Supporting Information

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Efficient synthesis of pyrido[2,3-*d*]pyrimidine-7-carboxylic acids catalyzed by a TiO_2/SiO_2 nanocomposite in aqueous media at room temperature

DOI: 10.1515/znb-2018-0076

Received April 19, 2018; accepted June 14, 2018

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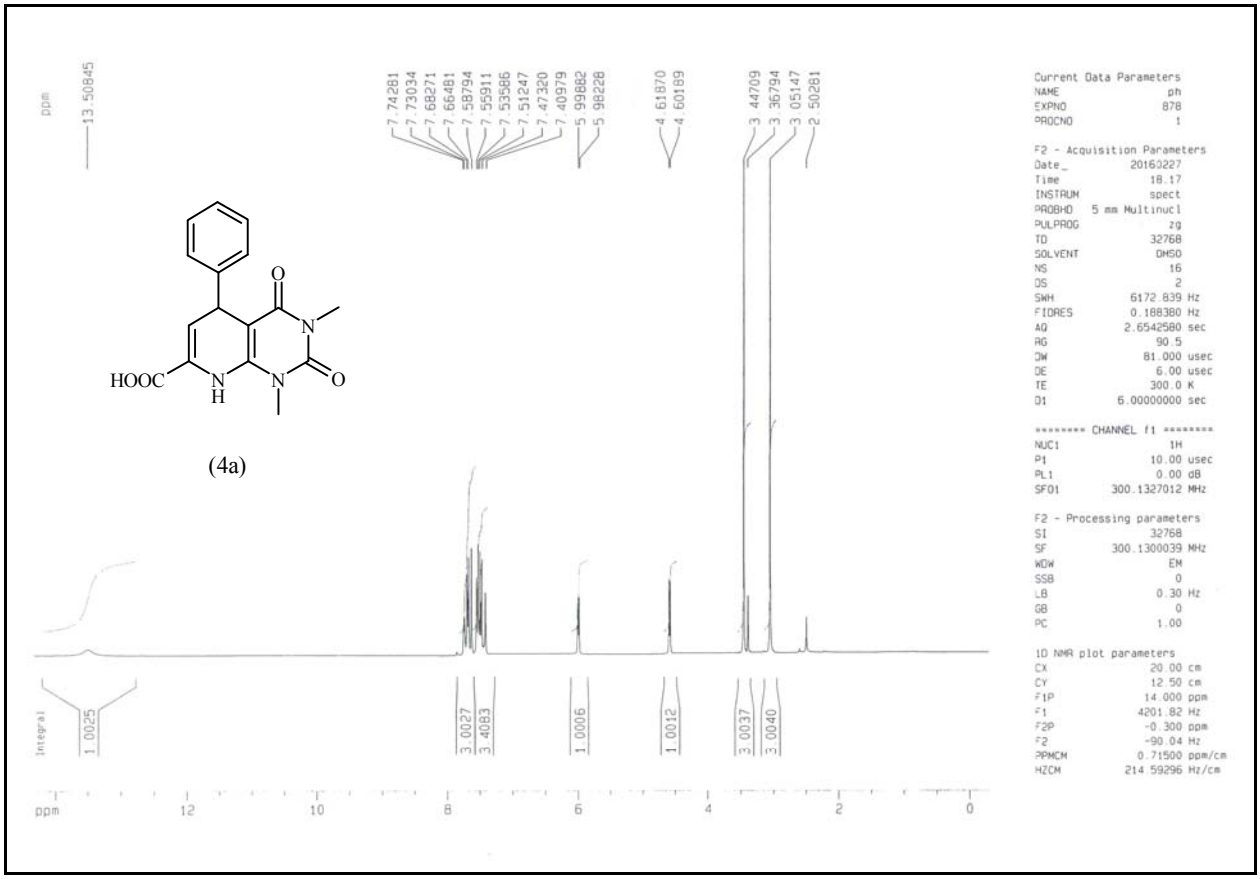
Abstract: A novel and efficient synthesis of eight 5-aryl-1,3-dimethyl-2,4-dioxo-1,2,3,4,5,8-hexahydropyrido[2,3-*d*]pyrimidine-7-carboxylic acids using a TiO_2/SiO_2 nanocomposite with a molar ratio of 1 : 1 as a recyclable heterogeneous catalyst is described. The desired products, five of which are new, are formed in short reaction times (2–3 h) with high to excellent yields (94–98%) under very moderate reaction conditions (room temperature, aqueous media).

^{1}H NMR of **4a**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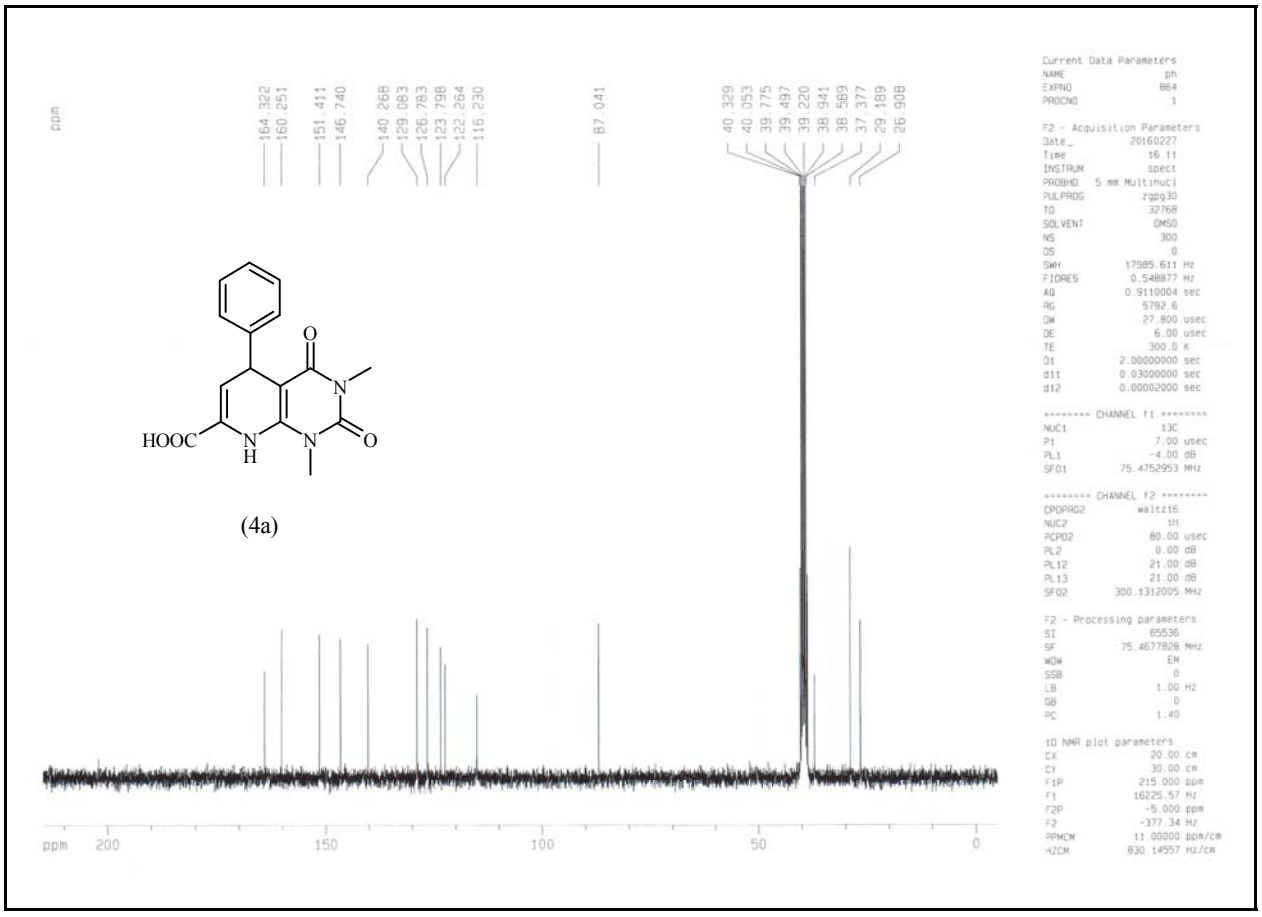
7.68271
7.66481
7.58794
7.55911
7.53586
7.51247
7.47320
7.40979
5.99882
5.98226
4.61870
4.60189
3.44709
3.36794
3.05147
2.50281
HOOC
N
H
O
(4a)
Integral
1.0025
3.0027
3.4083
1.0006
1.0012
3.0037
3.0040
ppm
12
10
8
6
4
2
0
Current Data Parameters
NAME ph
EXPNO 878
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160227
Time 18.17
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec
******** CHANNEL f1 ********
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SF01 300.1327012 MHz
F2 - Processing parameters
SI 32768
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 14.000 ppm
F1 4201.82 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.71500 ppm/cm
HZCM 214.59296 Hz/cm

^{13}C NMR of **4a**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151.411
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140.268
129.083
126.783
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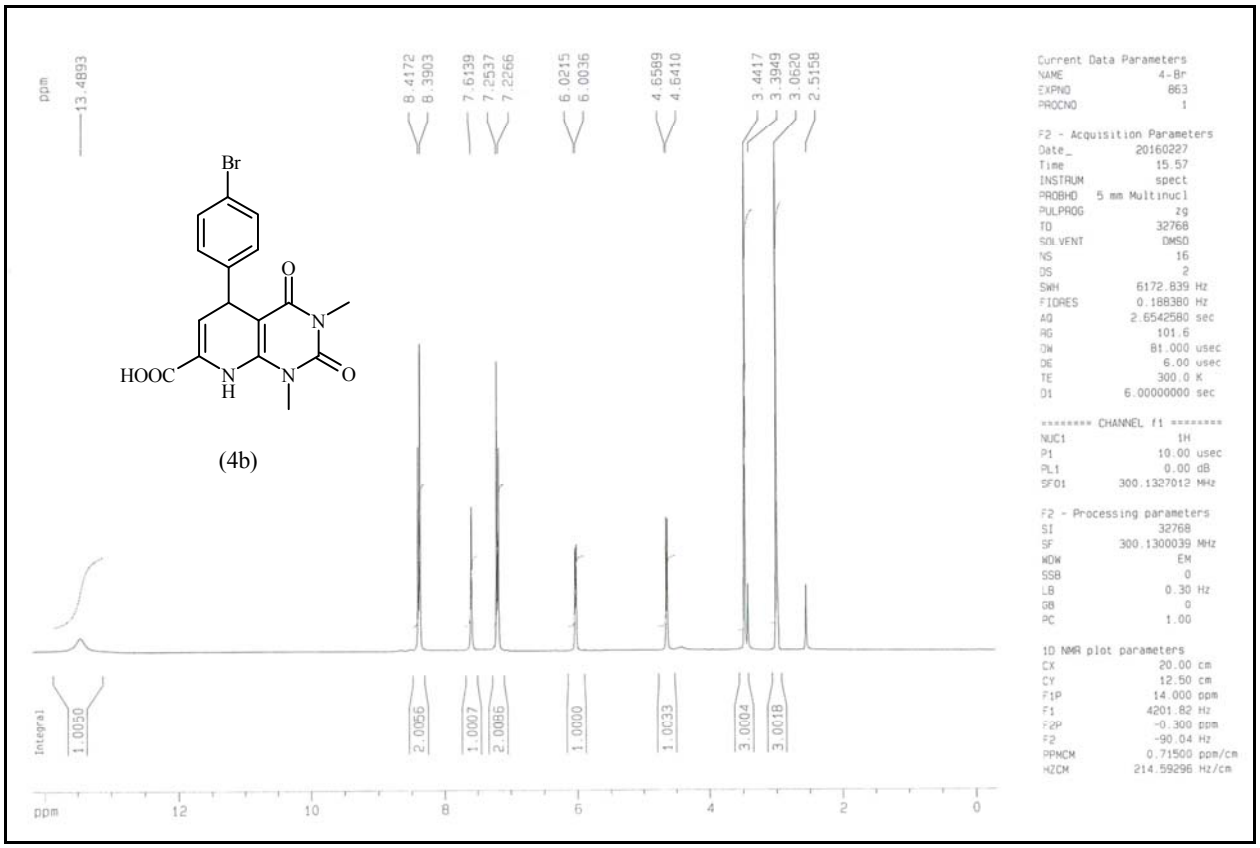
122.264
116.230
87.041
40.329
40.053
39.775
39.497
39.220
38.941
38.589
37.377
29.189
26.908
HOOC
N
H
N
N
O
O
(4a)
ppm 200 150 100 50 0
Current Data Parameters
NAME ph
EXPNO 864
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160227
Time 16.11
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 300
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 5792.6
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -4.00 dB
SF01 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677828 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 30.00 cm
F1P 215.000 ppm
F1 16225.57 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14557 Hz/cm

^{1}H NMR of **4b**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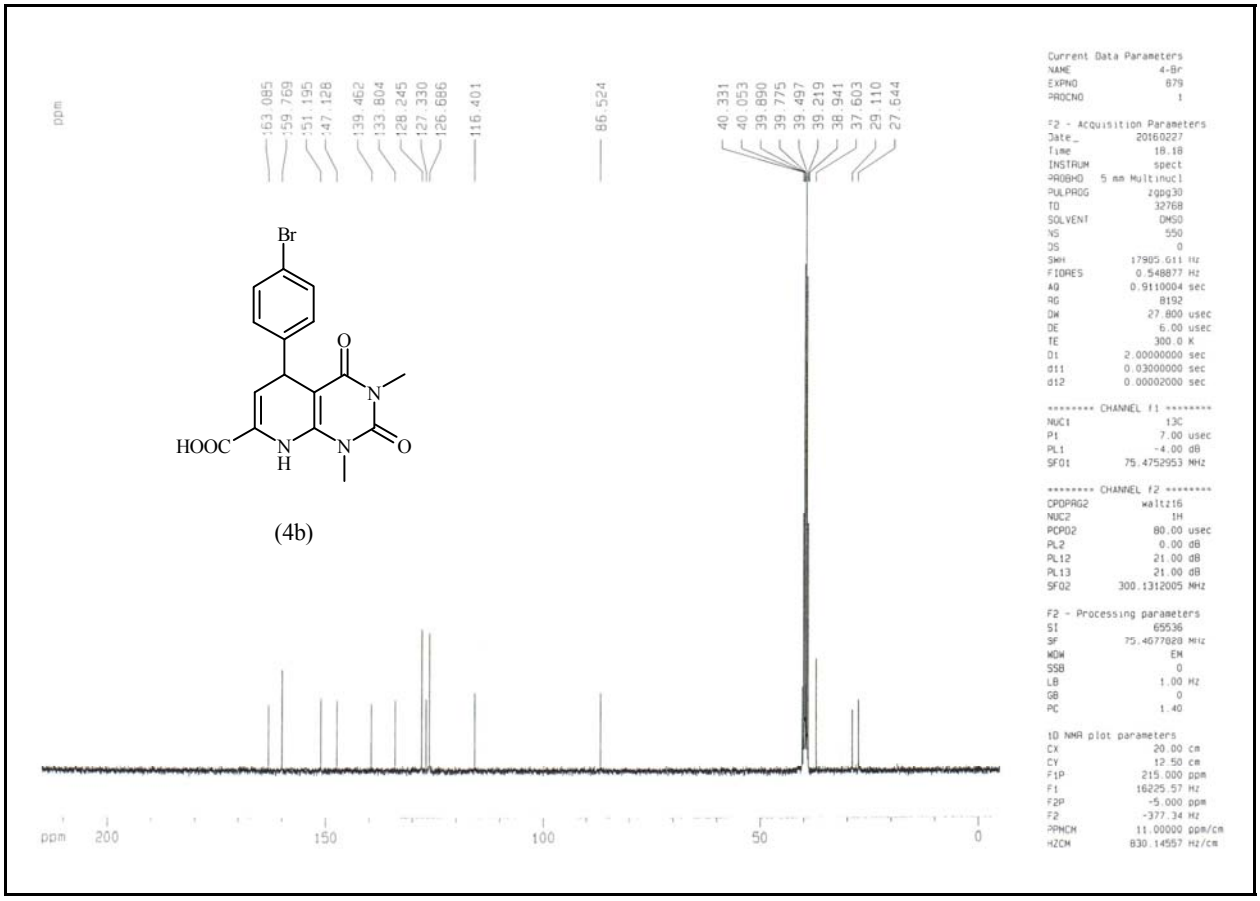
8.3903
7.6139
7.2537
7.2266
6.0215
6.0036
4.6589
4.6410
3.4417
3.3949
3.0620
2.5158
Br
O
N
HOOC
N
H
N
O
(4b)
Integral
1.0050
2.0056
1.0007
2.0086
1.0000
1.0033
3.0004
3.0018
ppm
12
10
8
6
4
2
0
Current Data Parameters
NAME 4-Br
EXPNO 863
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160227
Time 15.57
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 101.6
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SFO1 300.1327012 MHz
F2 - Processing parameters
SI 32768
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 14.000 ppm
F1 4201.82 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.71500 ppm/cm
HZCM 214.59296 Hz/cm

^{13}C NMR of **4b**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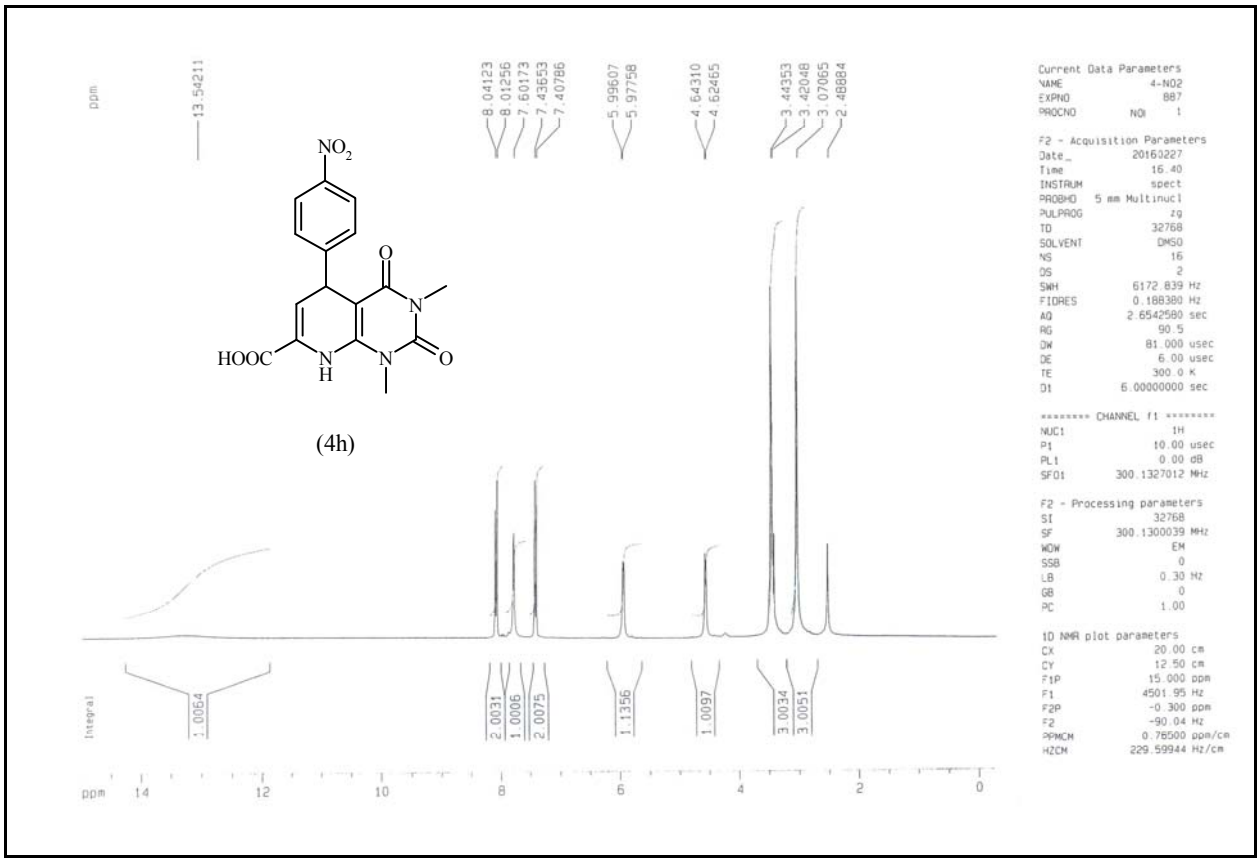
151.195
147.128
139.462
133.804
128.245
127.330
126.686
116.401
86.524
40.331
40.053
39.890
39.775
39.497
39.219
38.941
37.603
29.110
27.644
Br
O
N
HOOC
N
H
N
O
(4b)
ppm
200
150
100
50
0
Current Data Parameters
NAME 4-Br
EXPNO 879
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160227
Time 18.18
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 550
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 8192
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
******** CHANNEL f1 ********
NUC1 13C
P1 7.00 usec
PL1 -4.00 dB
SF01 75.4752953 MHz
******** CHANNEL f2 ********
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677020 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 16225.57 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14557 Hz/cm

^{1}H NMR of **4h**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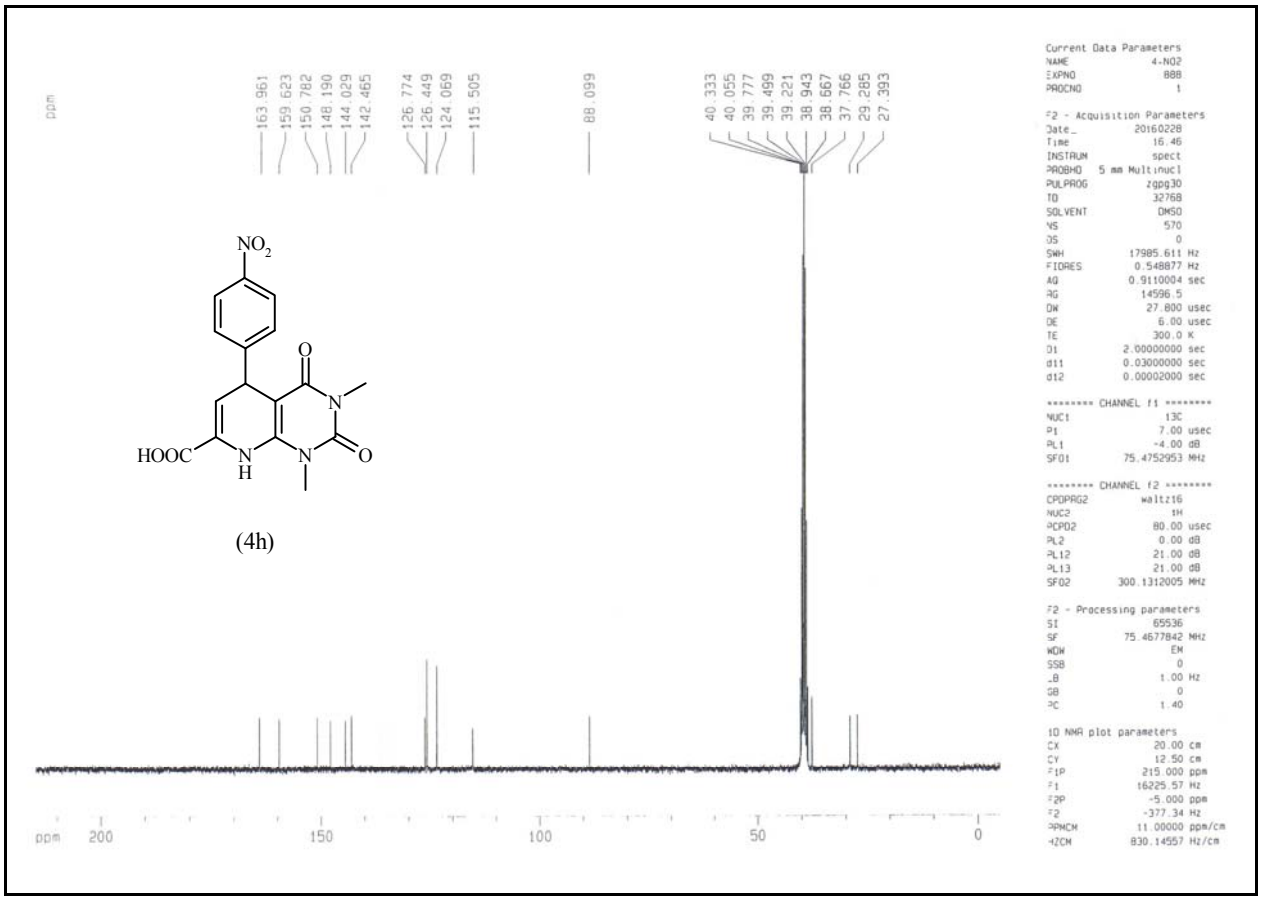
7.43653
7.40786
5.99607
5.97758
4.64310
4.62465
3.44353
3.42048
3.07065
2.48884
NO2
O
N
HOOC
N
H
N
O
(4h)
Integral
1.0064
2.0031
1.0006
2.0075
1.1356
1.0097
3.0034
3.0051
ppm 14 12 10 8 6 4 2 0
Current Data Parameters
NAME 4-NO2
EXPNO 887
PROCNO NOI 1
F2 - Acquisition Parameters
Date_ 20160227
Time 16.40
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.188380 Hz
AQ 2.6542580 sec
RG 90.5
DW 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 6.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 0.00 dB
SF01 300.1327012 MHz
F2 - Processing parameters
SI 32768
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 15.000 ppm
F1 4501.95 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPMCM 0.76500 ppm/cm
HZCM 229.59944 Hz/cm

^{13}C NMR of **4h**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159.623
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148.190
144.029
142.465
126.774
126.449
124.069
115.505
88.099
40.333
40.055
39.777
39.499
39.221
38.943
38.667
37.766
29.285
27.393
NO2
O
N
HOOC
N
H
N
O
(4h)
ppm 200 150 100 50 0
Current Data Parameters
NAME 4-NO2
EXPNO 888
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160228
Time 16.46
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 570
DS 0
SWH 17985.611 Hz
FIDRES 0.548877 Hz
AQ 0.9110004 sec
RG 14596.5
DW 27.800 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -4.00 dB
SF01 75.4752953 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.00 dB
PL13 21.00 dB
SF02 300.1312005 MHz
F2 - Processing parameters
SI 65536
SF 75.4677842 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 215.000 ppm
F1 16225.57 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 11.00000 ppm/cm
HZCM 830.14557 Hz/cm