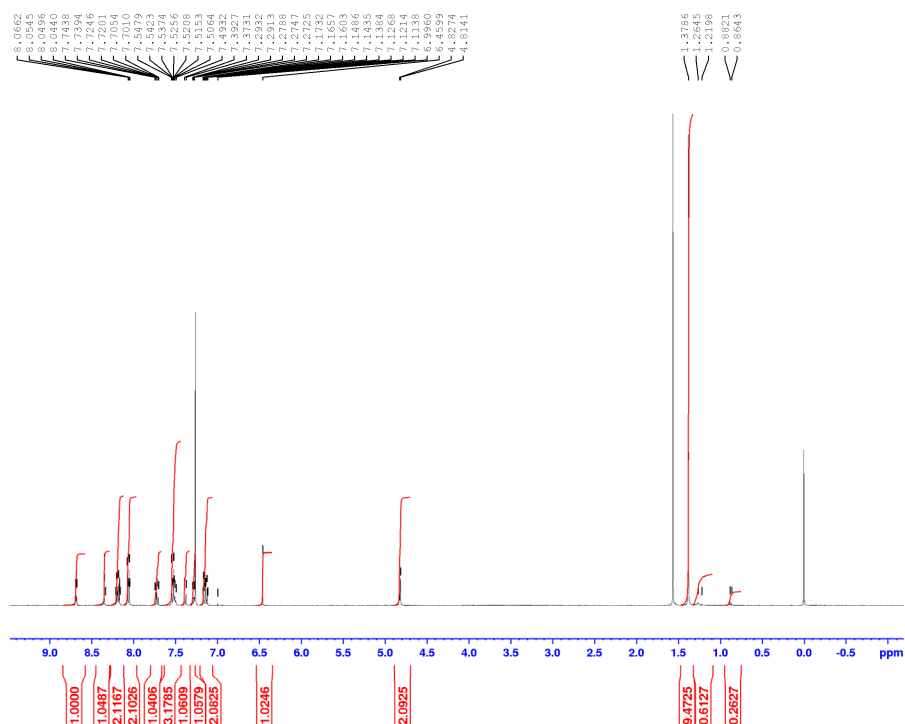


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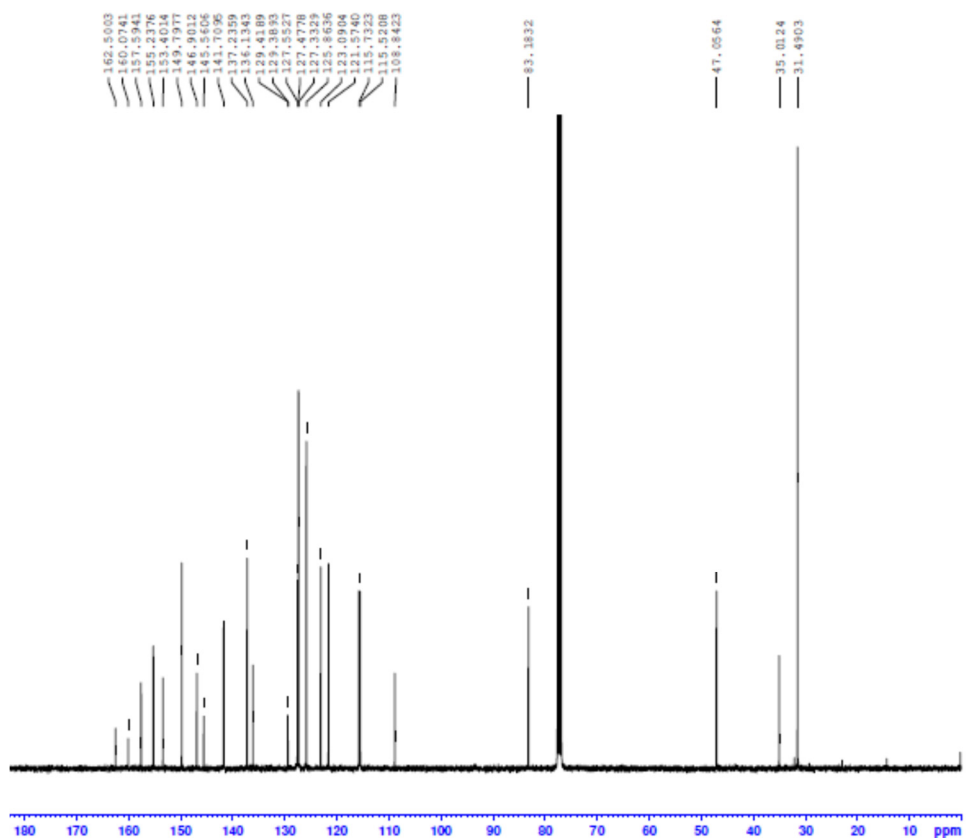
Compound 36



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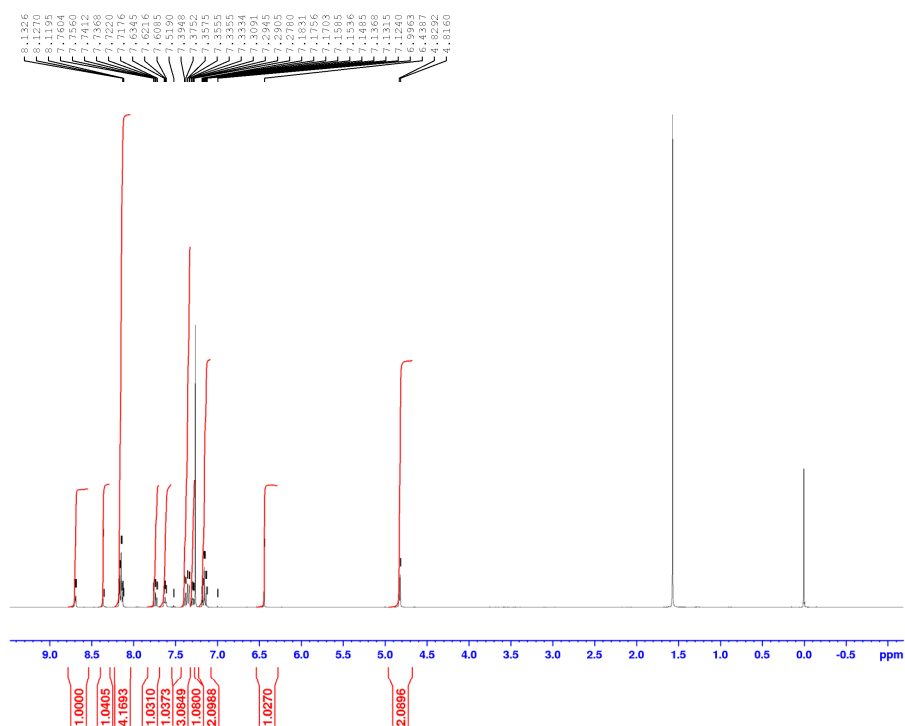


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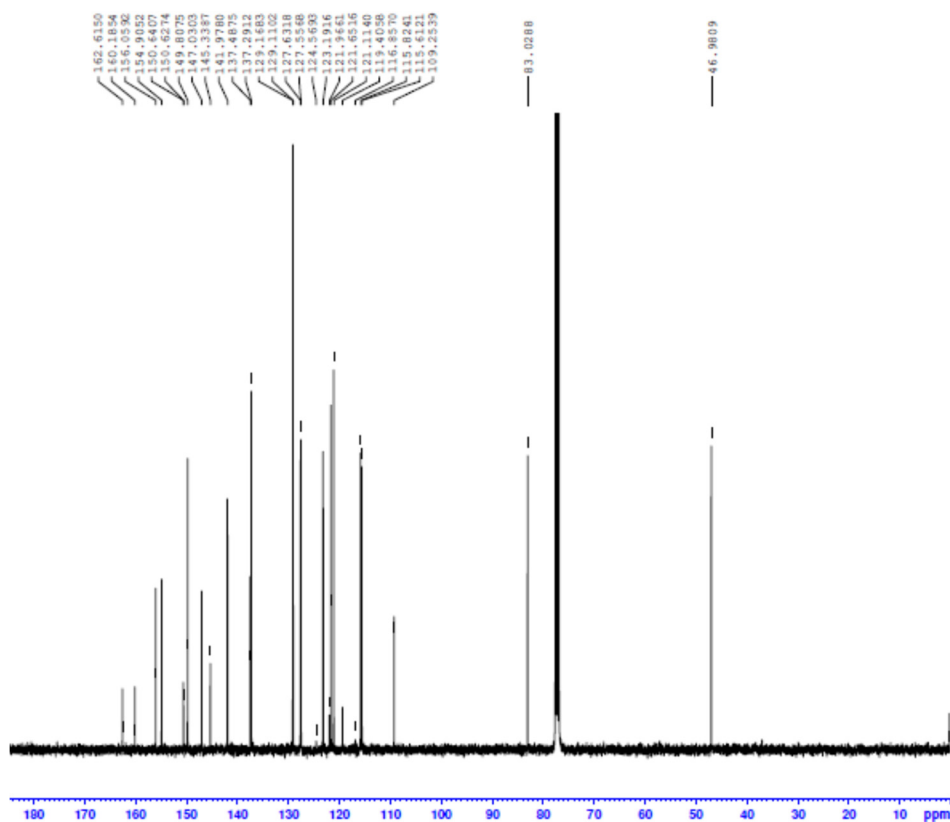
Compound 38



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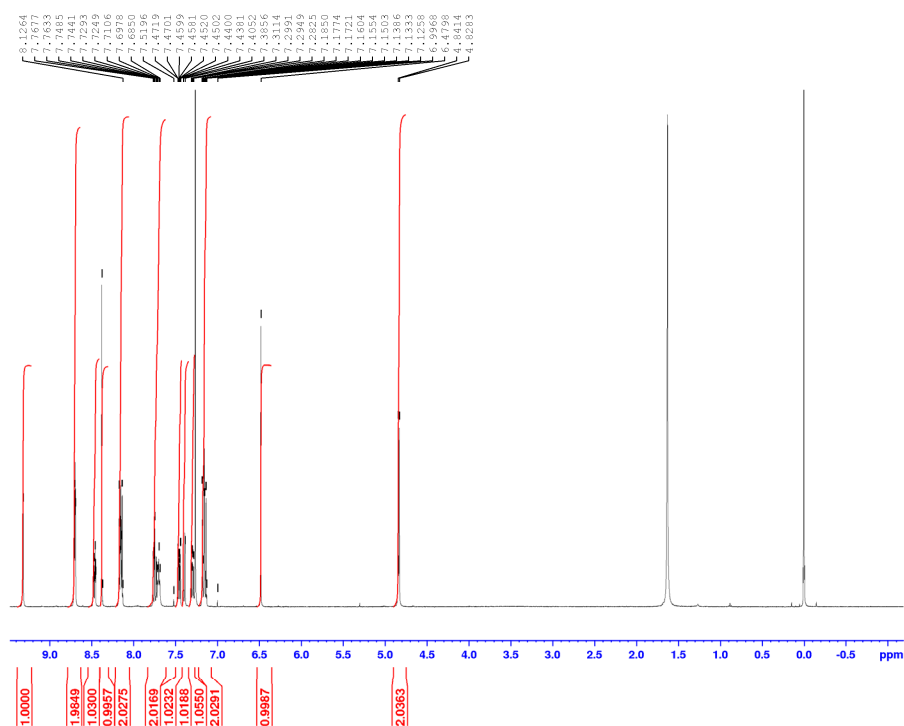


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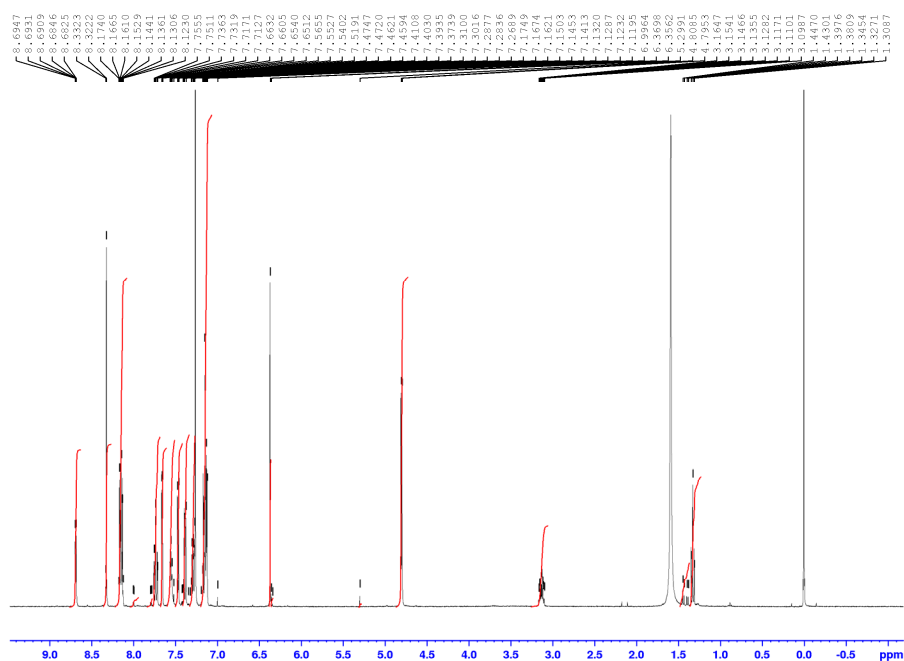
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Compound 39



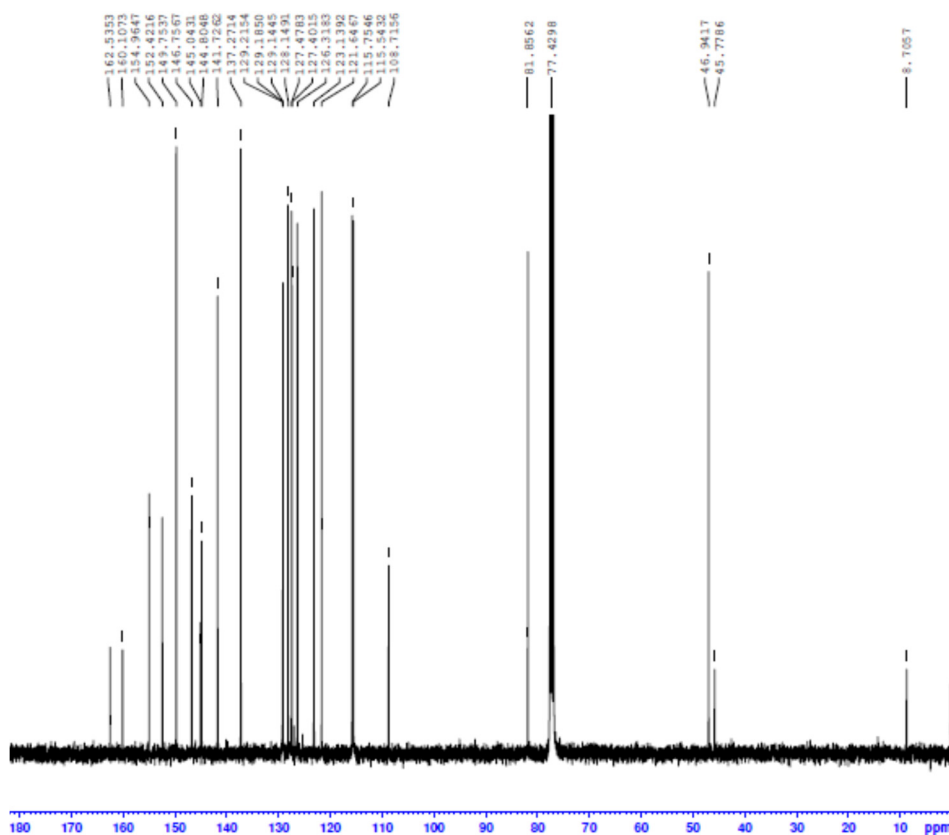
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 RG 190.50
 DW 62.400 usec
 DE 4.10 usec
 TE 298.0 K
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 NUC1 1H
 P1 13.60 usec
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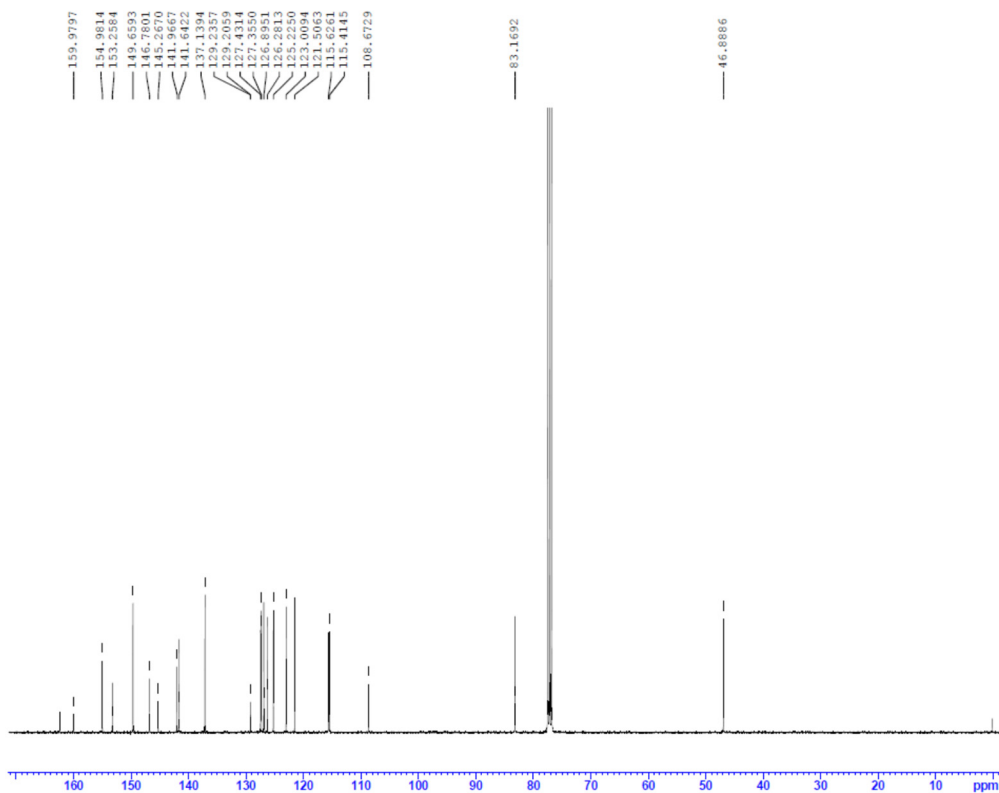
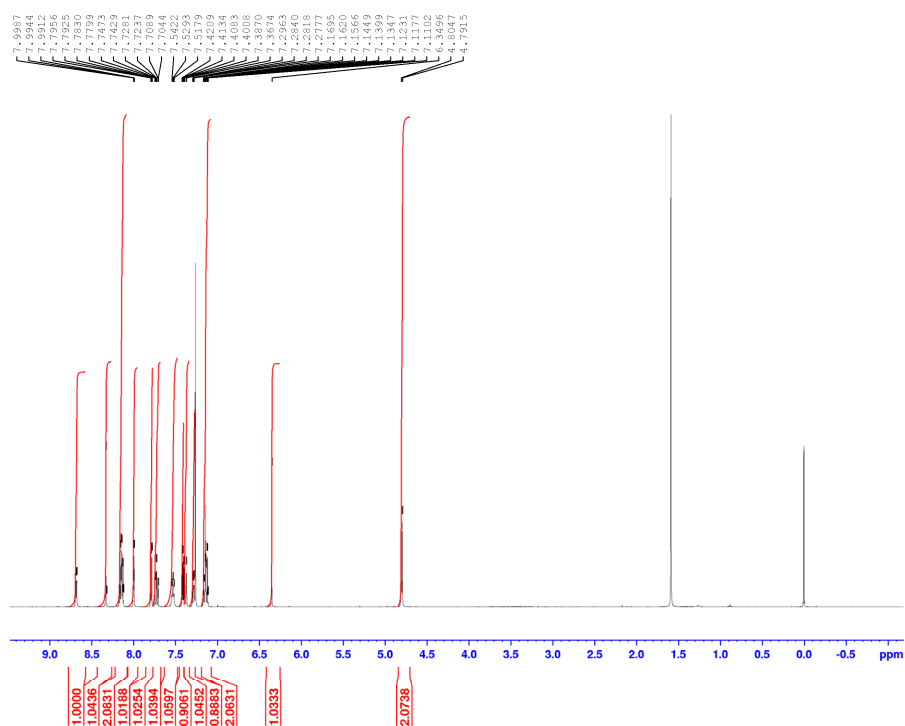


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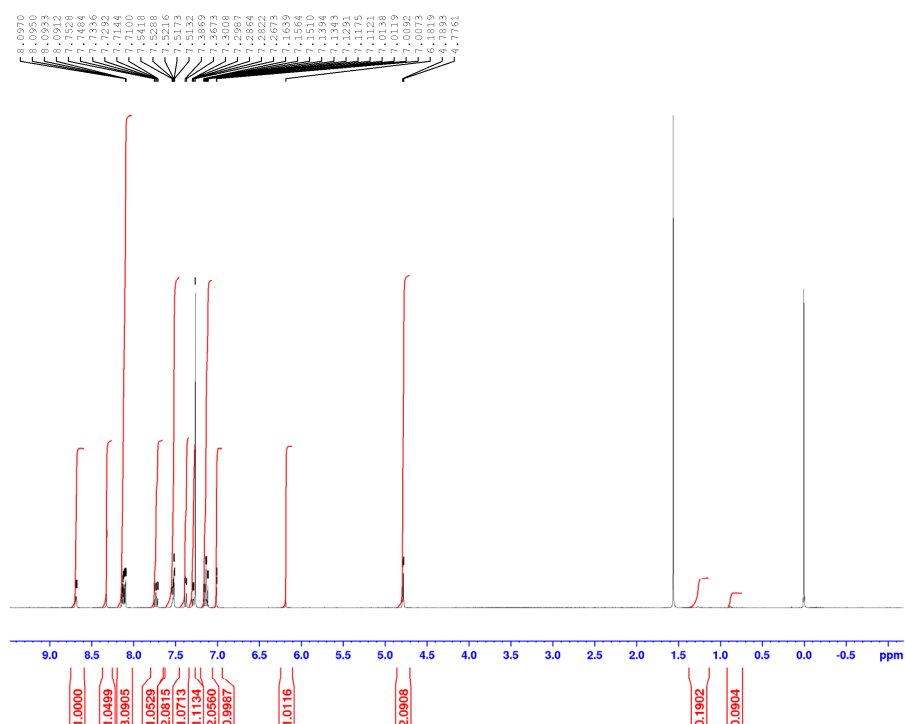
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 PLW13 0.15161000 W

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 PC 1.40

Compound 42



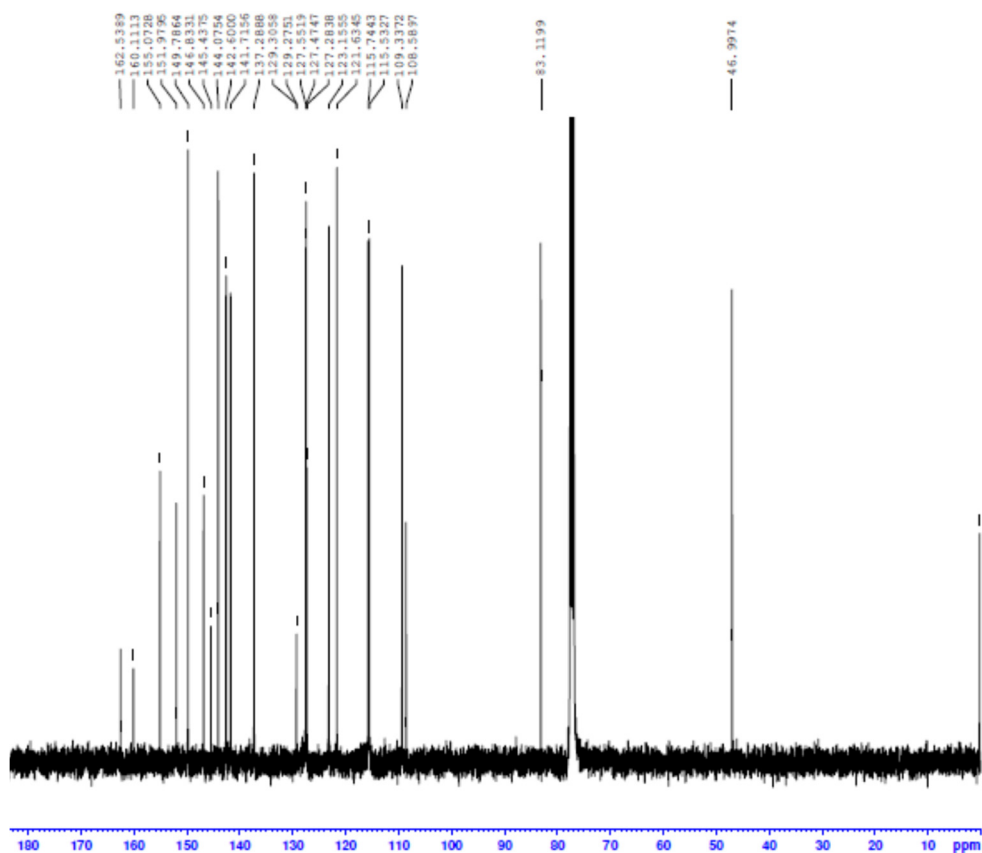
Compound 44



Current Data Parameters
 NAME Jul13-2017
 EXPNO 15
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170703
 Time 13:57 h
 INSTRUM spect
 PRGMRD Z108618_0860 (i
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 2
 FWH 8012.020 Hz
 FIDRES 0.244532 Hz
 AQ 4.898465 sec
 RG 198.55
 DW 60.400 usec
 DE 9.70 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 0
 SFO1 400.1324708 MHz
 NUC1 1H
 P1 13.60 usec
 PLW1 13.19999991 W

F2 - Processing parameters
 SI 60336
 SF 400.1300593 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.40

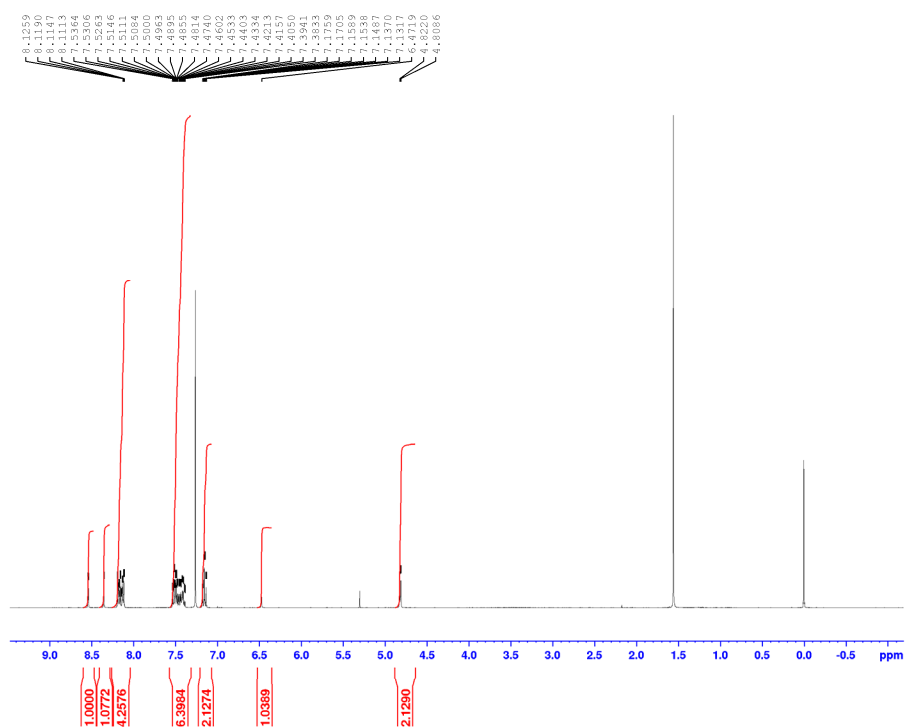


Current Data Parameters
 NAME Aug29-2022
 EXPNO 13
 PROCNO 1

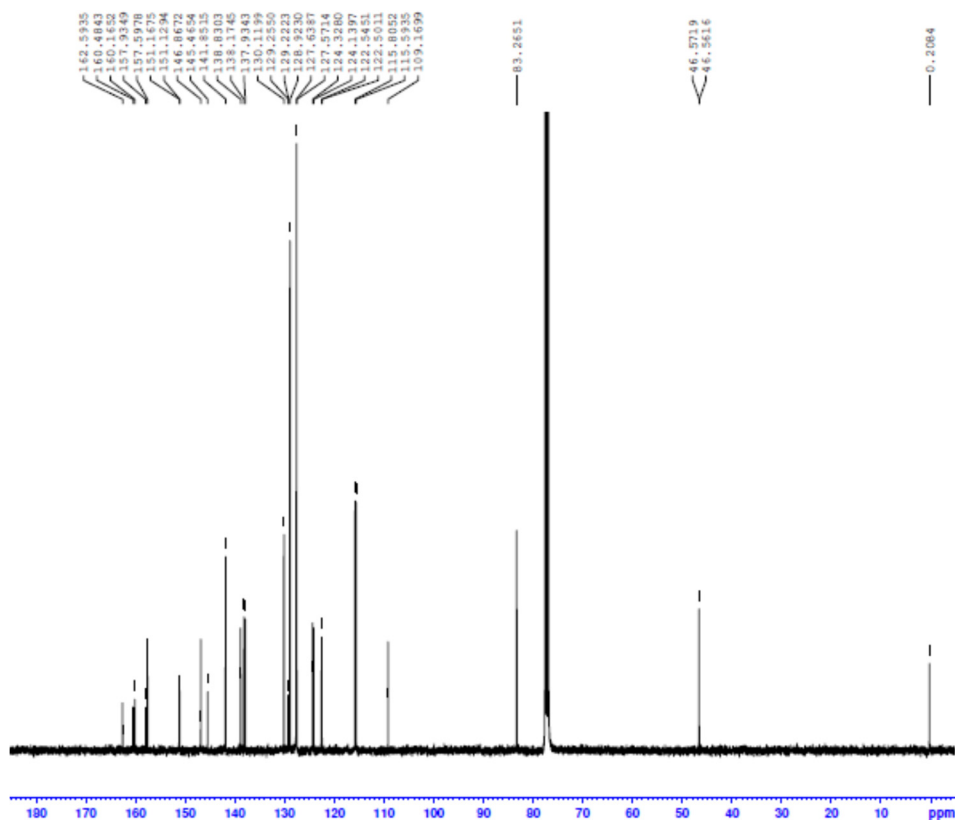
F2 - Acquisition Parameters
 Date_ 20220820
 Time 7:49 h
 INSTRUM spect
 PRGMRD Z108618_0860 (i
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 24000
 DS 4
 FWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 1.3631488 sec
 RG 198.55
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 0.63999999 sec
 D11 0.03000000 sec
 TDO 0
 SFO1 100.6228276 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 48.17399979 W
 SFO2 400.1316005 MHz
 NUC2 1H
 CPOPRG2 waltz16
 PCPD12 90.00 usec
 PLW2 13.19999981 W
 PLW12 0.30142000 W
 PLW13 0.15161000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127476 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Compound 52

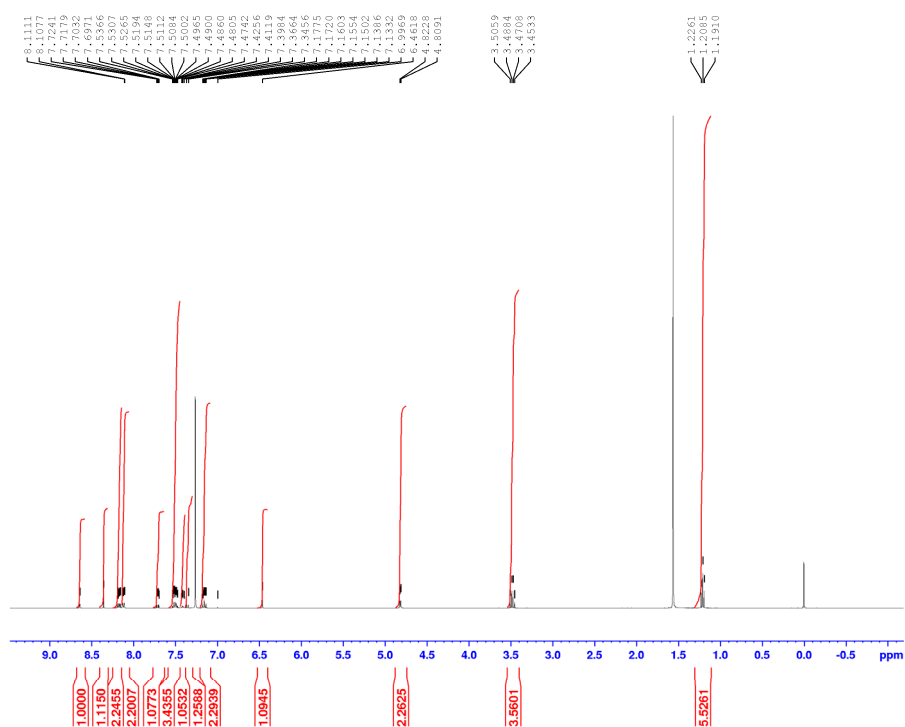


Current Data Parameters
 NAME Nov17-2016
 EXPNO 7
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20161117
 Time 11:47 h
 INSTRUM spect
 PRGNAME z108618_0860
 PULPROG zgpg30
 TD 65536
 SFO1 400.1324708 MHz
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 8012.020 Hz
 FIDRES 0.244532 Hz
 AQ 4.099465 sec
 RG 198.55
 DW 62.400 usec
 DE 9.70 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1324708 MHz
 NUC1 1H
 P1 13.60 usec
 PL1 13.19999991 W
 F2 - Processing parameters
 SI 60336
 SF 400.1300094 MHz
 WDW DM
 SSB 0 Hz
 LB 0.30
 GB 0
 PC 1.40

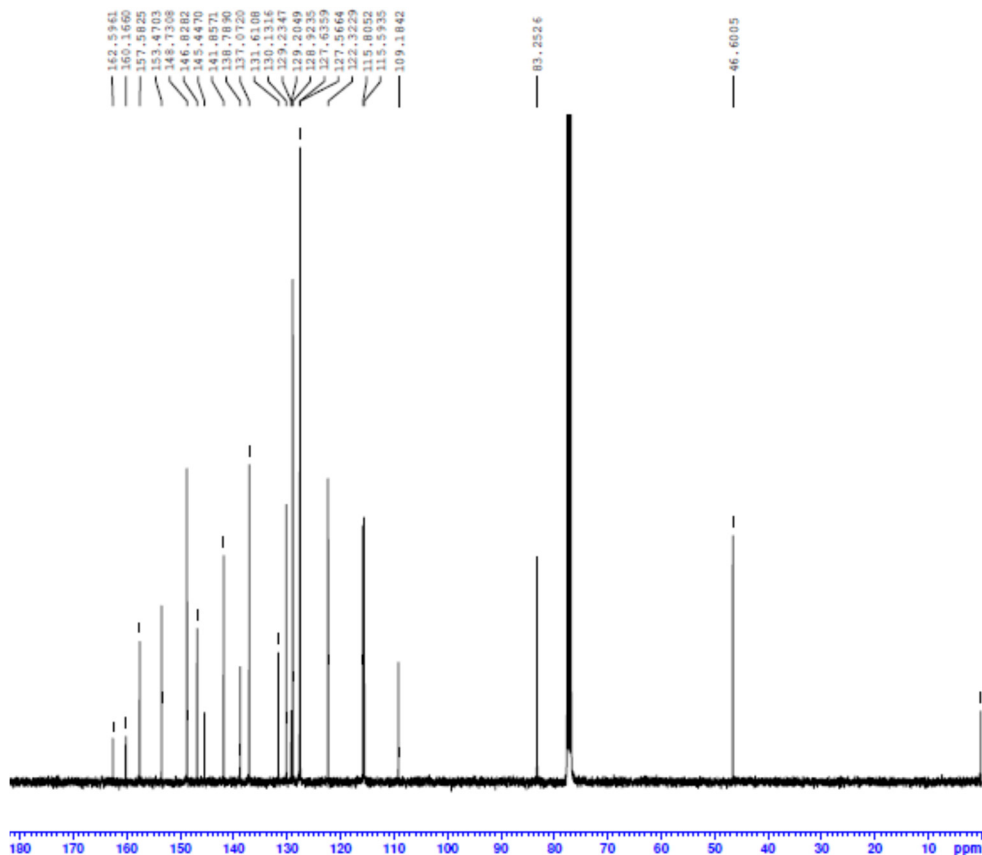


Current Data Parameters
 NAME Aug10-2022
 EXPNO 4
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20220811
 Time 9:32 h
 INSTRUM spect
 PRGNAME z108618_0860
 PULPROG zgpg30
 TD 65536
 SFO1 100.6228298 MHz
 SOLVENT CDCl3
 NS 20000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 1.351488 sec
 RG 198.55
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 0.63999999 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PL1 48.17399979 W
 SFO2 400.1316005 MHz
 NUC2 1H
 PCP2PRG2 wait16
 SFO2 400.1316005 MHz
 PL2 13.19999991 W
 SFO3 0.30142000 MHz
 PL3 0.13161000 W
 F2 - Processing parameters
 SI 32768
 SF 100.6127479 MHz
 WDW EM
 SSB 0 Hz
 LB 1.00 Hz
 GB 0
 PC 1.40

Compound 53



Current Data Parameters
 NAME Nov16-2016
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20161116
 Time 12.04 h
 INSTRUM spect
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 8012.620 Hz
 FIDRES 0.244532 Hz
 AQ 4.0094605 sec
 RG 196.55
 DW 62.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 TDO 1
 SFO1 400.1324708 MHz
 NUC1 1H
 P1 13.40 usec
 PLW1 13.19999981 W
 F2 - Processing parameters
 SI 400.14
 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME Aug22-2022
 EXPNO 12
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20220823
 Time 1.37 h
 INSTRUM spect
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 13000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 1.3631488 sec
 RG 196.55
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 0.63999999 sec
 D11 0.03000000 sec
 TDO 1
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 48.1739979 W
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.19999981 W
 PLW12 0.35142000 W
 PLW13 0.15161000 W
 F2 - Processing parameters
 SI 100.61
 SF 100.6127480 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

¹H NMR spectrum (CDCl₃) of compound 10a. The x-axis represents the chemical shift in ppm, ranging from 0.0 to 9.0. The spectrum shows several peaks, with integration values indicated above them.

Chemical shift values (ppm): 9.1465, 9.1374, 8.1299, 8.1259, 8.1259, 8.1257, 7.6262, 7.6300, 7.5256, 7.5192, 7.5185, 7.4981, 7.4899, 7.4859, 7.4850, 7.4849, 7.4699, 7.4159, 7.4025, 7.4025, 7.3248, 7.3248, 7.3034, 7.2486, 7.2486, 7.2474, 7.2474, 7.2199, 7.1727, 7.1674, 7.1674, 7.1507, 7.1456, 7.1338, 7.1338, 6.1265, 6.1265, 4.7733, 4.7599, 3.8819, 3.5058, 3.4883, 3.4708, 3.4553, 1.2261, 1.2086, 1.1911.

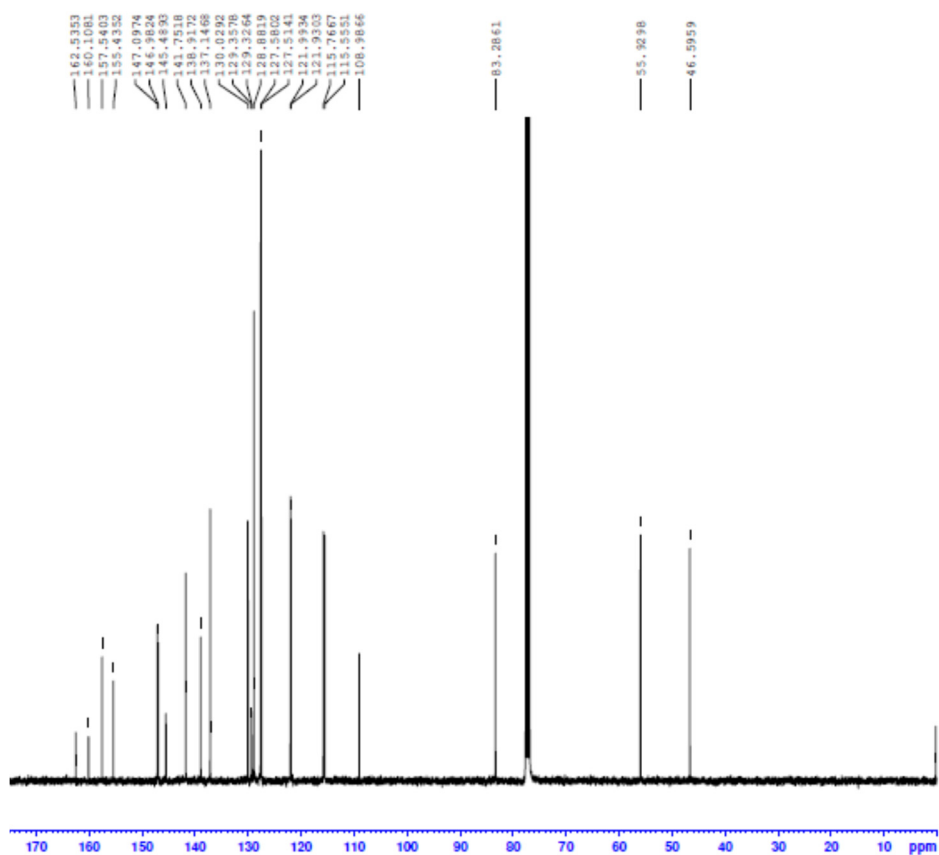
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Name      Data      Parameters
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CURRENT   Dec21-2016
EXPNO     19
PROCNO    1

F2 - Acquisition Parameters
-----
Date_     20161221
Time      15:47 h
INSTRUM   spect
PROCNO    2106010_0962
PULPROG   zgpg30
TD         65536
DO         2330
TO SOLVENT CDCL3
NS         64
DS         2
SWH        8012.020 Hz
FIDRES     0.2454532 Hz
AQ         4.4389465 sec
RG          198.55
DE         62.400 usec
TE         6.50 usec
UE         298.0 K
TE2        1.00000000 sec
TD0         1
SF01       400.132470 MHz
NC1        1H
PC1         13.60 usec
PL1        13.19999991 W
PL2

F2 - Processing parameters
-----
SI         65536
SF          400.1300091 MHz
WDW        EM
SSB         0
GB          0.30 Hz
CB          0
PC          1.00

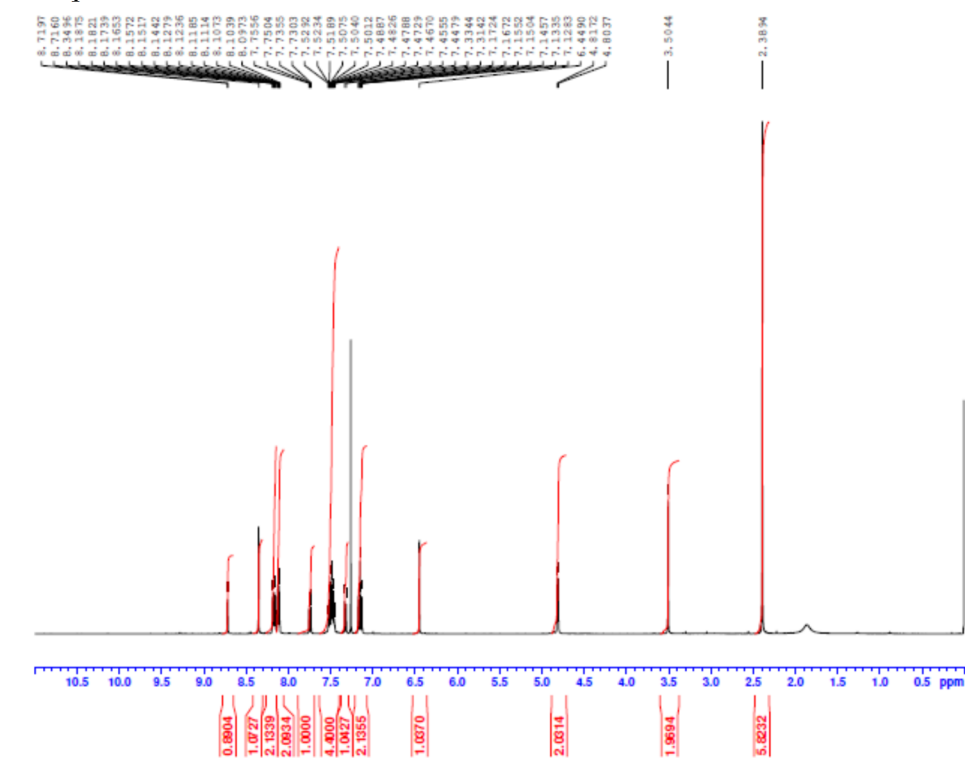
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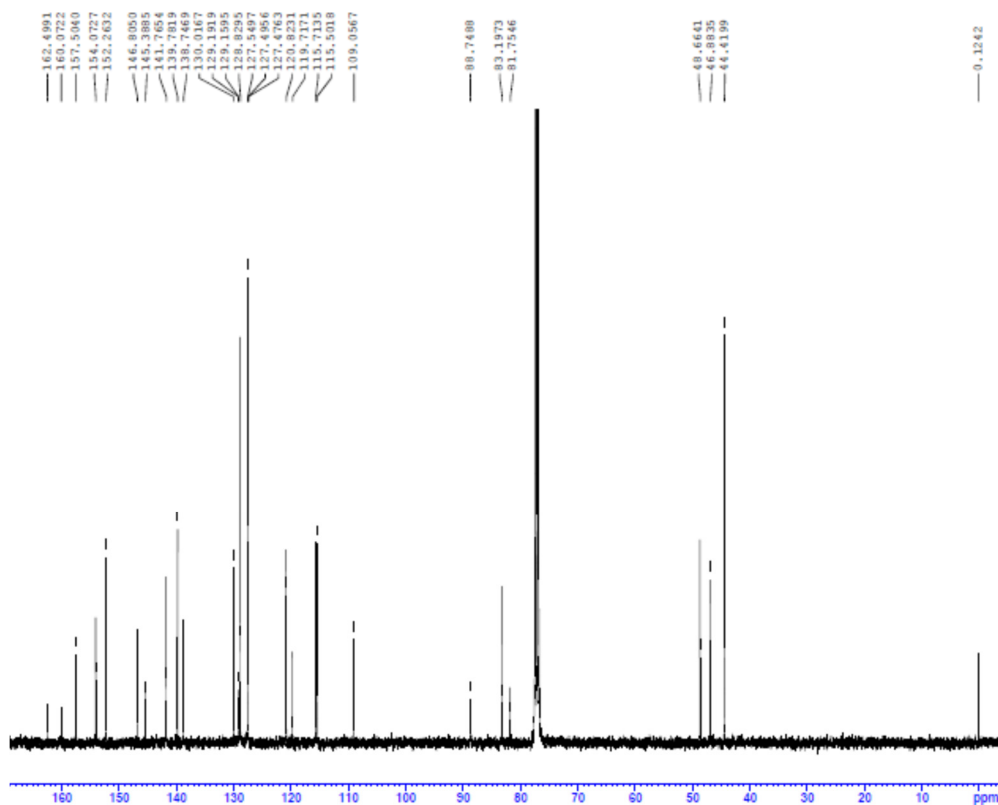
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Current Data Parameters
NAME      Aug22-2022
EXPNO     14
PROCNO    1
-----
P2 - Acquisition Parameters
Date_      20220223
Time       8.05 h
INSTRUM    spect
PROBHD     ZN04610.04610
PULPROG    zgpg30
TD         65536
SFOFREQ    CQC13
RG          32.00000000
DS         4
SS         2
FIDRES     2403.64 Hz
AQ         0.735594 sec
RG         1.3631448 sec
AQ         193.999999 sec
TE         29.80000000
DE         0.039999999
D1         0.020000000
D2         1
D3         1
SFO1       100.6222999 MHz
NUC1        13
P1         10.00000000
P2         10.00000000
P3         48.13999999 MHz
NUC2         1
P4         100.6136000 MHz
NUC3         1
P5         10.00000000
PCPD2       1
PCPD3       1
PCPD4       1
PCPD5       1
PCPD6       1
PCPD7       1
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PCPD
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Compound 55

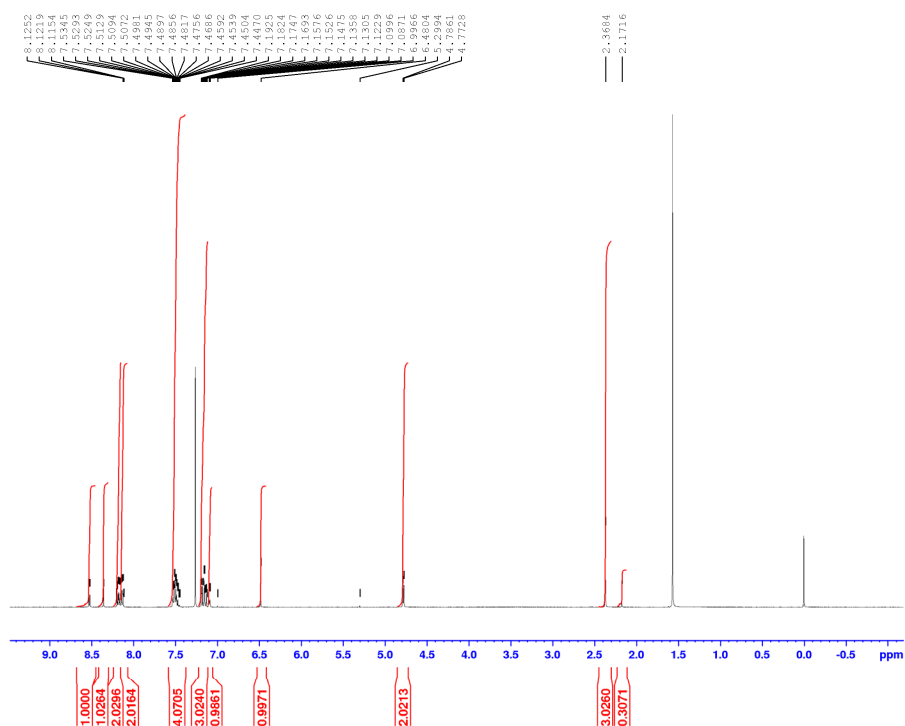


Current Data Parameters
 NAME Aug10-2022
 EXPNO 7
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20220831
 Time 14.52 h
 INSTRUM spect
 PROBRD Z100618_0560 (i
 PULPROG zgpg30
 TD 65536
 SFO 400.1324708 MHz
 SOLVENT CDCl3
 NS 64
 DS 4
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 4.303443 sec
 RG 198.55
 WC 62.400 umsec
 DE 6.50 umsec
 TE 298.2 K
 D1 1.00000000 sec
 D11 0.03000000 sec
 SFO1 400.1324708 MHz
 NUC1 13
 P1 4.13 umsec
 P2 13.50 umsec
 P3 13.19999991 W
 F2 - Processing parameters
 SI 65536
 SF 400.1324708 MHz
 WCN 0
 SGB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

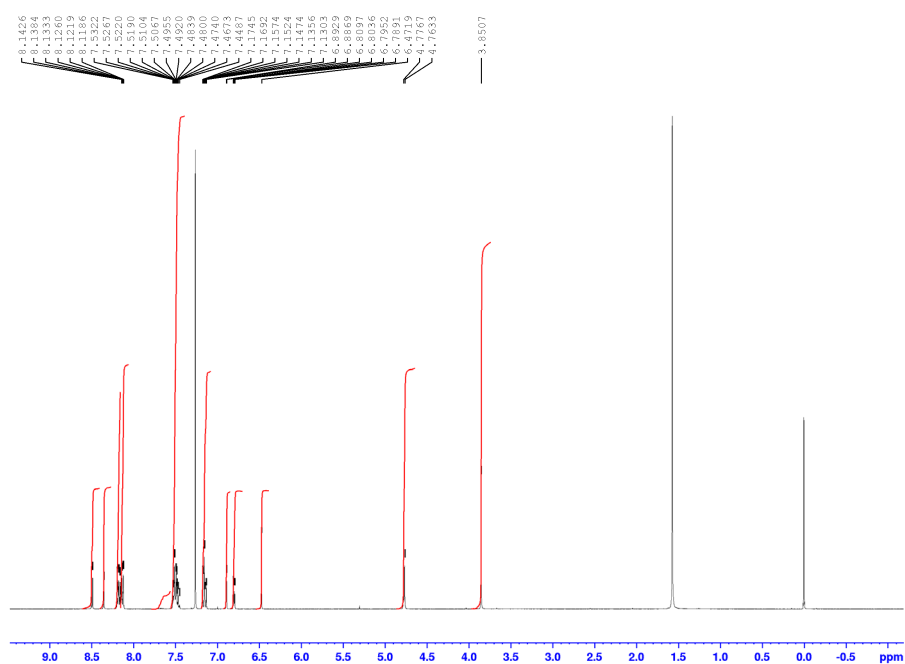


Current Data Parameters
 NAME Aug10-2022
 EXPNO 8
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20220831
 Time 2.05 h
 INSTRUM spect
 PROBRD Z100618_0560 (i
 PULPROG zgpg30
 TD 65536
 SFO 400.1324708 MHz
 SOLVENT CDCl3
 NS 14000
 DS 4
 SWH 24038.462 Hz
 FIDRES 0.733596 Hz
 AQ 1.9431488 sec
 RG 198.55
 WC 20.800 umsec
 DE 6.50 umsec
 TE 298.2 K
 D1 0.03000000 sec
 D11 0.03000000 sec
 SFO1 400.1324708 MHz
 NUC1 13
 P1 10.00 umsec
 P2 40.17599979 W
 SFO2 400.13140000 MHz
 NUC2 1H
 P3 13.19999991 W
 P4 0.15161000 W
 F2 - Processing parameters
 SI 32768
 SF 400.1324708 MHz
 WCN 0
 SGB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Compound 62



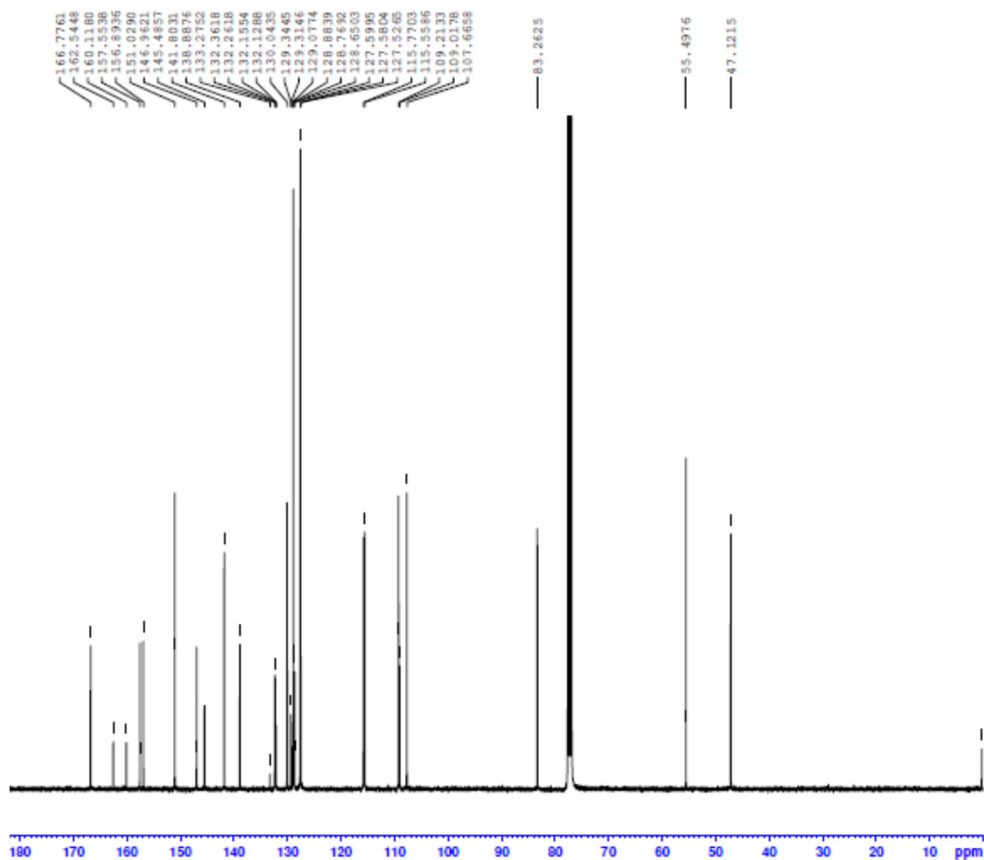
Compound 63



Current Data Parameters
 NAME Dec19-2016
 EXPNO 14
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161226
 Time 15:11
 INSTRUM spect
 PROBRD z100618_0860
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 64
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.244532 Hz
 AQ 4.0034165 sec
 RG 198.55
 DW 62.400 usec
 DE 4.50 usec
 TE 298.0 K
 D1 1.40000000 sec
 TDO 1
 SFO1 400.1324700 MHz
 NUC1 1H
 P1 13.60 usec
 PL1 13.19999991 W

F2 - Processing parameters
 SI 65536
 SF 400.1324700 MHz
 WDW EM
 GB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME Aug16-2022
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20220826
 Time 0:35 h
 INSTRUM spect
 PROBRD z100618_0860
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 32000
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.733596 Hz
 AQ 1.3631468 sec
 RG 198.55
 DW 20.800 usec
 DE 6.50 usec
 TE 298.0 K
 D1 0.639999999 sec
 D11 0.030000000 sec
 TDO 0
 SFO1 100.6228298 MHz
 NUC1 13C
 P1 10.00 usec
 PL1 48.173999775 W
 SFO2 400.1316000 MHz
 NUC2 1H
 PCPORG2 waltz16
 PCPD2 90.00 usec
 PLW2 13.199999901 W
 PLW12 0.30142000 W
 PLW13 0.15161000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127487 MHz
 WDW EM
 GB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

