

Supporting Information
for DOI: 10.1055/a-2016-4337

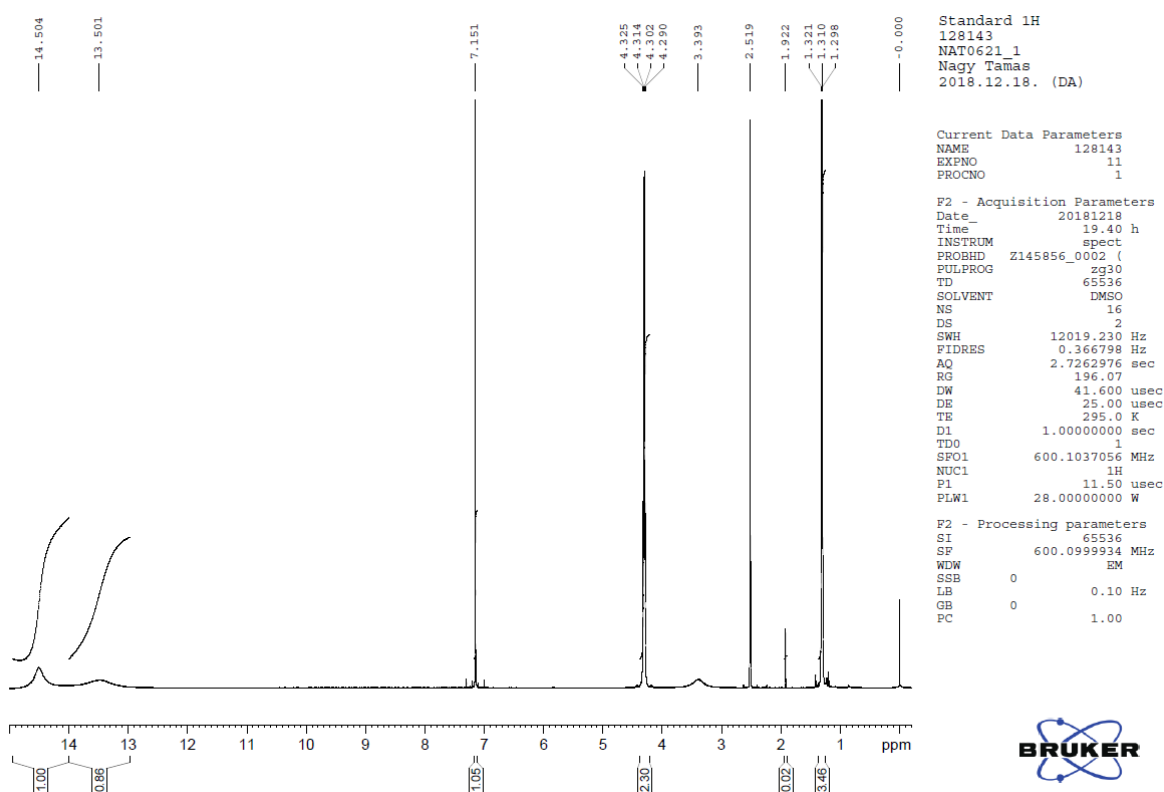
© 2023. Thieme. All rights reserved.

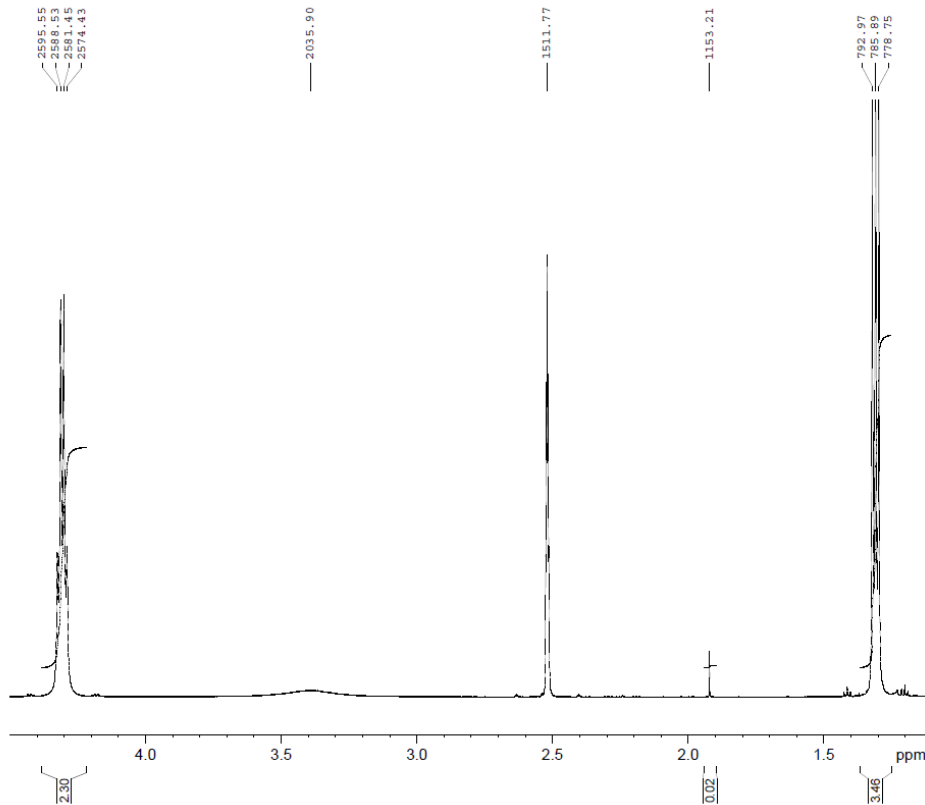
Georg Thieme Verlag KG, Rüdigerstraße 14, 70469 Stuttgart, Germany

New, scalable process for the preparation of 5-acetyl-1H-pyrazole-3-carboxylic acid, a key intermediate of darolutamide

¹H and ¹³C NMR spectra, and LC-MS data

5-(Ethoxycarbonyl)-1H-pyrazole-3-carboxylic acid (17a)



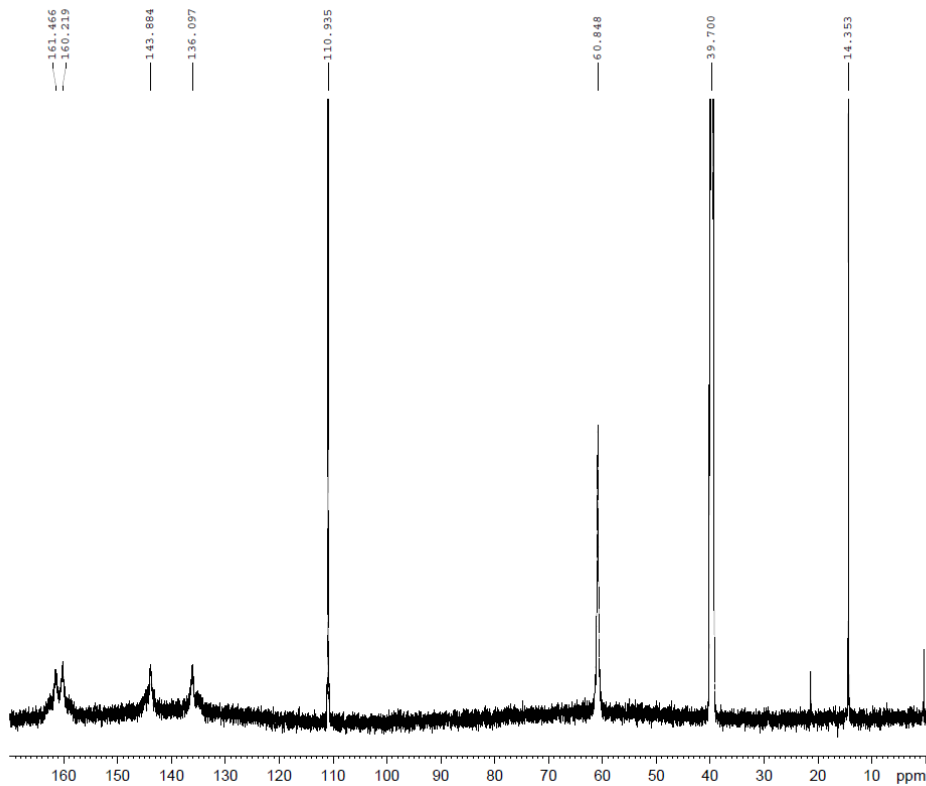


Standard 1H
128143
NAT0621_1
Nagy Tamas
2018.12.18. (DA)

Current Data Parameters
NAME 128143
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20181218
Time 19.40 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 196.07
DW 41.600 usec
DE 25.00 usec
TE 295.0 K
D1 1.0000000 sec
TD0 1
SFO1 600.1037056 MHz
NUC1 1H
P1 11.50 usec
PLW1 28.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0999934 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



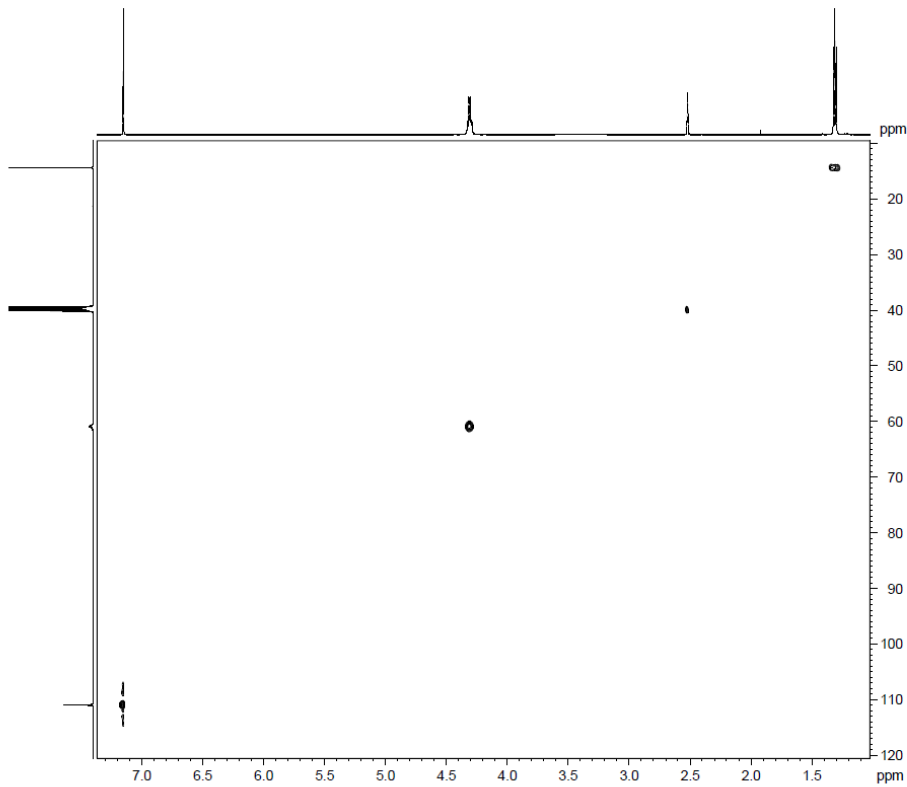
Standard 13C
128143
NAT0621_1
Nagy Tamas
2018.12.18. (DA)

Current Data Parameters
NAME 128143
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20181218
Time 20.48 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9043968 sec
RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.0000000 sec
D11 0.0300000 sec
TD0 1
SFO1 150.9103545 MHz
NUC1 13C
P1 10.00 usec
PLW1 60.3629896 W
SFO2 600.1024004 MHz
NUC2 1H
CPDPRG(2) waltz16
PCPD2 80.00 usec
PLW2 26.4260060 W
PLW12 0.59457999 W
PLW13 0.29859999 W

F2 - Processing parameters
SI 32768
SF 150.8952987 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





HSQC (140 Hz)
128143
NAT0621_1
Nagy Tamas
2018.12.18. (DA)

```

Current Data Parameters
NAME      128143
EXPNO    1
PROCNO   1

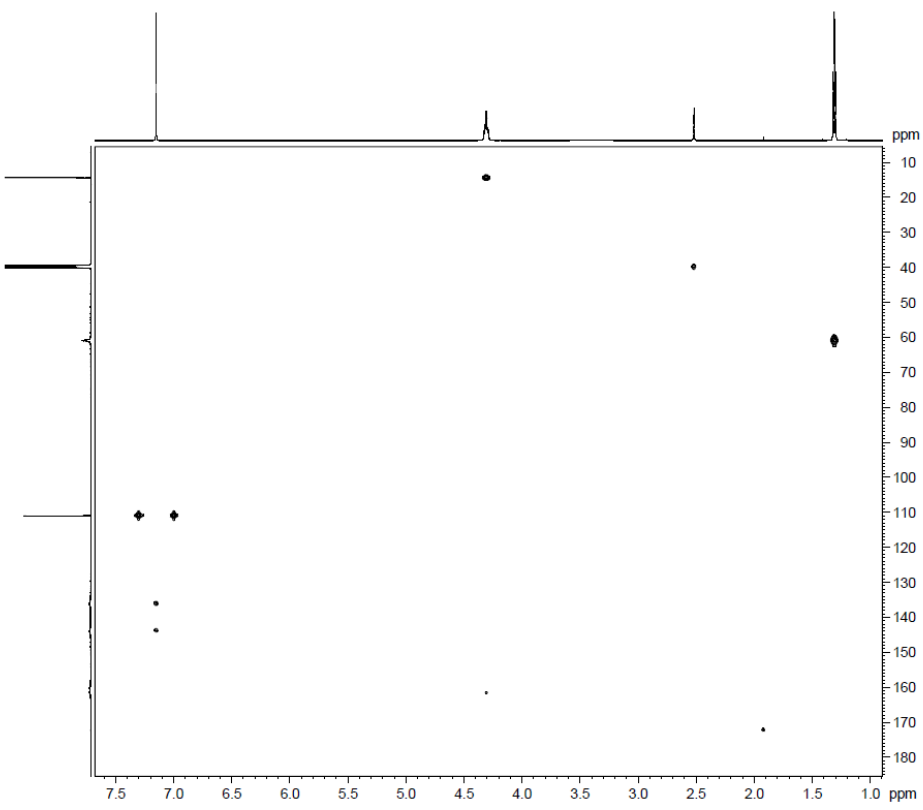
F2 - Acquisition Parameters
Date_    20181218
Time     20:50 h
INSTRUM  spect
PROBHD   214886_0002 [
PULPROG  hmcgpp1p0q0
TD        7048
SOLVENT  DMSO
NS        4
DS        4
SWH       7812.500 Hz
FIDRES   7.5229396 Hz
AQ        0.1310720 sec
RG        386.07
DM        64.000 usec
DE        25.00 usec
TE        298.2 K
CNET2    140.000000
D1        0.0000000 sec
D11       1.0000000 sec
D12       0.0071817 sec
D13       0.0300000 sec
D14       0.0001200 sec
D15       0.0008207 sec
D16       0.0000151 sec
TD0       1
TD0V      1
SFOF2     600.1039007 MHz
NUC1      13C
P1         11.50 usec
PC         21.00 usec
PL1        0 usec
PL12      28.0000000 W
SFO1      150.9118155 MHz
NUC2      1H
CDDPRG2   2
PC2        0 usec
PL2        0 usec
PL12      60.2629898 W
CNAME1(1) SMC01.100
CNAME1(2) SMC01.100
CNAME1(3) SMC01.100
CNAME1(4) SMC01.100
CP1        50.00 %
CP2        30.00 %
CP3        11.00 %
CP4        1.00 %
P19       1000.00 usec
P19       600.00 usec

F1 - Acquisition parameters
TD        256
SFO1      150.9119 MHz
FIDRES   268.492047 Hz
SW        219.417 ppm
PULPROG  hmcgpp1p0q0
PC        21.00 usec
SFOF1     600.0999234 MHz
NUC1      13C
P1         11.50 usec
PC         21.00 usec
PL1        0 usec
PL12      28.0000000 W
SFO1      150.9118155 MHz
NUC2      1H
CDDPRG2   2
PC2        0 usec
PL2        0 usec
PL12      60.2629898 W
CNAME1(1) SMC01.100
CNAME1(2) SMC01.100
CNAME1(3) SMC01.100
CNAME1(4) SMC01.100
CP1        50.00 %
CP2        30.00 %
CP3        11.00 %
CP4        1.00 %
P19       1000.00 usec
P19       600.00 usec

F2 - Processing parameters
SI        1024
SF        600.0999234 MHz
WDW       EM
SSB       2
LB         0 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI        1024
SF        150.9118155 MHz
WDW       EM
SSB       2
LB         0 Hz
GB         0
PC         1.40

```



HMBC (8 Hz, 140 Hz)
128143
NAT0621_1
Nagy Tamas
2018.12.18. (DA)

```

Current Data Parameters
NAME      128143
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_    20181218
Time     21:05 h
INSTRUM  spect
PROBHD   214886_0002 [
PULPROG  hmcgpp1p0q0
TD        7048
SOLVENT  DMSO
NS        4
DS        4
SWH       7812.500 Hz
FIDRES   7.5229396 Hz
AQ        0.1310720 sec
RG        386.07
DM        64.000 usec
DE        25.00 usec
TE        298.2 K
CNET2    140.000000
D1        0.0000000 sec
D11       1.0000000 sec
D12       0.0071817 sec
D13       0.0300000 sec
D14       0.0001200 sec
D15       0.0008207 sec
D16       0.0000151 sec
TD0       1
TD0V      1
SFOF2     600.1039007 MHz
NUC1      13C
P1         11.50 usec
PC         21.00 usec
PL1        0 usec
PL12      28.0000000 W
SFO1      150.9118155 MHz
NUC2      1H
CDDPRG2   2
PC2        0 usec
PL2        0 usec
PL12      60.2629898 W
CNAME1(1) SMC01.100
CNAME1(2) SMC01.100
CNAME1(3) SMC01.100
CNAME1(4) SMC01.100
CP1        50.00 %
CP2        30.00 %
CP3        11.00 %
CP4        1.00 %
P19       1000.00 usec
P19       600.00 usec

F1 - Acquisition parameters
TD        256
SFO1      150.9119 MHz
FIDRES   268.492047 Hz
SW        219.417 ppm
PULPROG  hmcgpp1p0q0
PC        21.00 usec
SFOF1     600.0999234 MHz
NUC1      13C
P1         11.50 usec
PC         21.00 usec
PL1        0 usec
PL12      28.0000000 W
SFO1      150.9118155 MHz
NUC2      1H
CDDPRG2   2
PC2        0 usec
PL2        0 usec
PL12      60.2629898 W
CNAME1(1) SMC01.100
CNAME1(2) SMC01.100
CNAME1(3) SMC01.100
CNAME1(4) SMC01.100
CP1        50.00 %
CP2        30.00 %
CP3        11.00 %
CP4        1.00 %
P19       1000.00 usec
P19       600.00 usec

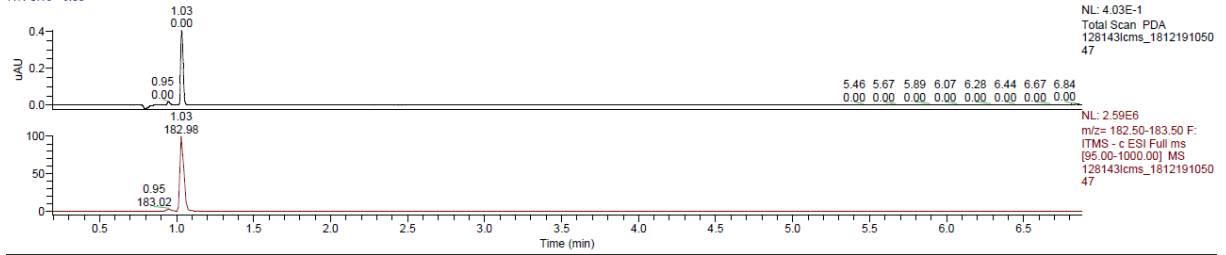
F2 - Processing parameters
SI        1024
SF        600.0999234 MHz
WDW       EM
SSB       2
LB         0 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI        1024
SF        150.9118155 MHz
WDW       EM
SSB       2
LB         0 Hz
GB         0
PC         1.40

```



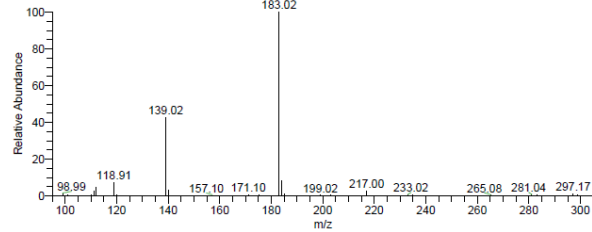
RT: 0.19 - 6.88



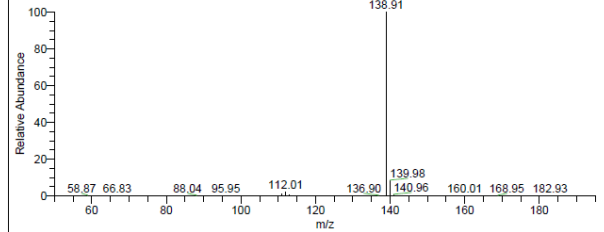
NL: 4.03E-1
 Total Scan PDA
 128143\cms_181219105047

NL: 2.59E6
 m/z= 182.50-183.50 F:
 ITMS - c ESI Full ms
 [95.00-1000.00] MS
 128143\cms_181219105047

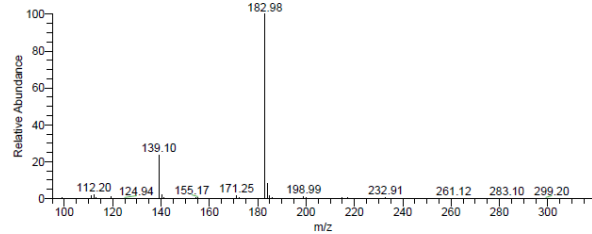
128143\cms_181219105047 #189 RT: 0.95 AV: 1 SB: 44 0.63-0.84, 1.25-1.48 NL: 6.41E4
 F: ITMS - c ESI Full ms [95.00-1000.00]



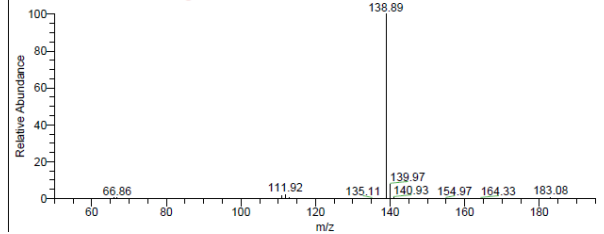
128143\cms_181219105047 #187 RT: 0.94 AV: 1 NL: 2.69E4
 F: ITMS - c ESI d Full ms2 182.95@cid35.00 [50.00-195.00]



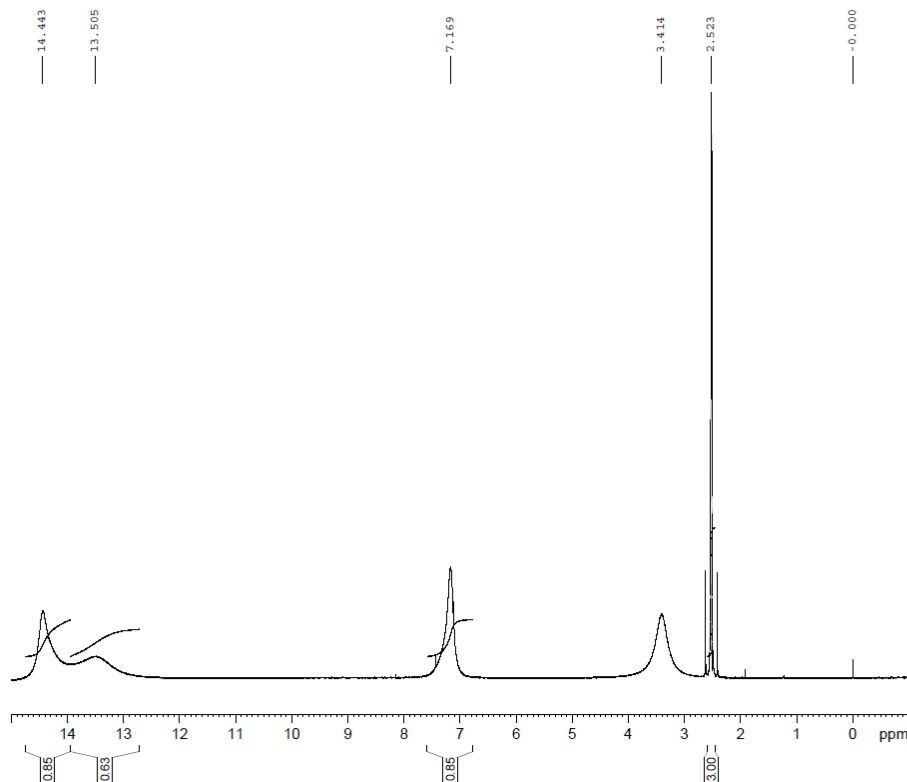
128143\cms_181219105047 #205 RT: 1.03 AV: 1 NL: 2.59E6
 F: ITMS - c ESI Full ms [95.00-1000.00]



128143\cms_181219105047 #210 RT: 1.05 AV: 1 NL: 4.96E5
 F: ITMS - c ESI d Full ms2 182.95@cid35.00 [50.00-195.00]



5-Acetyl-1H-pyrazole-3-carboxylic acid (2)

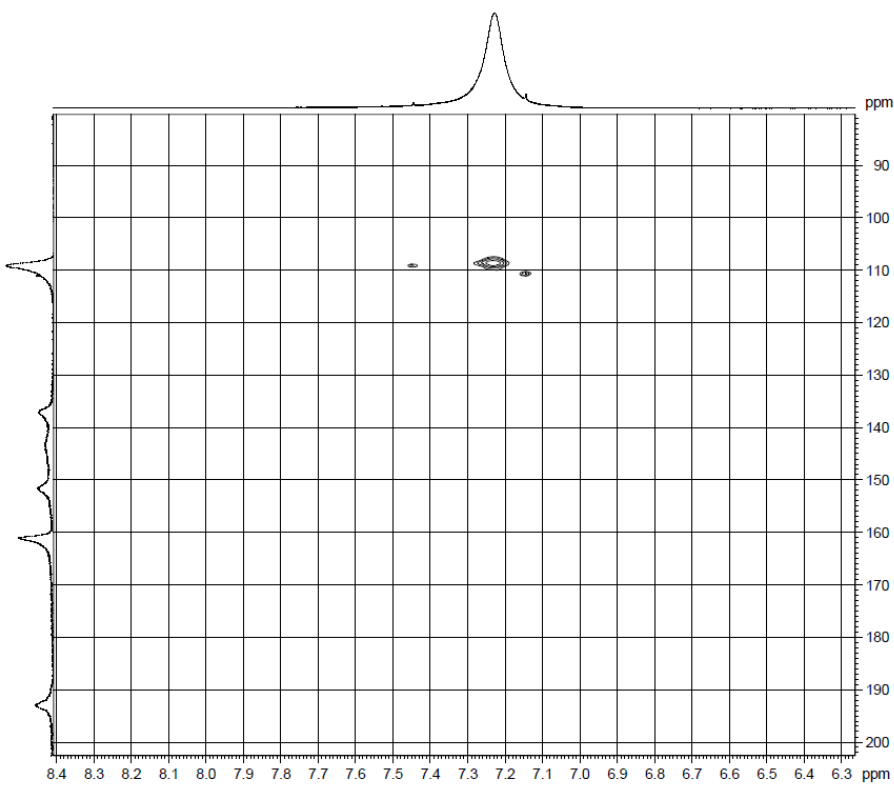


Standard 1H
136908
NAT0824_1
Nagy Tamas
2021.08.10. (KP)

Current Data Parameters
NAME 136908
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20210810
Time 21.24 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 196.07
DW 41.600 usec
DE 25.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 600.0537053 MHz
NUC1 1H
P1 11.50 usec
PLW1 28.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0499909 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00



HSQC (140 Hz)
140219
NAT0824_1
Poszavacz Laszlo
2022.12.20. (KP)

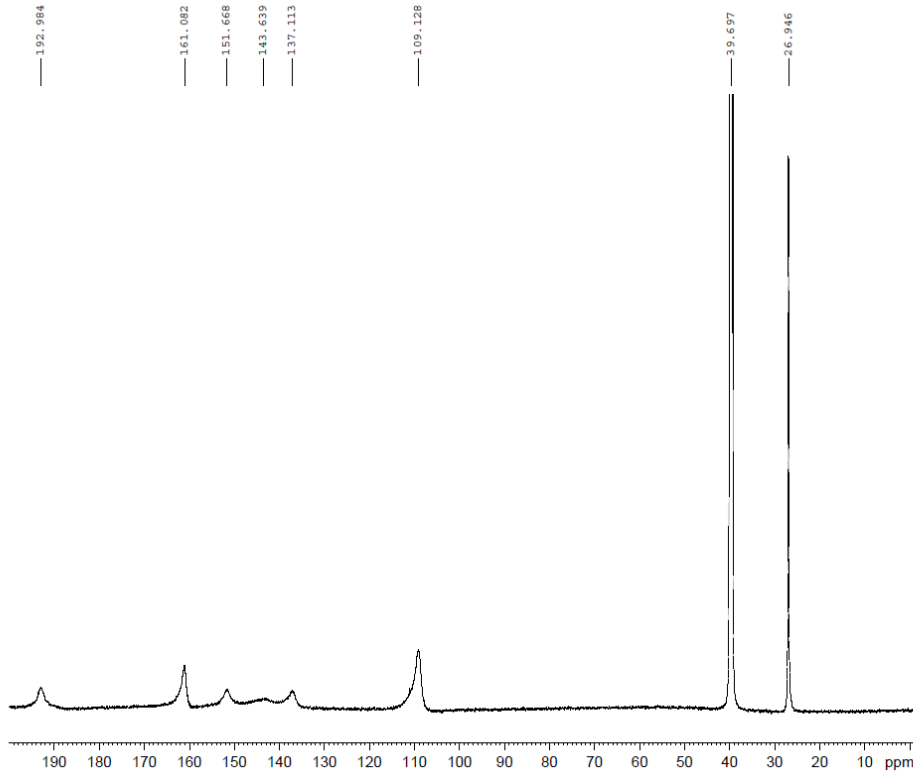
Current Data Parameters
NAME 140219
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20221220
Time 17.25 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG hsqc
TD 16384
SOLVENT DMSO
NS 33
DS 33
SWH 7853.530 Hz
FIDRES 18.258799 Hz
AQ 0.0053260 sec
RG 196.07
DW 64.000 usec
DE 25.00 usec
TE 295.0 K
D1 1.00000000 sec
D16 0.00000000 sec
D14 0.00178671 sec
D11 0.00000000 sec
D10 0.00000000 sec
D12 0.00000000 sec
D13 0.00000000 sec
TD0 1
SFO1 600.0537053 MHz
NUC1 1H
P1 11.50 usec
P2 21.00 usec
P2B 0 usec
SFO2 100.6281500 MHz
DE 25.00 usec
TE 295.0 K
P4 500.00 usec
P14 2000.00 usec
P43 1500.00 usec

PLAN0
PLAN1 70.48699951 W
PLAN2 2.31019994 W
SFO113 Cnp42,1,6,20,4
SFO114 Cnp42,1,6,20,4
SFO115 Cnp42,1,6,20,4
SFO116 Cnp42,1,6,20,4
SFO117 Cnp42,1,6,20,4
SFO118 Cnp42,1,6,20,4
SFO119 Cnp42,1,6,20,4
SFO120 Cnp42,1,6,20,4
SFO121 Cnp42,1,6,20,4
SFO122 Cnp42,1,6,20,4
SFO123 Cnp42,1,6,20,4
SFO124 Cnp42,1,6,20,4
SFO125 Cnp42,1,6,20,4
SFO126 Cnp42,1,6,20,4
SFO127 Cnp42,1,6,20,4
SFO128 Cnp42,1,6,20,4
SFO129 Cnp42,1,6,20,4
SFO130 Cnp42,1,6,20,4
SFO131 Cnp42,1,6,20,4
SFO132 Cnp42,1,6,20,4
SFO133 Cnp42,1,6,20,4
SFO134 Cnp42,1,6,20,4
SFO135 Cnp42,1,6,20,4
SFO136 Cnp42,1,6,20,4
SFO137 Cnp42,1,6,20,4
SFO138 Cnp42,1,6,20,4
SFO139 Cnp42,1,6,20,4
SFO140 Cnp42,1,6,20,4
SFO141 Cnp42,1,6,20,4
SFO142 Cnp42,1,6,20,4
SFO143 Cnp42,1,6,20,4
SFO144 Cnp42,1,6,20,4
SFO145 Cnp42,1,6,20,4
SFO146 Cnp42,1,6,20,4
SFO147 Cnp42,1,6,20,4
SFO148 Cnp42,1,6,20,4
SFO149 Cnp42,1,6,20,4
SFO150 Cnp42,1,6,20,4
SFO151 Cnp42,1,6,20,4
SFO152 Cnp42,1,6,20,4
SFO153 Cnp42,1,6,20,4
SFO154 Cnp42,1,6,20,4
SFO155 Cnp42,1,6,20,4
SFO156 Cnp42,1,6,20,4
SFO157 Cnp42,1,6,20,4
SFO158 Cnp42,1,6,20,4
SFO159 Cnp42,1,6,20,4
SFO160 Cnp42,1,6,20,4
SFO161 Cnp42,1,6,20,4
SFO162 Cnp42,1,6,20,4
SFO163 Cnp42,1,6,20,4
SFO164 Cnp42,1,6,20,4
SFO165 Cnp42,1,6,20,4
SFO166 Cnp42,1,6,20,4
SFO167 Cnp42,1,6,20,4
SFO168 Cnp42,1,6,20,4
SFO169 Cnp42,1,6,20,4
SFO170 Cnp42,1,6,20,4
SFO171 Cnp42,1,6,20,4
SFO172 Cnp42,1,6,20,4
SFO173 Cnp42,1,6,20,4
SFO174 Cnp42,1,6,20,4
SFO175 Cnp42,1,6,20,4
SFO176 Cnp42,1,6,20,4
SFO177 Cnp42,1,6,20,4
SFO178 Cnp42,1,6,20,4
SFO179 Cnp42,1,6,20,4
SFO180 Cnp42,1,6,20,4
SFO181 Cnp42,1,6,20,4
SFO182 Cnp42,1,6,20,4
SFO183 Cnp42,1,6,20,4
SFO184 Cnp42,1,6,20,4
SFO185 Cnp42,1,6,20,4
SFO186 Cnp42,1,6,20,4
SFO187 Cnp42,1,6,20,4
SFO188 Cnp42,1,6,20,4
SFO189 Cnp42,1,6,20,4
SFO190 Cnp42,1,6,20,4
SFO191 Cnp42,1,6,20,4
SFO192 Cnp42,1,6,20,4
SFO193 Cnp42,1,6,20,4
SFO194 Cnp42,1,6,20,4
SFO195 Cnp42,1,6,20,4
SFO196 Cnp42,1,6,20,4
SFO197 Cnp42,1,6,20,4
SFO198 Cnp42,1,6,20,4
SFO199 Cnp42,1,6,20,4
SFO200 Cnp42,1,6,20,4

F2 - Processing parameters
SI 16384
SF 600.0499909 MHz
WDW gq1m





Standard 13C
140219
NAT0824_1
PozzavaGz Laszlo
2022.12.20. (KP)

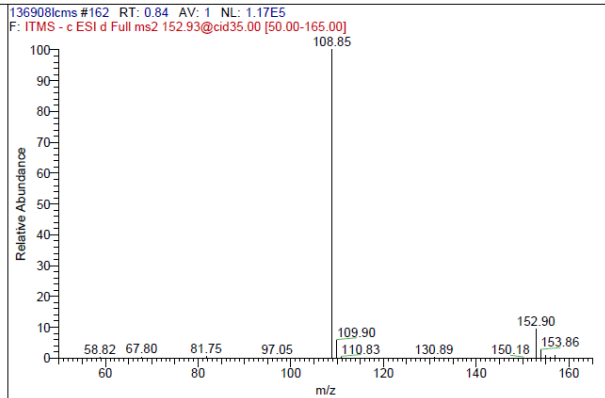
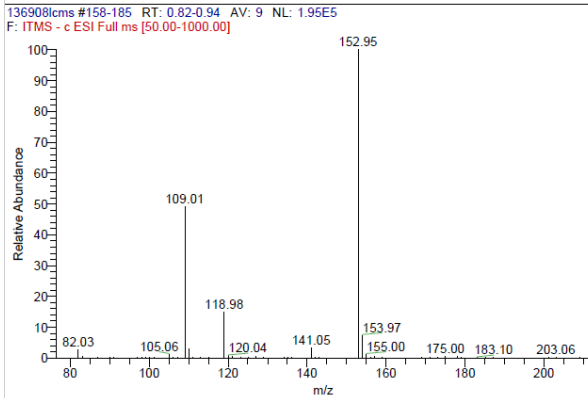
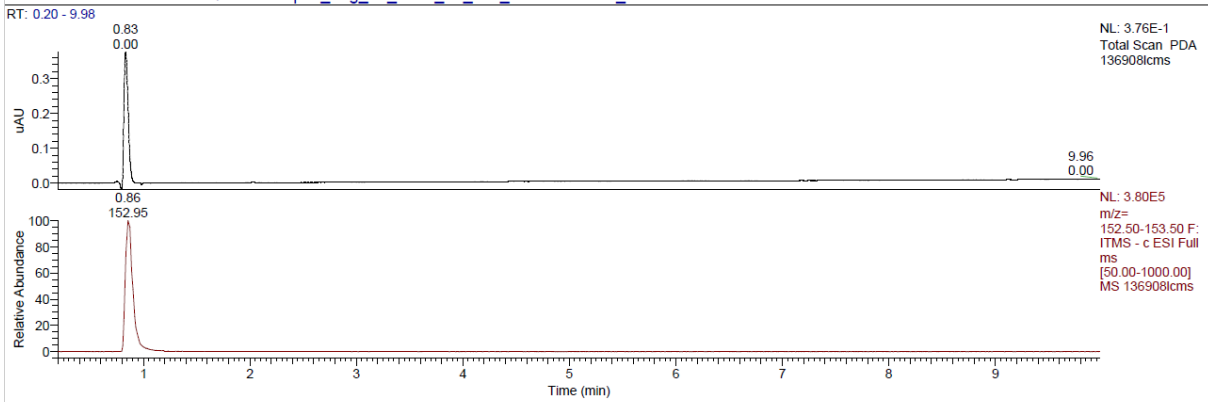
Current Data Parameters
NAME 140219
EXPRO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20221220
Time 17:23 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9043968 sec
RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8977808 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.48699951 W
SFO2 600.0524002 MHz
NUC2 1H
CPOPRG[2] waltz16
PCDD2 80.00 usec
PLW2 29.46299934 W
PLW12 0.66293001 W
PLW13 0.33292001 W

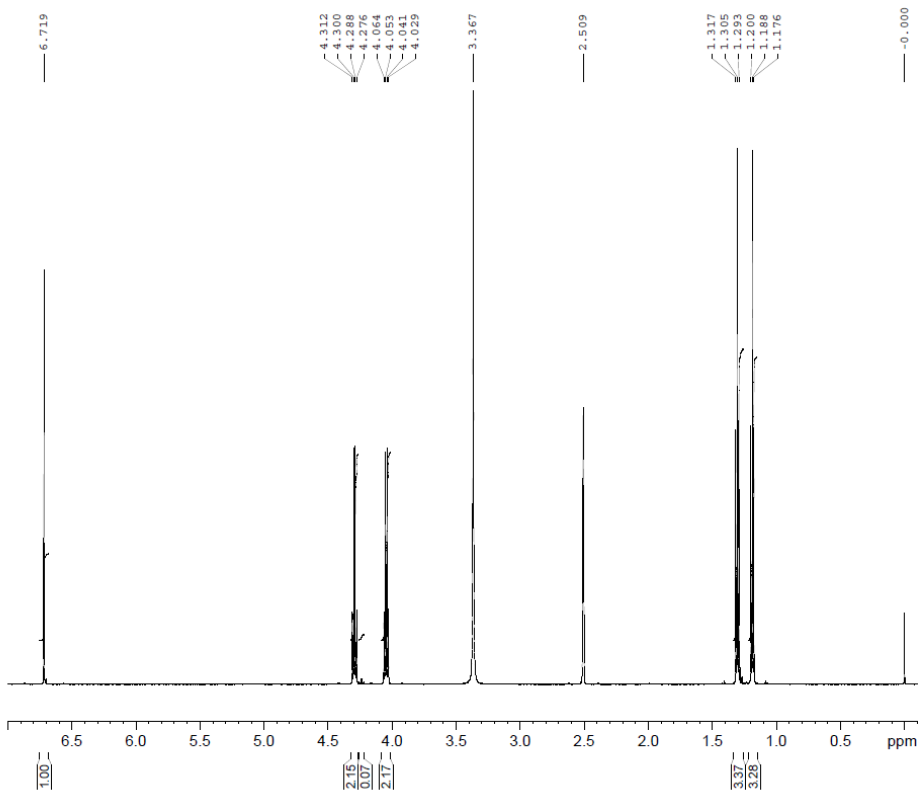
F2 - Processing parameters
SI 32768
SF 150.8827125 MHz
WDW EM
SSB 0
LB 6.00 Hz
GB 0
PC 1.40



C:\data\136908lcms 8/11/2021 9:36:38 AM
NAT0824_1, Nagy Tamas (MM)
C:\Thermo\Instruments\LTQ\methods\poz_neg_50_1000_10_min_CH3COONH4_c3.meth



2,5-Diethyl 4,6-dioxo-4H,5H,6H-pyrrolo[1,2-b]pyrazole-2,5-dicarboxylate (20)

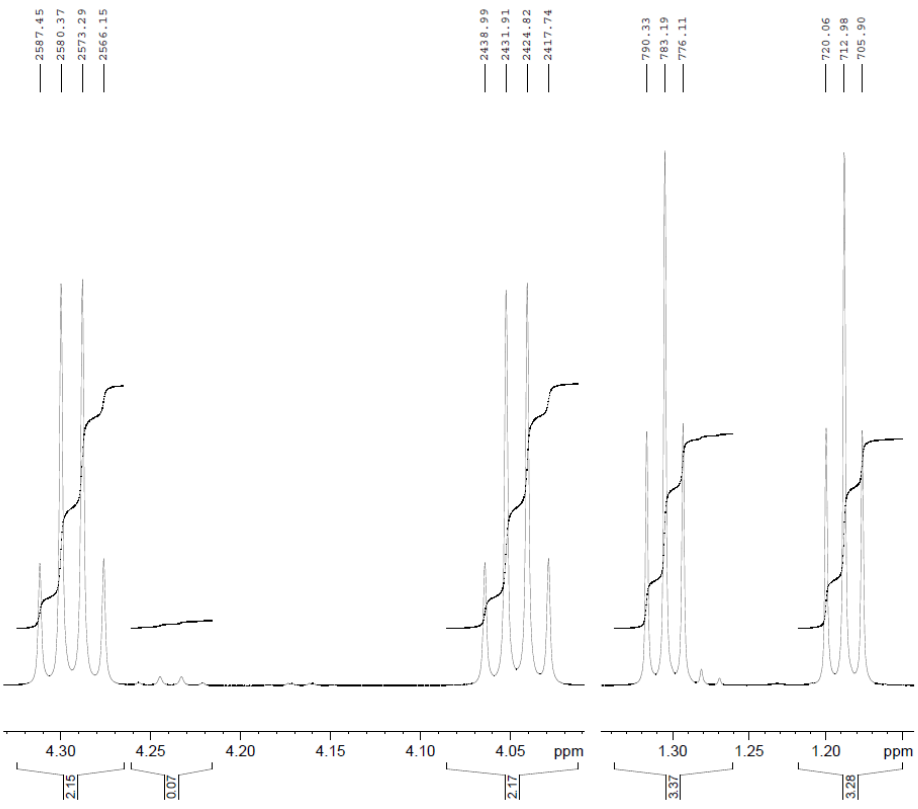


Standard 1H
130300
NAT0732_2
Nagy Tamas
2019.06.21. (KP)

Current Data Parameters
NAME 130300
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190621
Time_ 21.15 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 196.07
DW 41.600 usec
DE 25.00 usec
TE 295.0 K
D1 1.00000000 sec
TDO 1
SFO1 600.1037056 MHz
NUC1 1H
P1 11.50 usec
PLW1 28.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1000003 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



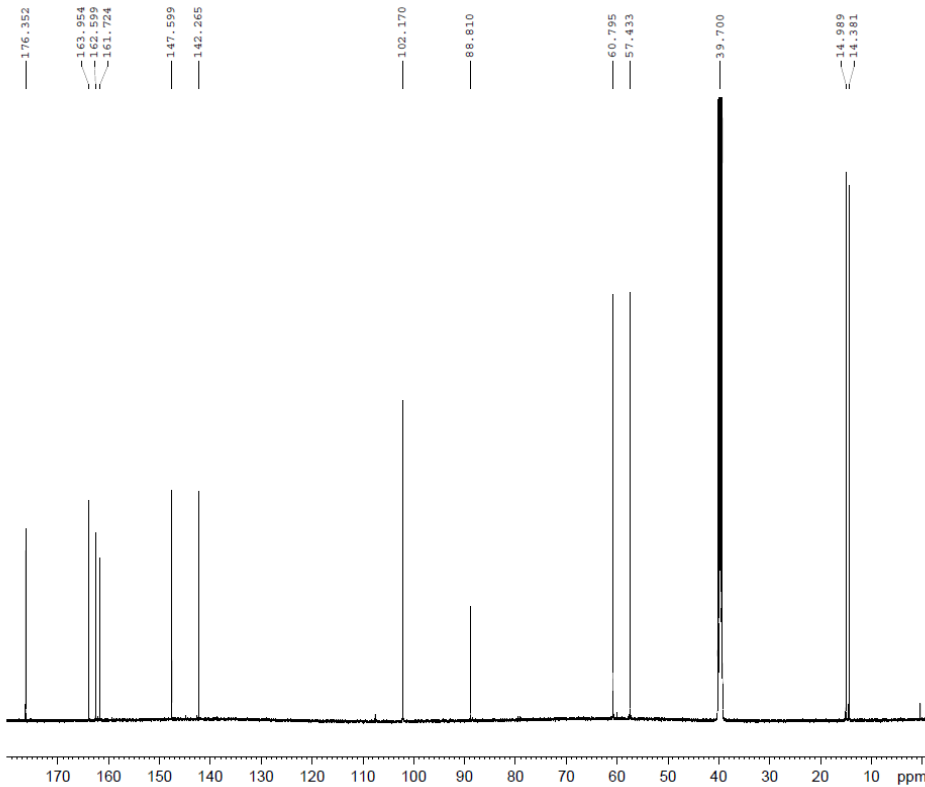
Standard 1H
130300
NAT0732_2
Nagy Tamas
2019.06.21. (KP)

Current Data Parameters
NAME 130300
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190621
Time_ 21.15 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 196.07
DW 41.600 usec
DE 25.00 usec
TE 295.0 K
D1 1.00000000 sec
TDO 1
SFO1 600.1037056 MHz
NUC1 1H
P1 11.50 usec
PLW1 28.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1000003 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



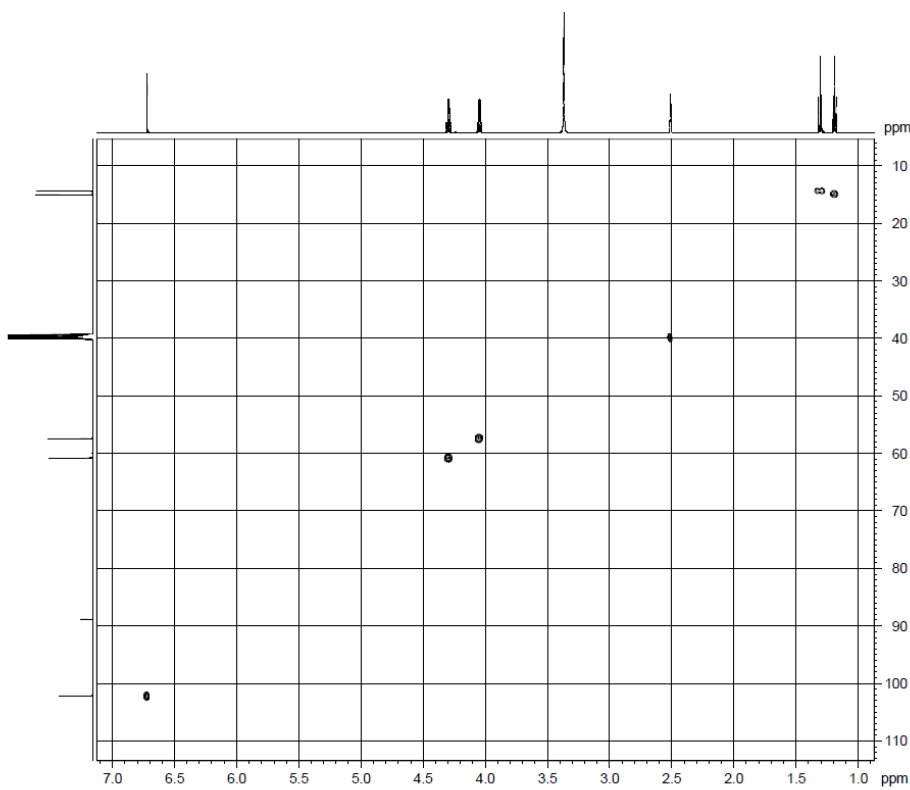


Standard 13C
130300
NAT0732_2
Nagy Tamas
2019.06.21. (KP)

Current Data Parameters
NAME 130300
EXFNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190621
Time_ 23:30 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zgpg30
TD 65536
SOLVENT EMSSO
NS 4096
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9043968 sec
RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.9103545 MHz
NUC1 13C
P1 10.00 usec
PLW1 63.07899857 W
SFO2 600.1024004 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 28.96599960 W
PLW12 0.65174001 W
PLW13 0.32730001 W

F2 - Processing parameters
SI 32768
SF 150.8953023 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HSQC (140Hz)
130300
NAT0732_2
Nagy Tamas
2019.06.21. (KP)

Current Data Parameters
NAME 130300
EXFNO 14
PROCNO 1

F2 - Acquisition Parameters
Date_ 20190621
Time_ 23:52 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG hsqc02
TD 65536
SOLVENT EMSSO
NS 2
DS 16
SWH 7812.1000 Hz
FIDRES 7.620390 Hz
AQ 0.1310720 sec
RG 64.07
DW 136.07 usec
DE 26.00 usec
TE 295.0 K
CHFT2 140.000000 sec
DD 0.00000000 sec
D1 1.00000000 sec
D4 0.00178171 sec
D11 0.01000000 sec
D14 0.00020000 sec
D24 0.00080207 sec
IN0 0.00001111 sec
TD0 1
SFO1 600.1038007 MHz
NUC1 1H
P1 11.00 usec
P2 0 usec
P2B 0 usec
PLW1 28.90000000 W
SFO2 150.9103545 MHz
NUC2 13C
CPDPRG2 hsqc02
P4 10.00 usec
P4B 20.00 usec
PLW2 63.07899857 W
PLW12 2.08529999 W
CHNAM(1) SMCQ1-100
CHNAM(2) SMCQ1-100
CHNAM(3) SMCQ1-100
CHNAM(4) SMCQ1-100
P14 1.00 usec
P14B 1000.00 usec
P19 600.00 usec

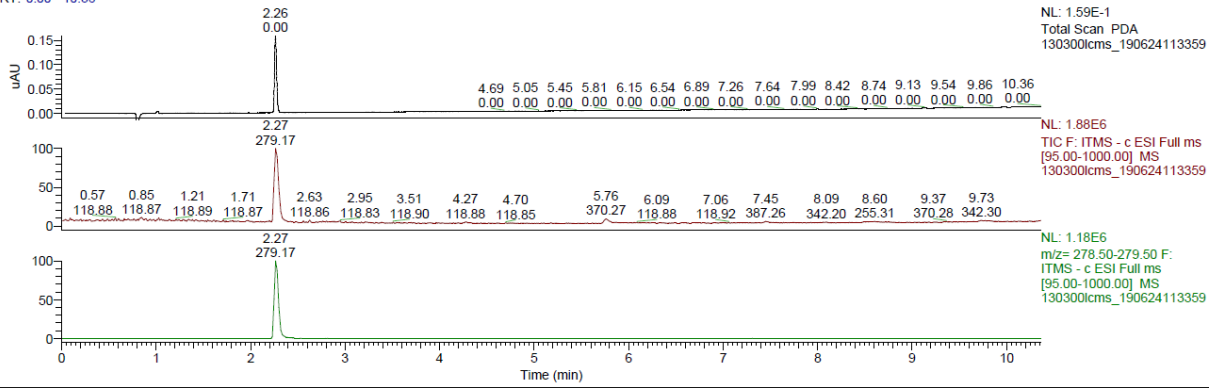
F1 - Acquisition parameters
TD 256
SFO1 150.9103 MHz
FIDRES 218.62047 Hz
SW 219.441 PPM
FPMODE Echo-AntiEcho

F2 - Processing parameters
SI 1024
SF 150.8953023 MHz
WDW GETHS
SSB 2
LB 0 Hz
GB 0
PC 1.40

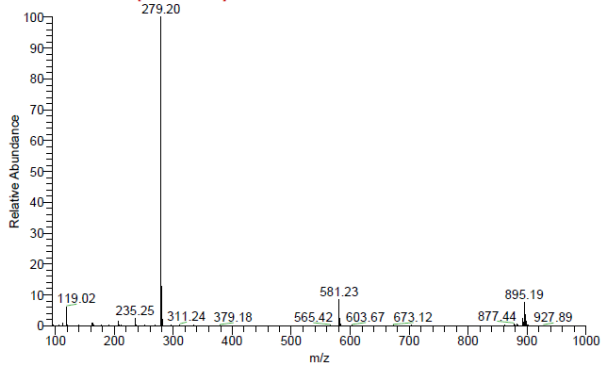
F1 - Processing parameters
SI 1024
MC2 echo-anti-echo
SF 150.8953023 MHz
WDW GETHS
SSB 2
LB 0 Hz
GB 0



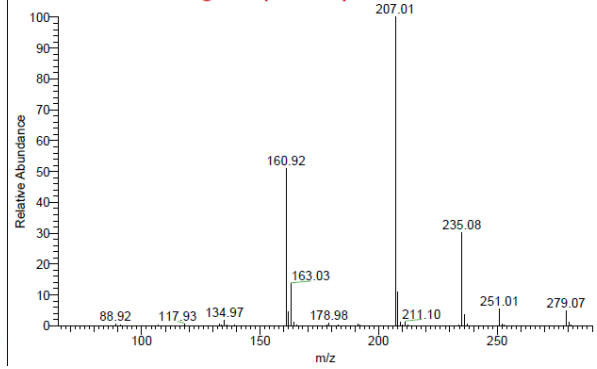
RT: 0.00 - 10.36



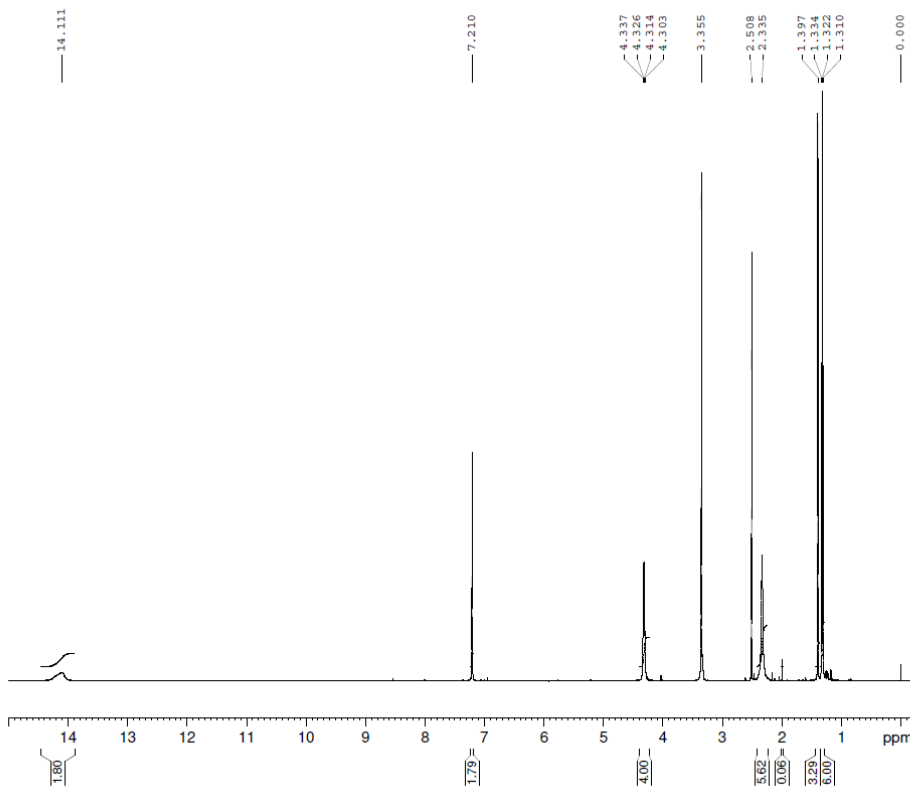
130300lcms_190624113359 #443-454 RT: 2.27-2.30 AV: 3 NL: 8.92E5
F: ITMS - c ESI Full ms [95.00-1000.00]



130300lcms_190624113359 #439-455 RT: 2.24-2.25 AV: 2 NL: 6.95E4
F: ITMS - c ESI d Full ms2 279.13@cid35.00 [65.00-290.00]



Diethyl 5,5'-[(1E/Z,1'E/Z)-hydrazine-1,2-diylidenebis(ethan-1-yl-1-ylidene)]bis(1H-pyrazole-3-carboxylate) (9)

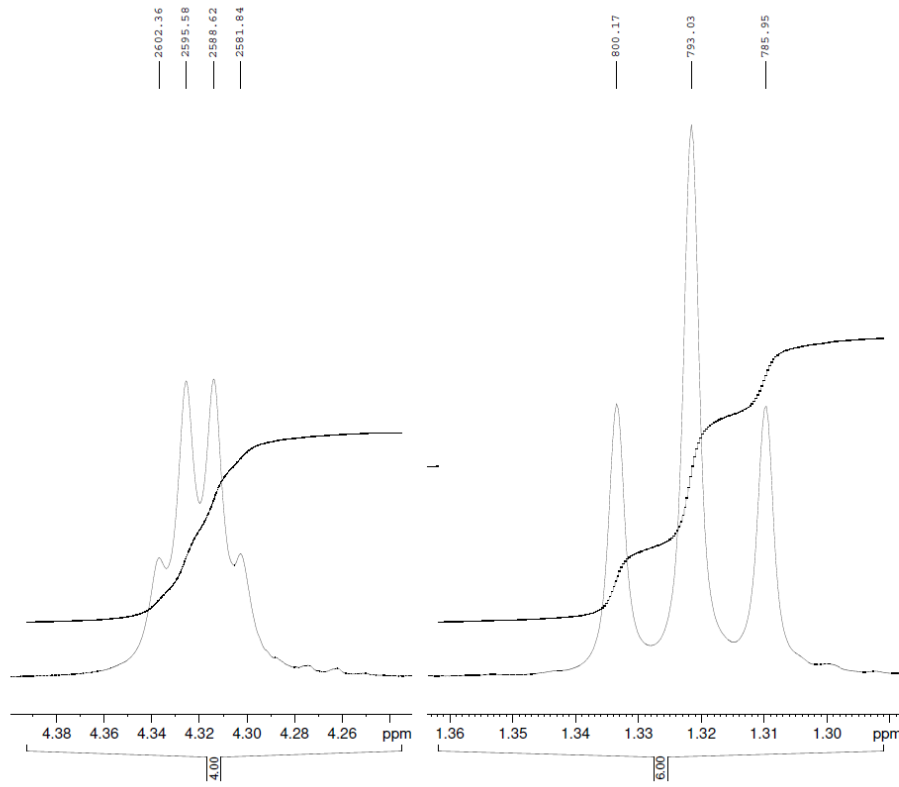


Standard 1H
 139534
 POL1489_2
 PoszavaCz Laszlo
 2022.09.08. (KP)

Current Data Parameters
 NAME 139534
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20220909
 Time 1.57 h
 INSTRUM spect
 PROBHD Z145856_0002 (
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.366798 Hz
 AQ 2.7262976 sec
 RG 174.19
 DW 41.600 usec
 DE 25.00 usec
 TE 295.0 K
 D1 1.00000000 sec
 TD0 1
 SFO1 600.0537053 MHz
 NUC1 1H
 P1 11.50 usec
 PLW1 28.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.0500000 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



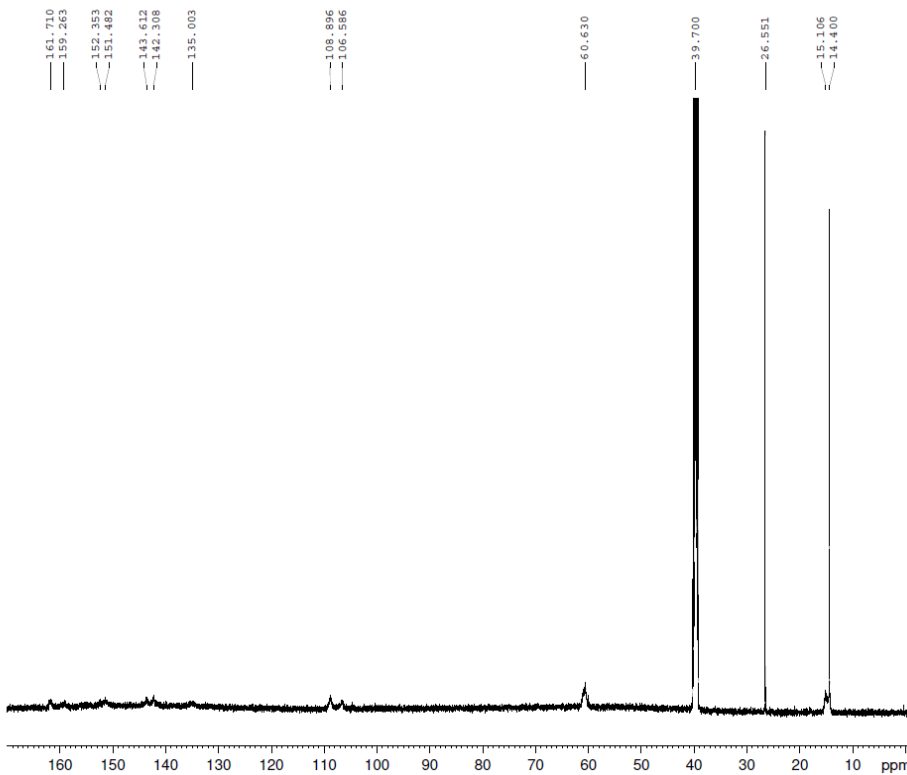
Standard 1H
 139534
 POL1489_2
 PoszavaCz Laszlo
 2022.09.08. (KP)

Current Data Parameters
 NAME 139534
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20220909
 Time 1.57 h
 INSTRUM spect
 PROBHD Z145856_0002 (
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.366798 Hz
 AQ 2.7262976 sec
 RG 174.19
 DW 41.600 usec
 DE 25.00 usec
 TE 295.0 K
 D1 1.00000000 sec
 TD0 1
 SFO1 600.0537053 MHz
 NUC1 1H
 P1 11.50 usec
 PLW1 28.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.0500000 MHz
 WDW no
 SSB 0
 LB 0 Hz
 GB 0
 PC 1.00



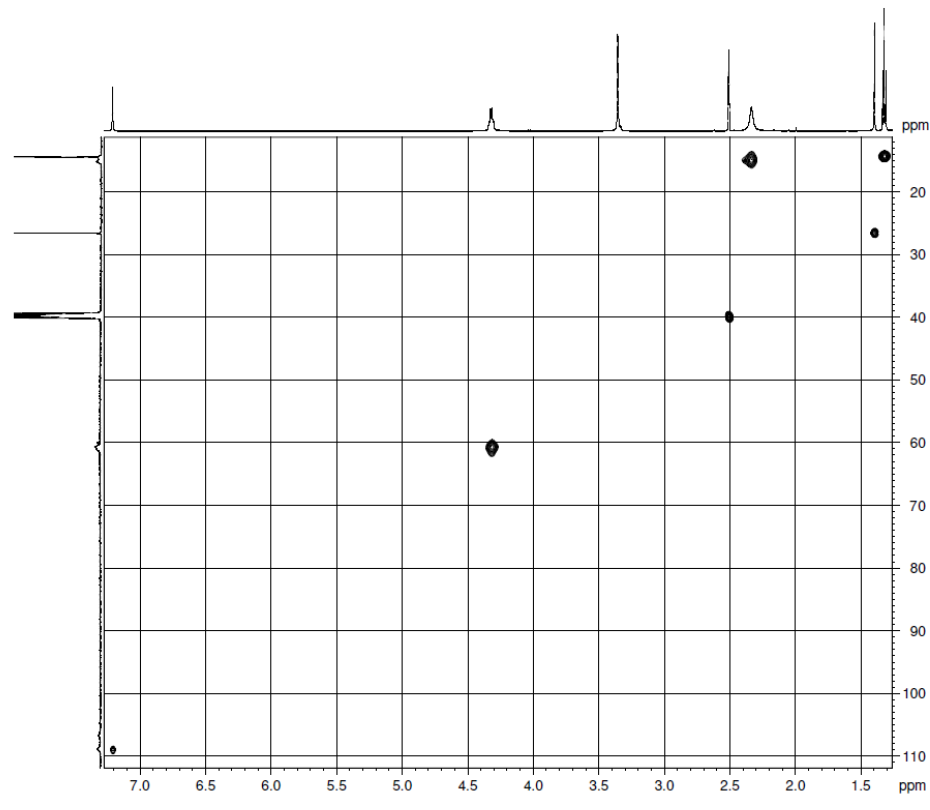


Standard 13C
139534
POL1489_2
Poszavacz Laszlo
2022.09.08.(KP)

Current Data Parameters
NAME 139534
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220909
Time 3.05 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2048
DS 4
SWH 36231.893 Hz
FIDRES 1.105709 Hz
AQ 0.9043968 sec
RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.0000000 sec
D11 0.0300000 sec
TD0 1
SFO1 150.8977808 MHz
NUC1 13C
P1 10.00 usec
PLW1 70.48699951 W
SFO2 600.0524002 MHz
NUC2 1H
CPDPRG12 waltz16
PCPD2 80.00 usec
PLW2 29.46299934 W
PLW12 0.66293001 W
PLW13 0.33292001 W

F2 - Processing parameters
SI 32768
SF 150.8827308 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HSQC (140 Hz)
139534
POL1489_2
Poszavacz Laszlo
2022.09.08.(KP)

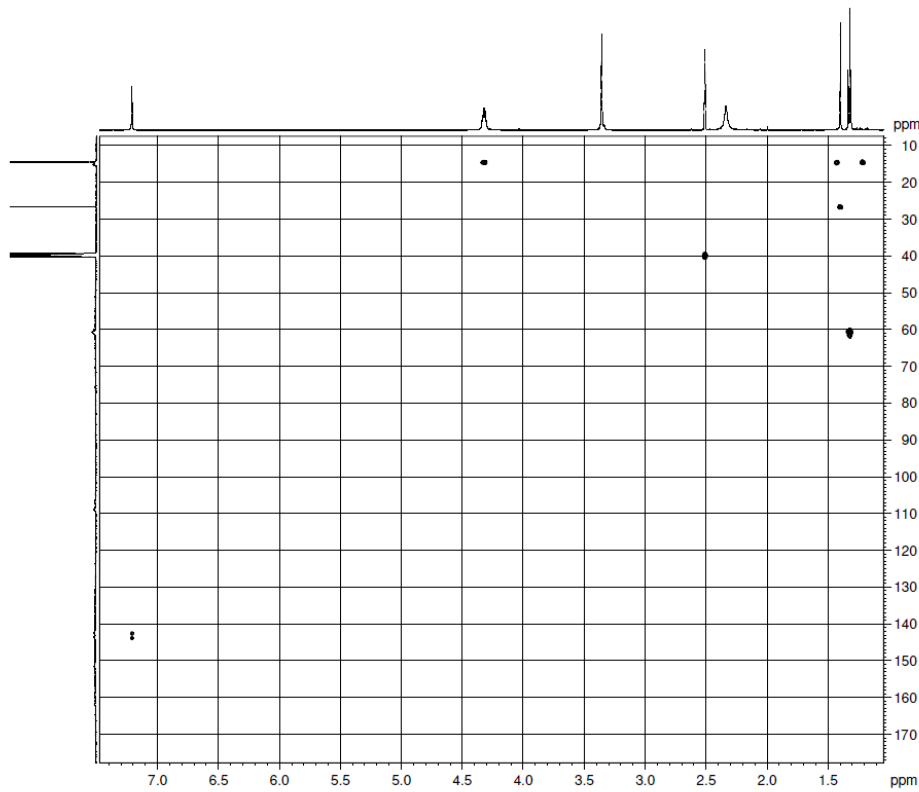
Current Data Parameters
NAME 139534
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220909
Time 3.27 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG hsqcetpsip02_2
TD 1324
SOLVENT DMSO
NS 4
DS 4
SWH 7812.600 Hz
FIDRES 16.218790 Hz
AQ 0.9453340 sec
RG 196.07
DW 44.000 usec
DE 26.00 usec
TE 295.0 K
D1 140.000000 sec
D11 0.0000000 sec
D12 1.0000000 sec
D13 0.0000000 sec
D14 0.0000000 sec
D15 0.0000000 sec
D16 0.0000000 sec
D17 0.0000000 sec
D18 0.0000000 sec
D19 0.0000000 sec
D20 0.0000000 sec
D21 0.0000000 sec
D22 0.0000000 sec
D23 0.0000000 sec
D24 0.0000000 sec
D25 0.0000000 sec
D26 0.0000000 sec
D27 0.0000000 sec
D28 0.0000000 sec
D29 0.0000000 sec
D30 0.0000000 sec
D31 0.0000000 sec
D32 0.0000000 sec
D33 0.0000000 sec
D34 0.0000000 sec
D35 0.0000000 sec
D36 0.0000000 sec
D37 0.0000000 sec
D38 0.0000000 sec
D39 0.0000000 sec
D40 0.0000000 sec
D41 0.0000000 sec
D42 0.0000000 sec
D43 0.0000000 sec
D44 0.0000000 sec
D45 0.0000000 sec
D46 0.0000000 sec
D47 0.0000000 sec
D48 0.0000000 sec
D49 0.0000000 sec
D50 0.0000000 sec
D51 0.0000000 sec
D52 0.0000000 sec
D53 0.0000000 sec
D54 0.0000000 sec
D55 0.0000000 sec
D56 0.0000000 sec
D57 0.0000000 sec
D58 0.0000000 sec
D59 0.0000000 sec
D60 0.0000000 sec
D61 0.0000000 sec
D62 0.0000000 sec
D63 0.0000000 sec
D64 0.0000000 sec
D65 0.0000000 sec
D66 0.0000000 sec
D67 0.0000000 sec
D68 0.0000000 sec
D69 0.0000000 sec
D70 0.0000000 sec
D71 0.0000000 sec
D72 0.0000000 sec
D73 0.0000000 sec
D74 0.0000000 sec
D75 0.0000000 sec
D76 0.0000000 sec
D77 0.0000000 sec
D78 0.0000000 sec
D79 0.0000000 sec
D80 0.0000000 sec
D81 0.0000000 sec
D82 0.0000000 sec
D83 0.0000000 sec
D84 0.0000000 sec
D85 0.0000000 sec
D86 0.0000000 sec
D87 0.0000000 sec
D88 0.0000000 sec
D89 0.0000000 sec
D90 0.0000000 sec
D91 0.0000000 sec
D92 0.0000000 sec
D93 0.0000000 sec
D94 0.0000000 sec
D95 0.0000000 sec
D96 0.0000000 sec
D97 0.0000000 sec
D98 0.0000000 sec
D99 0.0000000 sec
D100 0.0000000 sec

F2 - Acquisition parameters
TD 1324
SF 150.8827308 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

F2 - Processing parameters
SI 32768
SF 150.8827308 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





HMBC (8Hz, 140 Hz)
139534
POL1489_2
PoszavaCz Laszlo
2022.09.08. (KP)

```

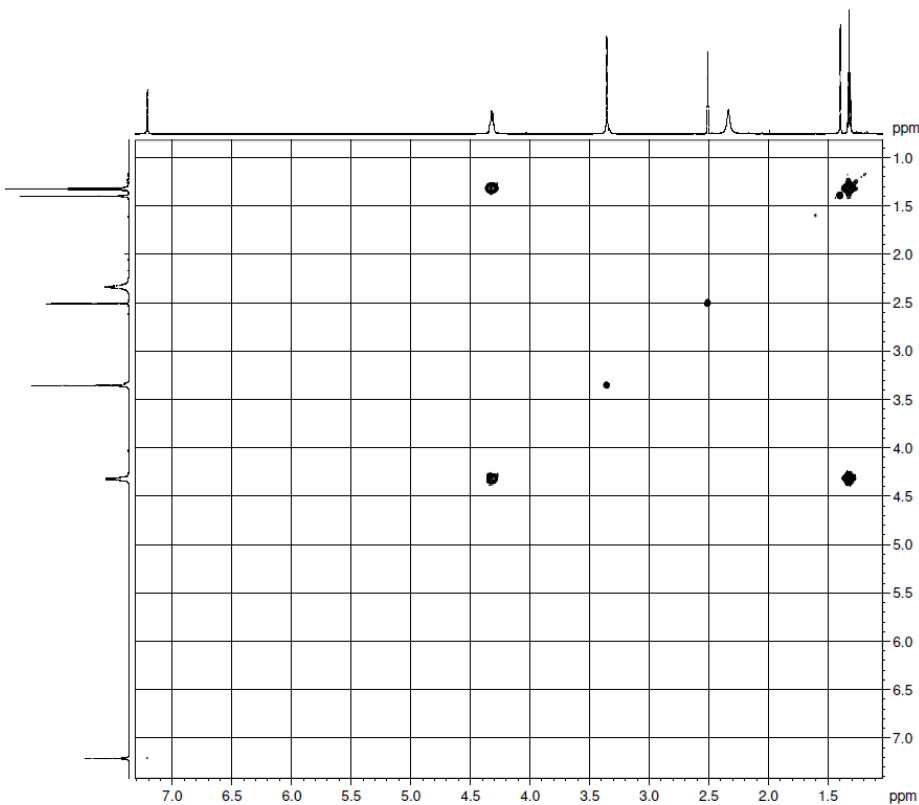
Current Data Parameters
NAME      139534
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20220909
TIME    3.14 h
INSTRUM spect
PROBHD  z14886_002 |
PULPROG mbogp1pwrfr
TD      65536
SOLVENT DMG0
NS      2
DS      4
SWH     7812.100 Hz
FIDRES 7.422996 Hz
AQ      0.1310720 sec
RG      196.07
RW      64.000 usec
DE      25.00 usec
TE      296.0 K
CNS222  140.0000000
CNS113  8.0000000
D1      0.0000000 sec
D2      1.1000000 sec
D3      0.0000000 sec
D14     0.0000000 sec
D16     0.0000000 sec
IND     0.0000151 sec
TNAV    2
SFO1    600.0537813 MHz
NUC1    13
P1      11.50 usec
P2      21.00 usec
P3      28.0000000 usec
P4      150.8981312 MHz
NUC4    13
P5      10.00 usec
P6      70.4480903 MHz
CPDPRG11 SMO10_100
CPDPRG12 SMO10_100
CPDPRG13 SMO10_100
CPDPRG14 SMO10_100
CPDPRG15 SMO10_100
P16     1000.00 usec

F1 - Acquisition parameters
TD      316
SFO1    600.0537813 MHz
F1RES   7.422996 Hz
SW       219.426 ppm
FWDKRES 0
PC       1.40

F2 - Processing parameters
SI      600.0500000 MHz
SF      600.0500000 MHz
WDW     SINC
SSB     0
LB      0 Hz
GB      0
PC      1.40

F1 - Processing parameters
SI      600.0500000 MHz
SF      600.0500000 MHz
WDW     SINC
SSB     0
LB      0 Hz
GB      0
PC      1.40
  
```



COSY
139534
POL1489_2
PoszavaCz Laszlo
2022.09.08. (KP)

```

Current Data Parameters
NAME      139534
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20220909
TIME    3.07 h
INSTRUM spect
PROBHD  z14886_002 |
PULPROG cosygprfr
TD      65536
SOLVENT DMG0
NS      2
DS      4
SWH     7812.100 Hz
FIDRES 7.422996 Hz
AQ      0.1310720 sec
RG      196.07
RW      64.000 usec
DE      25.00 usec
TE      296.0 K
D0      0.0000000 sec
D1      2.0000000 sec
D13     0.0000000 sec
D14     0.0000000 sec
D16     0.0001280 sec
IND     0.0001280 sec
TNAV    2
SFO1    600.0536003 MHz
NUC1    13
P1      11.50 usec
P2      28.0000000 usec
P3      150.8981312 MHz
CPDPRG11 SMO10_100
CPDPRG12 SMO10_100
CPDPRG13 SMO10_100
CPDPRG14 SMO10_100
CPDPRG15 SMO10_100
P16     1000.00 usec

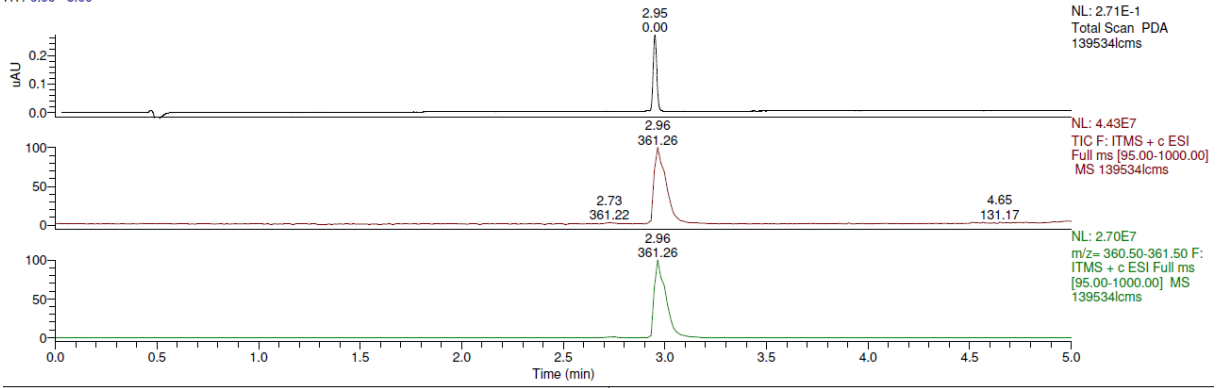
F1 - Acquisition parameters
TD      316
SFO1    600.0536003 MHz
F1RES   7.422996 Hz
SW       219.426 ppm
FWDKRES 0
PC       1.40

F2 - Processing parameters
SI      600.0500000 MHz
SF      600.0500000 MHz
WDW     SINC
SSB     0
LB      0 Hz
GB      0
PC      1.40

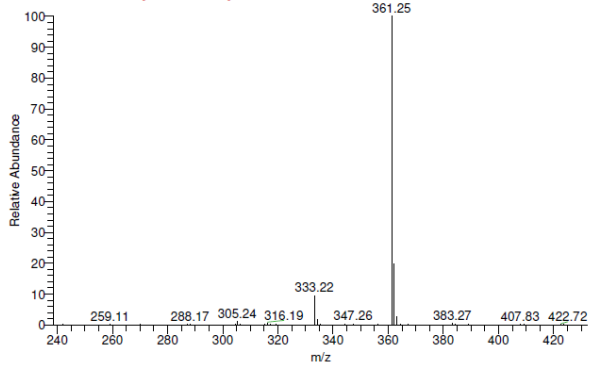
F1 - Processing parameters
SI      600.0500000 MHz
SF      600.0500000 MHz
WDW     SINC
SSB     0
LB      0 Hz
GB      0
PC      1.40
  
```



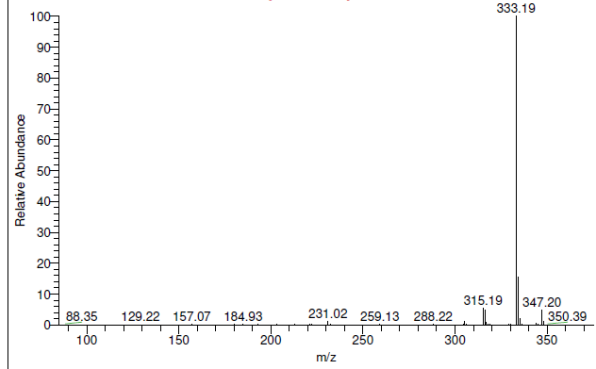
D:\data\139534cms 9/20/2022 1:11:20 PM
POL1489_2_Poszavacz László (MM)
D:\Thermo_LTQ\methods\poz_neg_95_1000_5min_HCOOH_c2_20220121.meth
RT: 0.00 - 5.00



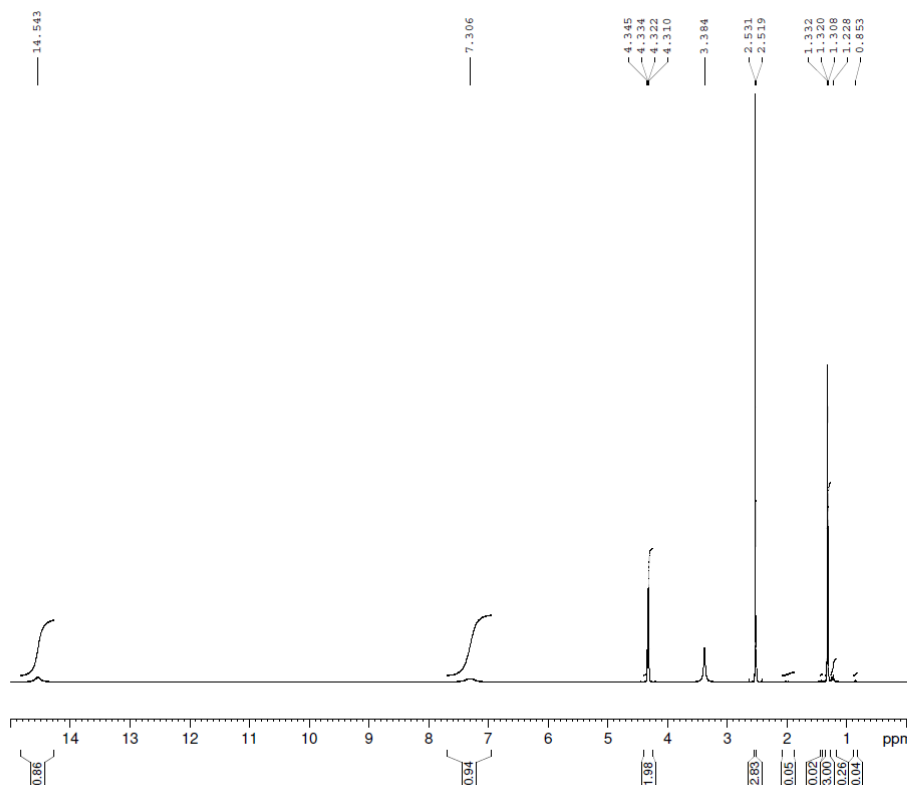
139534cms #613-635 RT: 2.95-3.03 AV: 6 SB: 98 1.38-1.76, 2.49-3.16 NL: 1.57E7
F: ITMS + c ESI Full ms [95.00-1000.00]



139534cms #614 RT: 2.95 AV: 1 NL: 2.16E7
F: ITMS + c ESI d Full ms2 361.21@cid35.00 [85.00-375.00]



5-Acetyl-1H-pyrazole-3-carboxylic acid ethyl ester (8a)

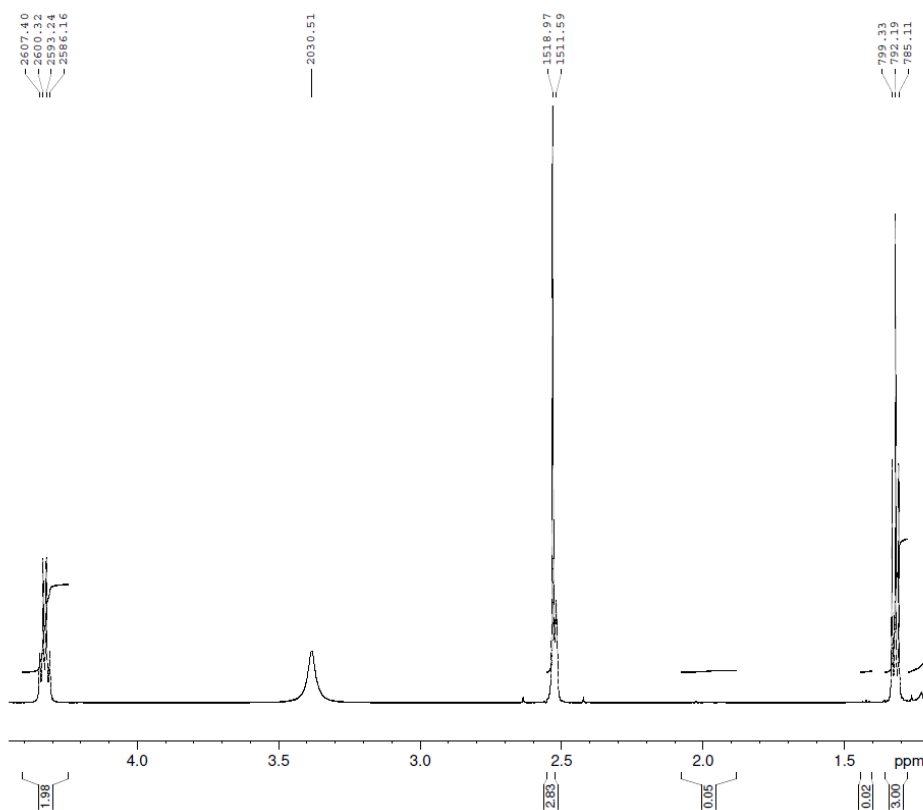


Standard 1H
139609
POL1490_1
PoszawaCz Laszlo
2022.09.16. (KP)

Current Data Parameters
NAME 139609
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220916
Time 16.03 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 111.4
DW 41.600 usec
DE 25.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 600.0537053 MHz
NUC1 1H
P1 11.50 usec
PLW1 28.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0499934 MHz
WDW EM
SSB 0
LB 0.40 Hz
GB 0
PC 1.00



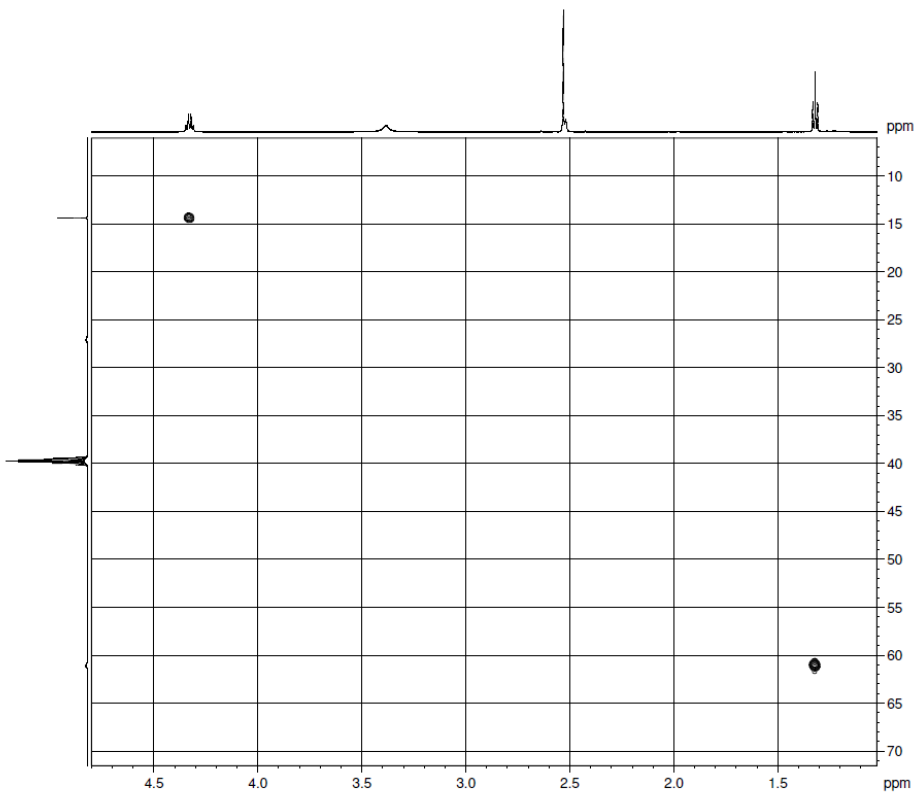
Standard 1H
139609
POL1490_1
PoszawaCz Laszlo
2022.09.16. (KP)

Current Data Parameters
NAME 139609
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20220916
Time 16.03 h
INSTRUM spect
PROBHD Z145856_0002 (
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.366798 Hz
AQ 2.7262976 sec
RG 111.4
DW 41.600 usec
DE 25.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
SFO1 600.0537053 MHz
NUC1 1H
P1 11.50 usec
PLW1 28.00000000 W

F2 - Processing parameters
SI 65536
SF 600.0499934 MHz
WDW EM
SSB 0
LB 0.40 Hz
GB 0
PC 1.00





HMBC (8Hz, 140Hz)
139609
POL1490_1
Pozzavacz Laszlo
2022.09.16. (KP)

```

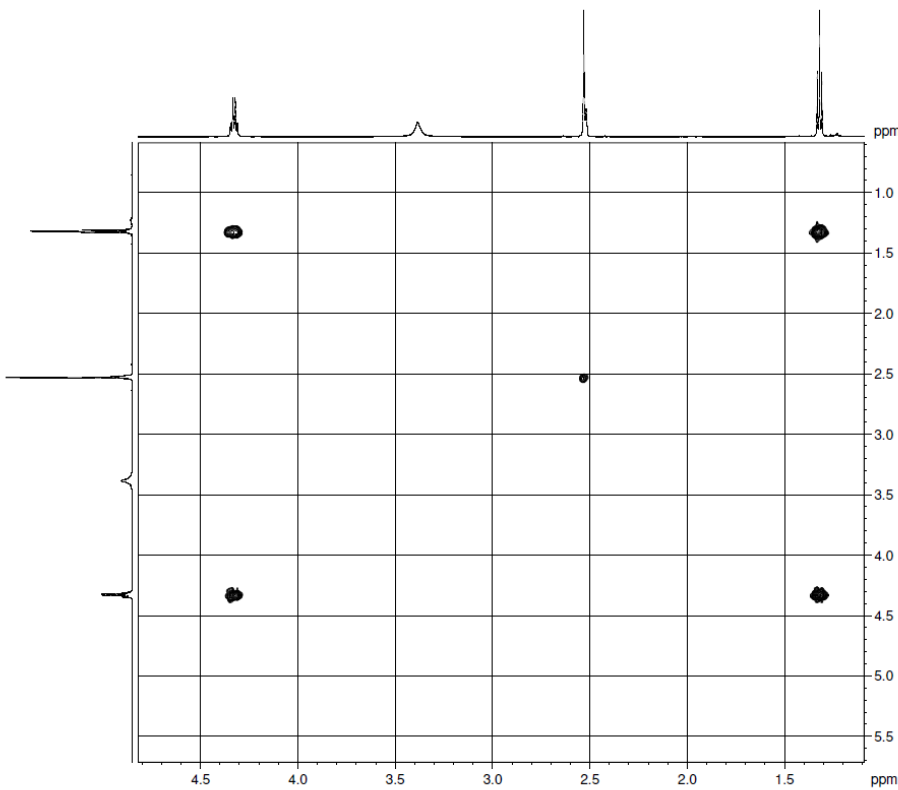
Current Data Parameters
NAME      139609
EXPNO     11
PROCNO    1

F2 - Acquisition Parameters
Date_     20220916
Time      18.21 s
INSTRUM   spect
PROBHD    z14586_0002_1
PULPROG   hmcoppgp2pr
TD         65536
SOLVENT   DMSO
NS         4
DS         2
SWH        7812.500 Hz
FIDRES     7.629395 Hz
AQ         0.1310710 sec
RG         194.07
DM         64.000 usec
DE         26.00 usec
TE         295.0 K
CMT01     140.000000 sec
CMT02     8.000000 sec
D1         1.0000000 sec
D11        0.0000000 sec
D12        0.0000000 sec
D13        0.0000000 sec
D14        0.0000000 sec
IR0        0.0000150 sec
TSDV
SFO1       600.5537803 MHz
NUC1        13C
P1          11.10 usec
PL1         23.00 usec
PL12        28.00000000
SFO2        150.8827271 MHz
NUC2         13C
P2          10.00 usec
PL2         70.88209911 W
CPRM(1)    SMDQ15.100
CPRM(2)    SMDQ15.100
CPRM(3)    SMDQ15.100
CPRM(4)    SMDQ15.100
CPRM(5)    SMDQ15.100
CPRM(6)    SMDQ15.100
CPRM(7)    SMDQ15.100
CPRM(8)    SMDQ15.100
CPRM(9)    SMDQ15.100
CPRM(10)   SMDQ15.100
CPRM(11)   SMDQ15.100
CPRM(12)   SMDQ15.100
CPRM(13)   SMDQ15.100
CPRM(14)   SMDQ15.100
CPRM(15)   SMDQ15.100
CPRM(16)   SMDQ15.100
CPRM(17)   SMDQ15.100
CPRM(18)   SMDQ15.100
CPRM(19)   SMDQ15.100
CPRM(20)   SMDQ15.100
CPRM(21)   SMDQ15.100
CPRM(22)   SMDQ15.100
CPRM(23)   SMDQ15.100
CPRM(24)   SMDQ15.100
CPRM(25)   SMDQ15.100
CPRM(26)   SMDQ15.100
CPRM(27)   SMDQ15.100
CPRM(28)   SMDQ15.100
CPRM(29)   SMDQ15.100
CPRM(30)   SMDQ15.100
CPRM(31)   SMDQ15.100
CPRM(32)   SMDQ15.100
CPRM(33)   SMDQ15.100
CPRM(34)   SMDQ15.100
CPRM(35)   SMDQ15.100
CPRM(36)   SMDQ15.100
CPRM(37)   SMDQ15.100
CPRM(38)   SMDQ15.100
CPRM(39)   SMDQ15.100
CPRM(40)   SMDQ15.100
CPRM(41)   SMDQ15.100
CPRM(42)   SMDQ15.100
CPRM(43)   SMDQ15.100
CPRM(44)   SMDQ15.100
CPRM(45)   SMDQ15.100
CPRM(46)   SMDQ15.100
CPRM(47)   SMDQ15.100
CPRM(48)   SMDQ15.100
CPRM(49)   SMDQ15.100
CPRM(50)   SMDQ15.100
CPRM(51)   SMDQ15.100
CPRM(52)   SMDQ15.100
CPRM(53)   SMDQ15.100
CPRM(54)   SMDQ15.100
CPRM(55)   SMDQ15.100
CPRM(56)   SMDQ15.100
CPRM(57)   SMDQ15.100
CPRM(58)   SMDQ15.100
CPRM(59)   SMDQ15.100
CPRM(60)   SMDQ15.100
CPRM(61)   SMDQ15.100
CPRM(62)   SMDQ15.100
CPRM(63)   SMDQ15.100
CPRM(64)   SMDQ15.100
CPRM(65)   SMDQ15.100
CPRM(66)   SMDQ15.100
CPRM(67)   SMDQ15.100
CPRM(68)   SMDQ15.100
CPRM(69)   SMDQ15.100
CPRM(70)   SMDQ15.100
CPRM(71)   SMDQ15.100
CPRM(72)   SMDQ15.100
CPRM(73)   SMDQ15.100
CPRM(74)   SMDQ15.100
CPRM(75)   SMDQ15.100
CPRM(76)   SMDQ15.100
CPRM(77)   SMDQ15.100
CPRM(78)   SMDQ15.100
CPRM(79)   SMDQ15.100
CPRM(80)   SMDQ15.100
CPRM(81)   SMDQ15.100
CPRM(82)   SMDQ15.100
CPRM(83)   SMDQ15.100
CPRM(84)   SMDQ15.100
CPRM(85)   SMDQ15.100
CPRM(86)   SMDQ15.100
CPRM(87)   SMDQ15.100
CPRM(88)   SMDQ15.100
CPRM(89)   SMDQ15.100
CPRM(90)   SMDQ15.100
CPRM(91)   SMDQ15.100
CPRM(92)   SMDQ15.100
CPRM(93)   SMDQ15.100
CPRM(94)   SMDQ15.100
CPRM(95)   SMDQ15.100
CPRM(96)   SMDQ15.100
CPRM(97)   SMDQ15.100
CPRM(98)   SMDQ15.100
CPRM(99)   SMDQ15.100
CPRM(100)  SMDQ15.100
F1 - Acquisition parameters
TD         256
SFO1       150.8888 MHz
FIDRES     218.182047 Hz
RG         219.454 ppm
PULPROG    cp
PCMDR      CP

F2 - Processing parameters
SI         256
SF         600.0499234 MHz
WDW        EM
SSB         0 Hz
GB          0 Hz
DC          0
SC          1.40

F1 - Processing parameters
SI         1024
SF         150.8827271 MHz
WDW        EM
SSB         0 Hz
GB          0 Hz
DC          0
SC          1.40

```



COSY
139609
POL1490_1
Pozzavacz Laszlo
2022.09.16. (KP)

```

Current Data Parameters
NAME      139609
EXPNO     12
PROCNO    1

F2 - Acquisition Parameters
Date_     20220916
Time      19.13 s
INSTRUM   spect
PROBHD    z14586_0002_1
PULPROG   cosygpgpr2
TD         65536
SOLVENT   DMSO
NS         2
DS         2
SWH        7812.500 Hz
FIDRES     7.629395 Hz
AQ         0.1310710 sec
RG         194.07
DM         64.000 usec
DE         26.00 usec
TE         295.0 K
CMT01     140.000000 sec
CMT02     2.0000000 sec
D1         1.0000000 sec
D11        0.0000000 sec
D12        0.0000000 sec
D13        0.0000000 sec
D14        0.0000000 sec
IR0        0.0001280 sec
TSDV
SFO1       600.5537803 MHz
NUC1        13C
P1          11.10 usec
PL1         23.00 usec
PL12        28.00000000
SFO2        150.8827271 MHz
NUC2         13C
P2          10.00 usec
PL2         70.88209911 W
CPRM(1)    SMDQ15.100
CPRM(2)    SMDQ15.100
CPRM(3)    SMDQ15.100
CPRM(4)    SMDQ15.100
CPRM(5)    SMDQ15.100
CPRM(6)    SMDQ15.100
CPRM(7)    SMDQ15.100
CPRM(8)    SMDQ15.100
CPRM(9)    SMDQ15.100
CPRM(10)   SMDQ15.100
CPRM(11)   SMDQ15.100
CPRM(12)   SMDQ15.100
CPRM(13)   SMDQ15.100
CPRM(14)   SMDQ15.100
CPRM(15)   SMDQ15.100
CPRM(16)   SMDQ15.100
CPRM(17)   SMDQ15.100
CPRM(18)   SMDQ15.100
CPRM(19)   SMDQ15.100
CPRM(20)   SMDQ15.100
CPRM(21)   SMDQ15.100
CPRM(22)   SMDQ15.100
CPRM(23)   SMDQ15.100
CPRM(24)   SMDQ15.100
CPRM(25)   SMDQ15.100
CPRM(26)   SMDQ15.100
CPRM(27)   SMDQ15.100
CPRM(28)   SMDQ15.100
CPRM(29)   SMDQ15.100
CPRM(30)   SMDQ15.100
CPRM(31)   SMDQ15.100
CPRM(32)   SMDQ15.100
CPRM(33)   SMDQ15.100
CPRM(34)   SMDQ15.100
CPRM(35)   SMDQ15.100
CPRM(36)   SMDQ15.100
CPRM(37)   SMDQ15.100
CPRM(38)   SMDQ15.100
CPRM(39)   SMDQ15.100
CPRM(40)   SMDQ15.100
CPRM(41)   SMDQ15.100
CPRM(42)   SMDQ15.100
CPRM(43)   SMDQ15.100
CPRM(44)   SMDQ15.100
CPRM(45)   SMDQ15.100
CPRM(46)   SMDQ15.100
CPRM(47)   SMDQ15.100
CPRM(48)   SMDQ15.100
CPRM(49)   SMDQ15.100
CPRM(50)   SMDQ15.100
CPRM(51)   SMDQ15.100
CPRM(52)   SMDQ15.100
CPRM(53)   SMDQ15.100
CPRM(54)   SMDQ15.100
CPRM(55)   SMDQ15.100
CPRM(56)   SMDQ15.100
CPRM(57)   SMDQ15.100
CPRM(58)   SMDQ15.100
CPRM(59)   SMDQ15.100
CPRM(60)   SMDQ15.100
CPRM(61)   SMDQ15.100
CPRM(62)   SMDQ15.100
CPRM(63)   SMDQ15.100
CPRM(64)   SMDQ15.100
CPRM(65)   SMDQ15.100
CPRM(66)   SMDQ15.100
CPRM(67)   SMDQ15.100
CPRM(68)   SMDQ15.100
CPRM(69)   SMDQ15.100
CPRM(70)   SMDQ15.100
CPRM(71)   SMDQ15.100
CPRM(72)   SMDQ15.100
CPRM(73)   SMDQ15.100
CPRM(74)   SMDQ15.100
CPRM(75)   SMDQ15.100
CPRM(76)   SMDQ15.100
CPRM(77)   SMDQ15.100
CPRM(78)   SMDQ15.100
CPRM(79)   SMDQ15.100
CPRM(80)   SMDQ15.100
CPRM(81)   SMDQ15.100
CPRM(82)   SMDQ15.100
CPRM(83)   SMDQ15.100
CPRM(84)   SMDQ15.100
CPRM(85)   SMDQ15.100
CPRM(86)   SMDQ15.100
CPRM(87)   SMDQ15.100
CPRM(88)   SMDQ15.100
CPRM(89)   SMDQ15.100
CPRM(90)   SMDQ15.100
CPRM(91)   SMDQ15.100
CPRM(92)   SMDQ15.100
CPRM(93)   SMDQ15.100
CPRM(94)   SMDQ15.100
CPRM(95)   SMDQ15.100
CPRM(96)   SMDQ15.100
CPRM(97)   SMDQ15.100
CPRM(98)   SMDQ15.100
CPRM(99)   SMDQ15.100
CPRM(100)  SMDQ15.100
F1 - Acquisition parameters
TD         256
SFO1       600.0556 MHz
FIDRES     41.035156 Hz
RG         13.000 ppm
PULPROG    cp
PCMDR      CP

F2 - Processing parameters
SI         1024
SF         600.0499234 MHz
WDW        EM
SSB         0 Hz
GB          0 Hz
DC          0
SC          1.40

F1 - Processing parameters
SI         1024
SF         600.0499234 MHz
WDW        EM
SSB         0 Hz
GB          0 Hz
DC          0
SC          1.40

```



