Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

Greener and expeditious one-pot synthesis of dihydropyrimidinone derivatives using non-commercial β-ketoesters *via* Biginelli reaction

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Contents

1. General details

Melting points were determined with open capillary tube on a Gallenkamp (variable heater) melting point apparatus and are uncorrected. The ^{1}H and ^{13}C NMR spectra of the synthesized compounds were recorded at 400 MHz and 100 MHz respectively using Bruker AVANCE 400 MHz NMR spectrometer in DMSO-d_6 solvent and the chemical shifts are expressed in δ (ppm) relative to TMS as internal standard and coupling constants (*J*) in Hz. Spin multiplicities are shown as s (singlet), d (doublet), t (triplet) and m (multiplet). Mass analyses were performed on quadruple-time of flight (Q-Tof) mass spectrometer (Micromass, USA) using electron spray ionization (ESI) in positive mode. TLC analyses were performed using precoated aluminium sheets with silica gel 60 F_{254}.

2. Experimental Procedure

An oven-dried 25ml round bottomed flask equipped with a refluxed condenser was charged with arylaldehyde (1.0 mmol) and urea (1.2 mmol). The precursors were finely powdered and mixed together and allowed to mechanical stirring for 30min at room temperature. The *tert*-butyl β-ketoester (1.0 mmol) and an alcohol (1.5 mmol) were added to above mixture subsequently. The resulting reaction mixture was heated for 3hr at 110˚C (oil bath) with constant stirring till the reaction was completed. The progress of reaction was monitored by TLC. After completion of reaction as indicated on TLC, the contents of reaction mixture was cooled to room temperature and the crude reaction mixture was crushed and washed with chilled water (15ml x 3), filtered and dried under vacuum. For analytically pure products, the final solid mass was washed with diethyl ether (10ml x 3) to remove the un-reacted reactants to afford the pure product in 85-94% yield.

3. Spectroscopic characterization data

Isopentyl-4-(4-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 1): White colored solid, 148-150°C, IR (ν_{Max}): 782, 836, 1095, 1226, 1513, 1654, 1726, 2958, 3116, 3246. ^{1}H-NMR (400MHz, DMSO-d_6): δ 0.752 (d, 6H), 1.336-1.379 (m, 2H), 1.398-1.447 (m, 1H), 2.202 (s, 3H), 3.716 (s, 3H), 3.921-4.002 (m, 2H), 5.078 (d, 1H), 6.857 (d, *J* = 8.4 Hz, 2H), 7.117 (d, *J* = 8.4 Hz, 2H), 7.618 (s, 1H), 9.117 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.663, 22.035, 22.209, 24.307, 36.899, 53.352, 55.015, 61.426, 99.354, 113.653, 127.333, 136.920, 148.141, 152.011, 158.441, 165.322. ESI-MS: 333.520 [M+1].

Prop-2-ynyl-4-(4-ethylphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 8): White colored solid, M. P. 150-152°C, IR (ν_{Max}): 683, 790, 1083, 1224, 1511, 1680, 1713, 2967, 3250, 3293. ^{1}H-NMR (400MHz, DMSO-d_6): δ 1.130 (t, *J* = 7.6 Hz, 3H), 2.252 (s, 3H), 2.530-2.587 (q, *J* = 7.6Hz, 2H), 3.450 (t, 1H), 4.632 (d, 2H), 50105(d, 1H), 7.148 (s, 4H), 7.739 (s, 1H), 9.273 (s, 1H). ^{13}C NMR (DMSO-d_6): 15.524, 17.847, 27.737, 50.893, 53.292, 77.104, 78.749, 98.545, 126.039, 127.715, 141.830, 142.774, 149.546, 152.078, 164.461. ESI-MS:- 299.68 [M+1].

Prop-2-ynyl-4-(4-chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 9): Pale yellow colored solid, M. P. 174-176°C, IR (ν_{Max}): 679, 789, 847, 1081, 1239, 1374, 1490, 1726, 2947, 3121, 3251. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.263 (s, 3H), 3.445 (t, 1H), 4.634 (t, 2H), 5.138 (d, 1H), 7.246 (d, *J* = 8.4 Hz, 2H), 7.376 (d, *J* = 8.4 Hz, 2H), 7.821 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.895, 50.952, 53.132, 77.142, 78.684, 97.960, 128.059, 128.378, 131.852, 143.415, 150.082, 151.808, 164.309. ESI-MS:- 305.578 [M+1].

Prop-2-ynyl-4-(3-chlorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 10): Yellow colored solid, M. P. 156-158°C, IR (ν_{Max}): 691, 786, 1090, 1227, 1430, 1648, 1710, 2930, 3247, 3302. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.27 (s, 3H), 3.447 (t, 1H), 4.645 (d, 2H), 5.150 (d, 1H), 7.197 (d, 1H), 7.256 (s, 1H), 7.309-7.350 (m, 1H), 7.369 (d, 1H), 7.851 (s, 1H), 9.396 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.918, 51.021, 53.292, 77.179, 78.641, 97.766, 124.765, 126.115, 127.311, 130.457, 133.003, 146.847, 150.330, 151.863, 159.679, 164.298. ESI-MS: 304.241 [M+1].

Prop-2-ynyl-4-(4-fluorophenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 11): Cream colored solid: M. P. 172-174°C, IR (ν_{Max}): 795, 851, 1078, 1240, 1462, 1675, 1724, 2950, 3270, 3291. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.264 (s, 3H), 3.439 (t, 1H), 4.631 (d, 2H), 5.141 (d, 1H), 7.120 (d, 2H), 7.252 (d, 2H), 7.801 (s, 1H), 9.342 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.868, 50.915, 53.055, 77.094, 78.698, 98.273, 114.993, 115.205, 128.088, 128.169, 140.728, 140.757, 149.878, 151.863, 159.616, 160.120, 162.536, 164.357. ESI-MS: 289.560 [M+1].

Prop-2-ynyl-6-methyl-2-oxo-4-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 12): White colored solid: M. P. 166-168°C, IR (ν_{Max}): 761, 832, 920, 1077, 1237, 1334, 1443, 1641, 1712, 3282, 3341. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.276 (s, 3H), 3.447 (t, 1H), 4.642 (d, 2H), 5.229 (d, 1H), 7.456 (d, *J* = 8.0 Hz, 2H), 7.694 (d, *J* = 8.4 Hz, 2H), 7.896 (s, 1H), 9.426 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.955, 51.034, 53.515, 77.164, 78.662, 97.707, 122.858, 125.391, 125.428, 125.466, 125.499, 125.561, 127.071, 127.891, 128.066, 128.205, 148.938, 150.460, 151.842, 159.882, 164.311. ESI-MS: 339.521 [M+1].

Prop-2-ynyl-6-methyl-2-oxo-4-phenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 13): Pale yellow colored solid: 182-184°C, IR (ν_{Max}): 662, 696, 793, 1083, 1291, 1644, 1704,

2939, 3116, 3291. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.246 (s, 3H), 3.445 (t, 1H), 4.637 (d, 2H), 5.143 (d, 1H), 7.225-7.339 (m, 5H), 7.795 (s, 1H), 9.312 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.913, 50.944, 53.702, 77.063, 78.750, 98.485, 126.146, 127.307, 128.402, 144.515, 149.739, 152.128, 164.485. ESI-MS: 271.751 [M+1].

Prop-2-ynyl-6-methyl-4-(4-nitrophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 14): White colored solid: 180-182°C, IR (ν_{Max}): 698, 787, 1227, 1350, 1648, 1729, 2928, 3239, 3372. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.281 (s, 3H), 3.447 (t,1H), 4.639 (s, 2H), 5.274 (d, 1H), 7.505 (d, *J* = 8.4 Hz, 2H), 7.950 (s, 1H), 8.194 (d, *J* = 8.4 Hz, 2H), 9.483 (s,1H). ^{13}C NMR (DMSO-d_6): 17.957, 51.037, 53.456, 77.203, 78.609, 97.334, 123.771, 125.460, 127.580, 128.074, 146.767, 150.722, 151.615, 151.662, 159.815, 164.186. ESI-MS: 316.109 [M+1].

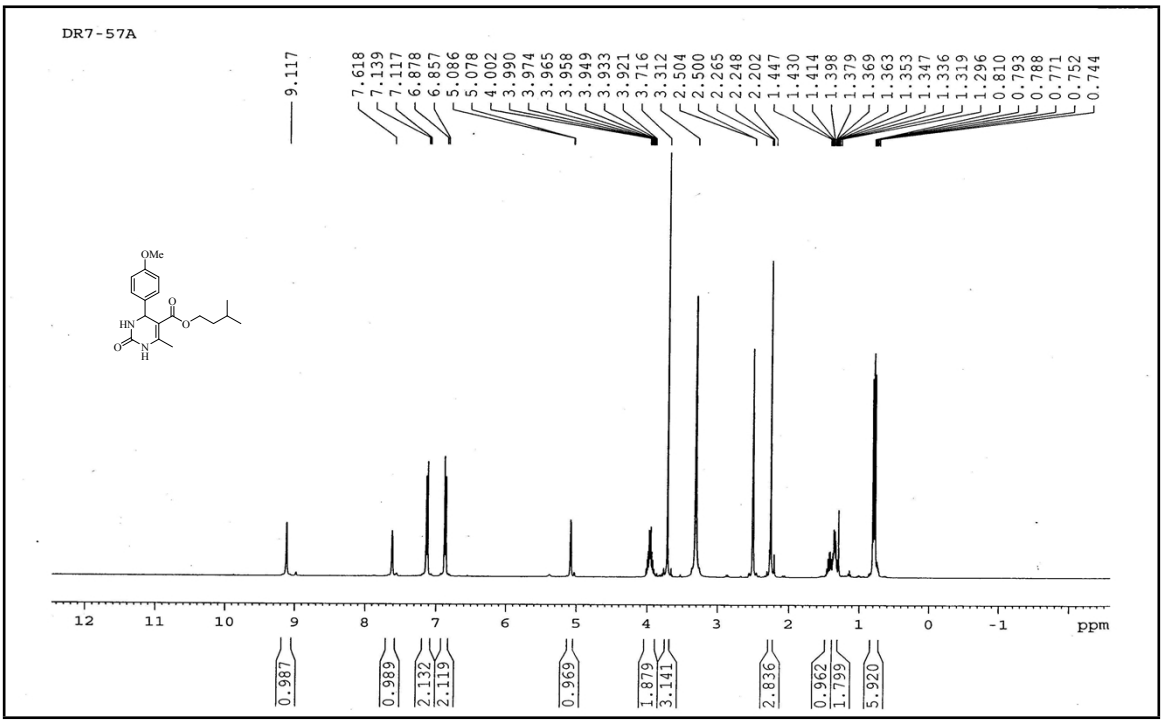
Prop-2-ynyl-6-methyl-4-(4-ethoxyphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 15): Yellow colored solid, M. P. 196-198°C, IR (ν_{Max}): 684, 792, 1087, 1222, 1512, 1644, 1708, 2936, 3246, 3299. ^{1}H-NMR (400MHz, DMSO-d_6): δ 1.281 (t, *J* = 6.8 Hz, 3H), 2.253 (s, 3H), 3.442 (t, 1H), 3.957-3.991 (q, *J* = 6.8 Hz, 2H), 4.626 (t, 2H), 5.080 (d, 1H), 6.840 (d, *J* = 8.4 Hz, 2H), 7.128 (d, *J* = 8.8 Hz, 2H), 7.714 (s, 1H), 9.259 (s, 1H). ^{13}C NMR (DMSO-d_6): 14.864, 18.124, 51.150, 53.317, 63.229, 77.354, 79.047, 99.003, 114.490, 127.545, 136.834, 149.657, 152.319, 158.016, 164.763. ESI-MS: 315.761 [M+1].

Prop-2-ynyl-6-methyl-4-(3-fluorophenyl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 16): White colored solid: M. P. 184-186°C, IR (ν_{Max}): 694, 789, 990, 1225, 1429, 1647, 1705, 2947, 3243, 3302. ^{1}H-NMR (400MHz, DMSO-d_6): δ 2.270 (d, 3H), 3.442 (t, 1H), 4.650 (t, 2H), 5.162 (d, 1H), 6.994 (d, 1H), 7.086 (d, 2H), 7.351-7.406 (m, 1H), 7.840 (s, 1H), 9.366 (s, 1H). ^{13}C NMR (DMSO-d_6): 17.904, 50.997, 53.240, 77.117, 78.676, 97.869, 112.812, 113.027, 113.991, 114.200, 122.085, 130.437, 130.518, 147.271, 147.330, 150.292, 151.911, 160.899, 163.325, 164.339. ESI-MS: 289.914 [M+1].

2-(dimethylamino)ethyl-6-methyl-4-(4-ethoxyphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 18):

White colored solid: M. P. 178-180°C, IR = 3241, 3111, 2973, 2770, 1724, 1706, 1650, 1612, 1513, 1460, 1390, 1225, 1048, 955, 793. ^{1}H-NMR (400MHz, $CDCl_3$) δ 1.374 (t, *J* = 6.8Hz, 3H), 2.167 (s, 6H), 2.300 (s, 3H), 2.423 (t, *J* = 6.0Hz, 2H), 3.990-3.973 (q, *J* = 6.8Hz, 2H), 4.075 (t, *J* = 6.0Hz, 2H), 5.311 (s, 1H), 5.660 (s, 1H), 6.765 (d, *J* = 8.4Hz, 2H), 7.176 (d, *J* = 8.4Hz, 2H), 8.016 (s, 1H). ^{13}C NMR ($CDCl_3$) δ = 14.940, 18.857, 45.770, 55.309, 57.862, 62.006, 63.611, 101.613, 114.745, 127.944, 136.022, 146.375, 153.260, 158.808, 165.785, ESI-MS: *m/z* = 348 [M+1].

2-(dimethylamino)ethyl-6-methyl-4-(4-methylphenyl)-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (Table 1, entry 19): White colored solid: M. P. 168-170°C, IR = 3245, 3118, 2949, 2825, 2775, 2361, 1709, 1651, 1462, 1287, 1225, 1102, 783, 501. ^{1}H-NMR (400MHz, $CDCl_3$) δ 2.206 (s, 6H), 2.316 (s, 3H), 2.337 (s, 3H), 2.446 (t, *J* = 5.6Hz, 2H), 4.103 (t, *J* = 5.6Hz, 2H), 5.369 (s, 1H), 5.642 (s, 1H), 7.095 (d, *J* = 7.6Hz, 2H), 7.199 (d, *J* = 7.6Hz, 2H), 7.858 (s, 1H). ^{13}C NMR ($CDCl_3$) δ = 18.845, 21.283, 45.808, 55.536, 57.926, 62.032, 101.434, 126.715, 129.561, 137.800, 141.073, 146.853, 153.675, 165.871. ESI-MS: *m/z* = 318.67 [M+1].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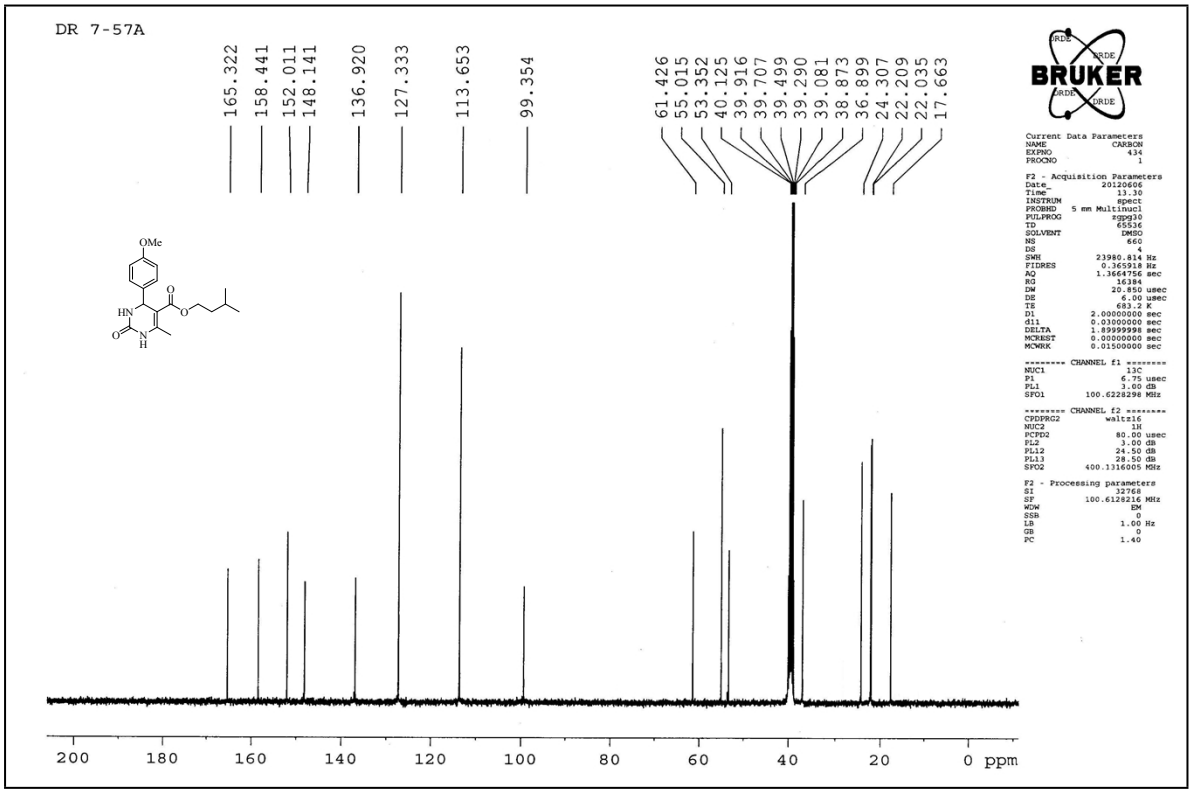
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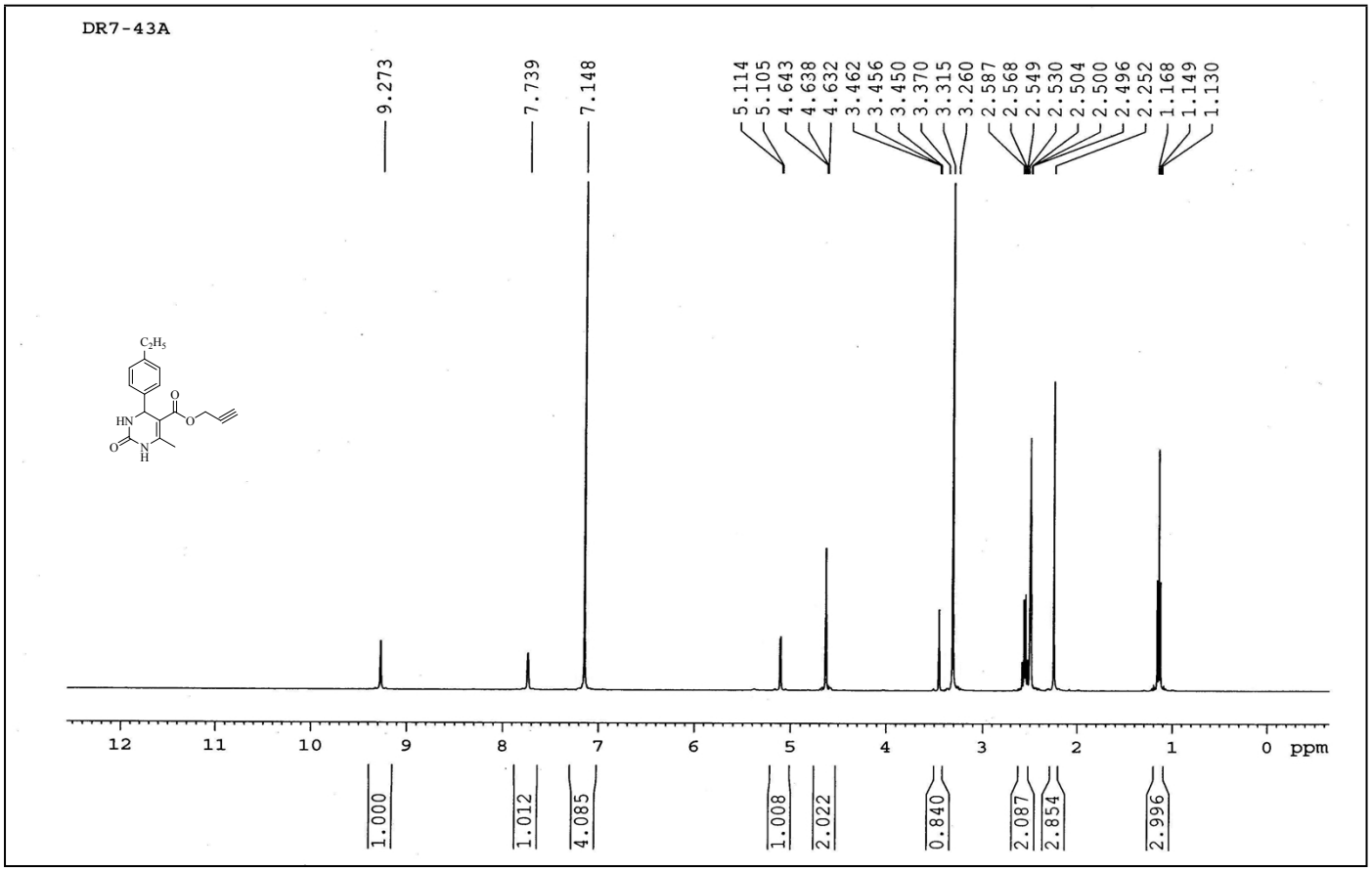
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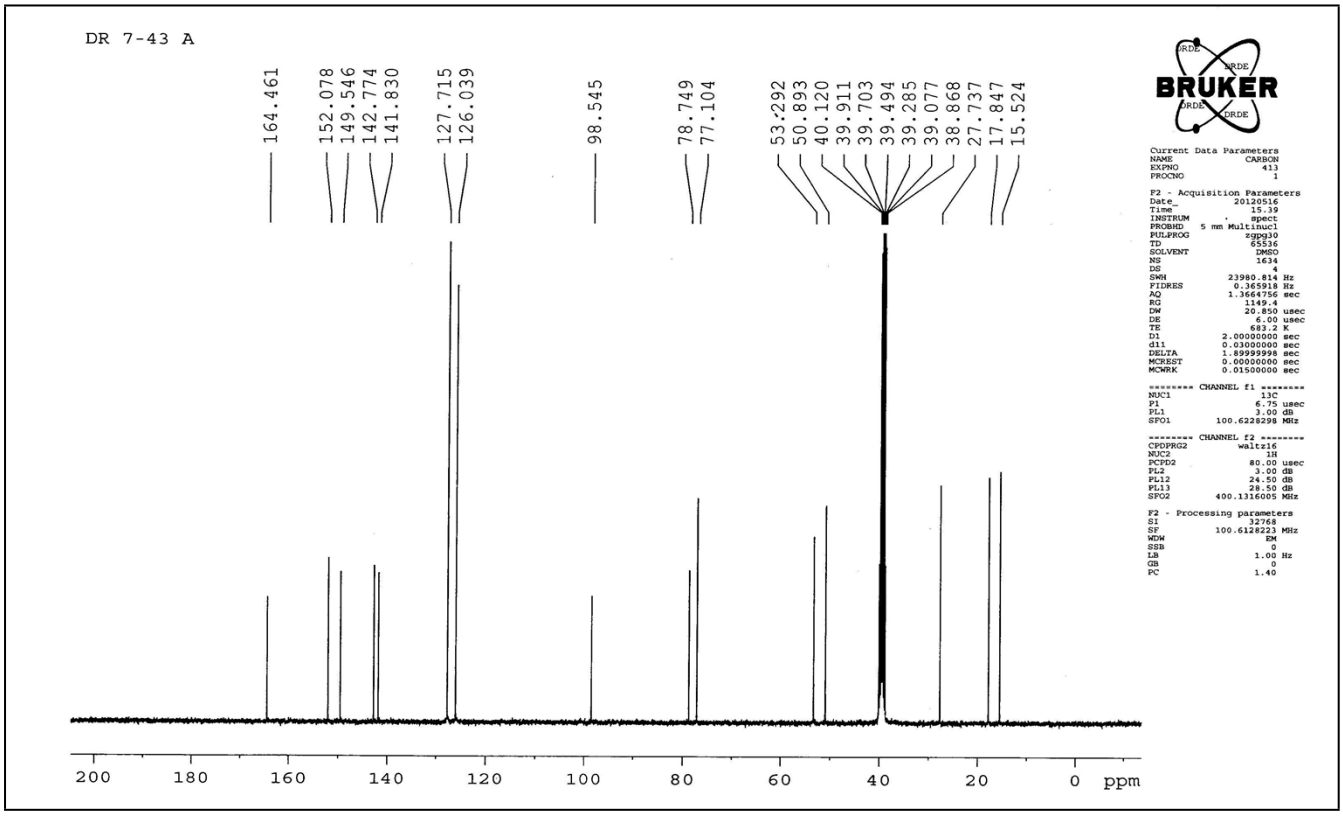
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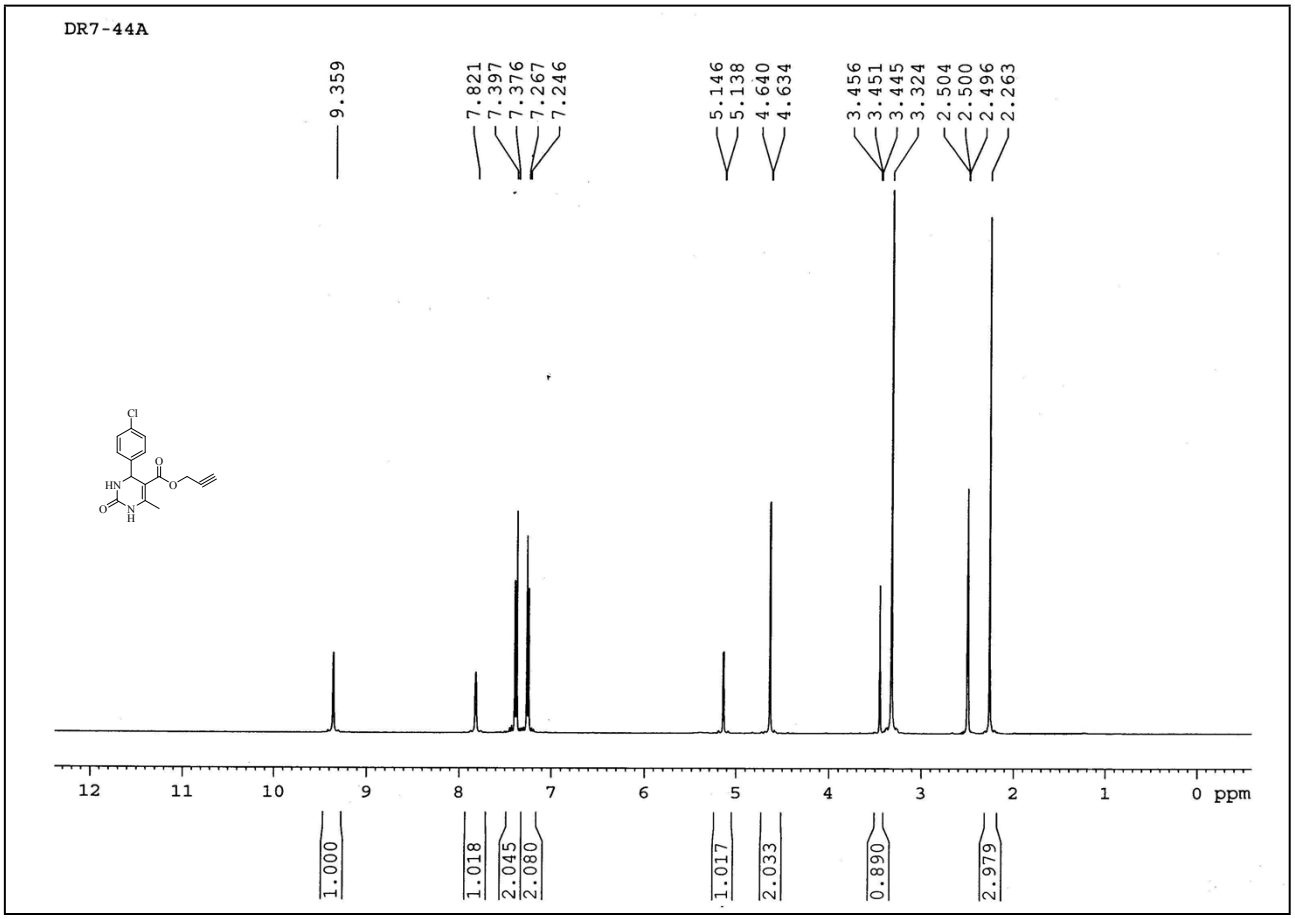
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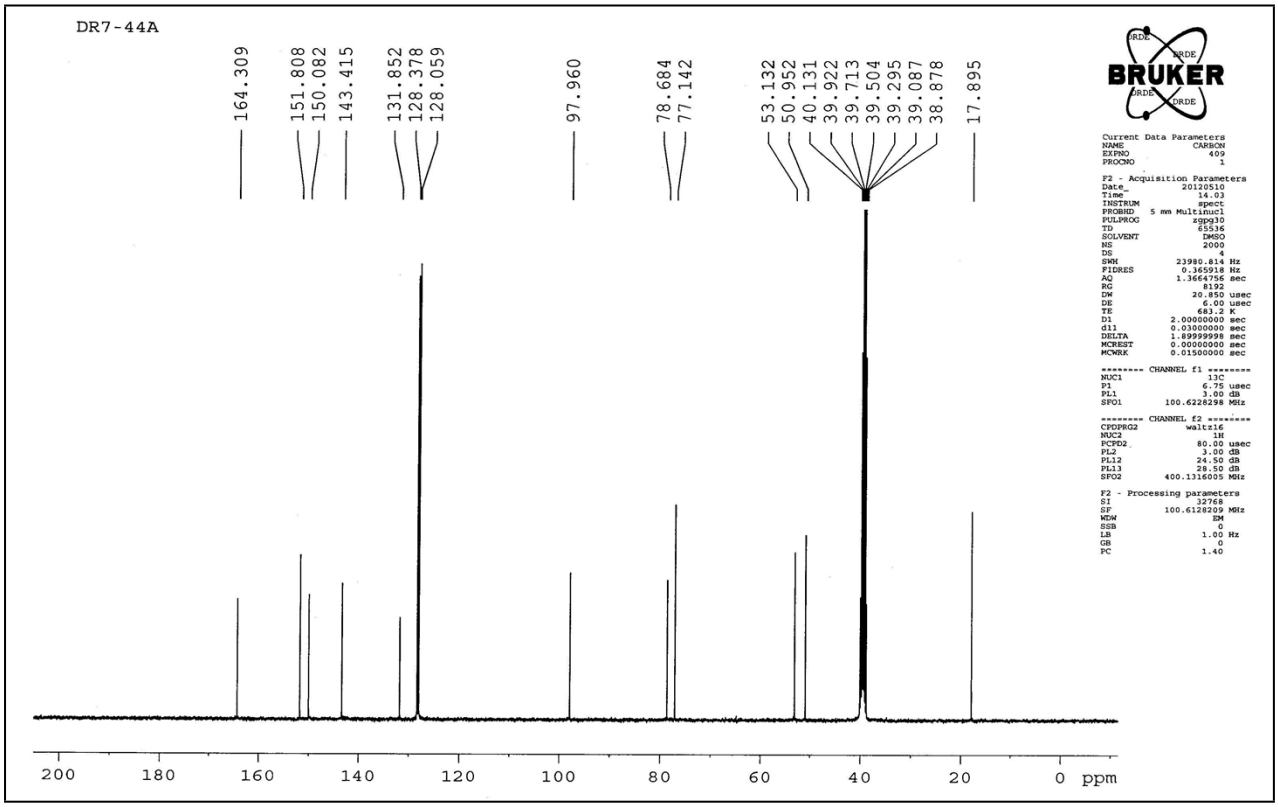
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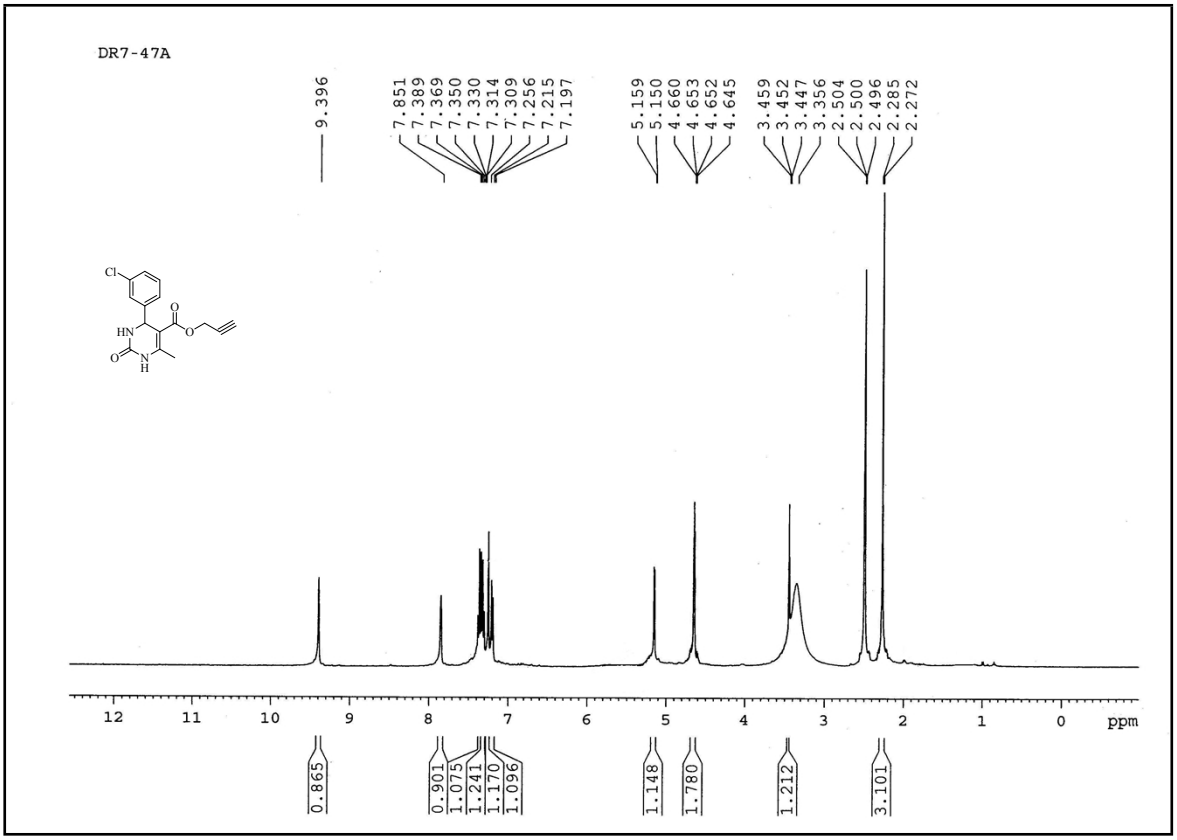
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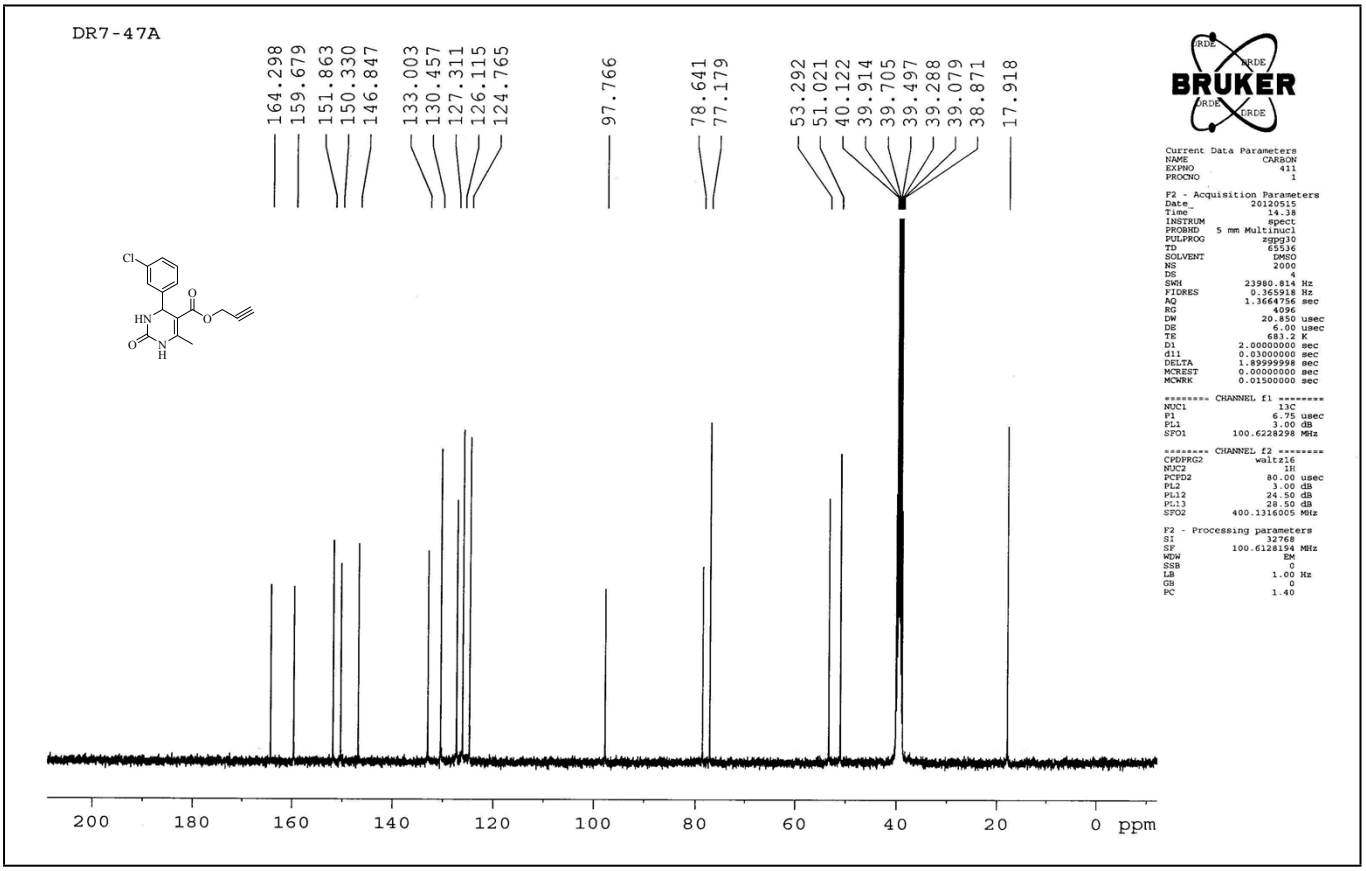
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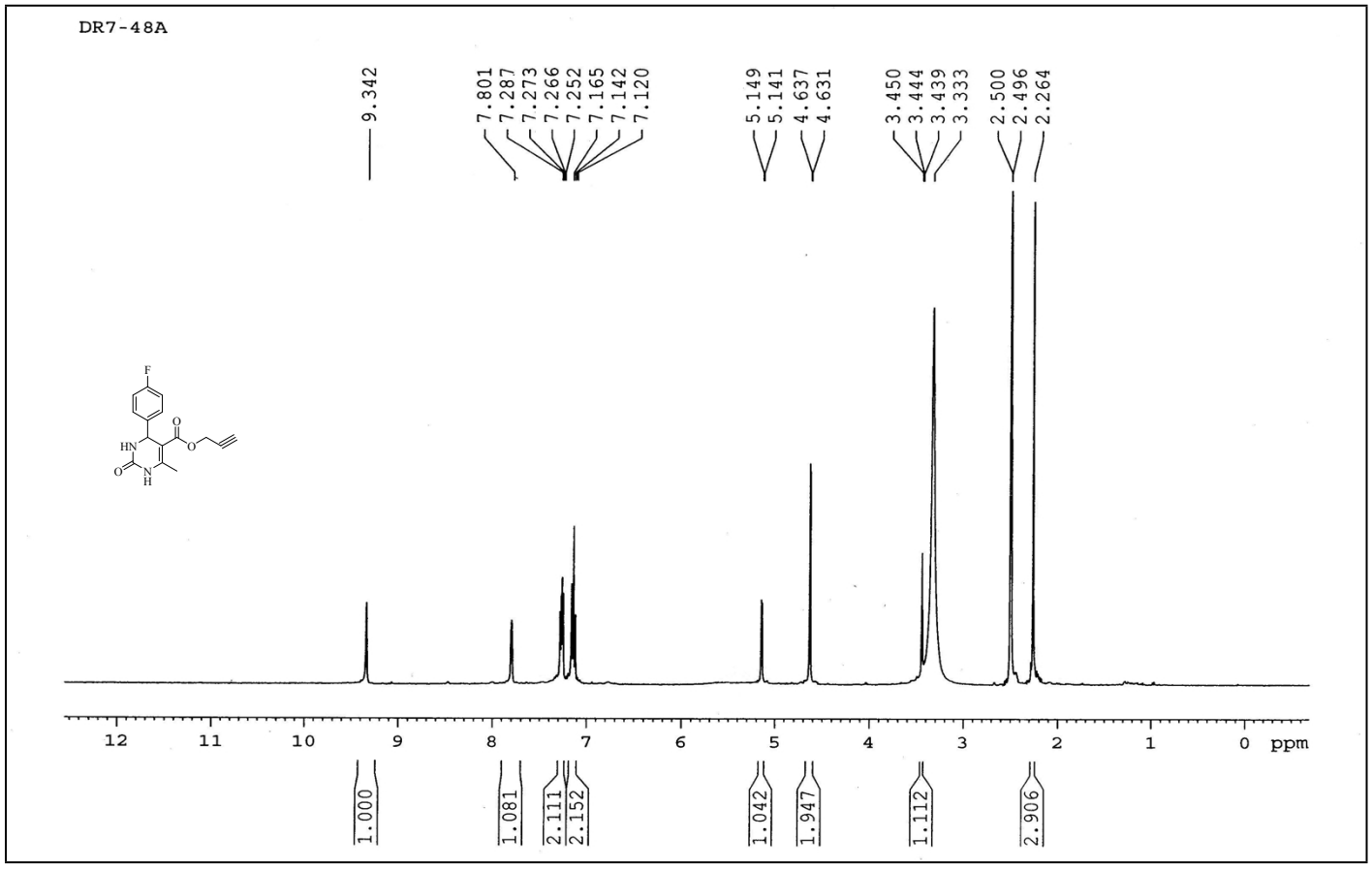
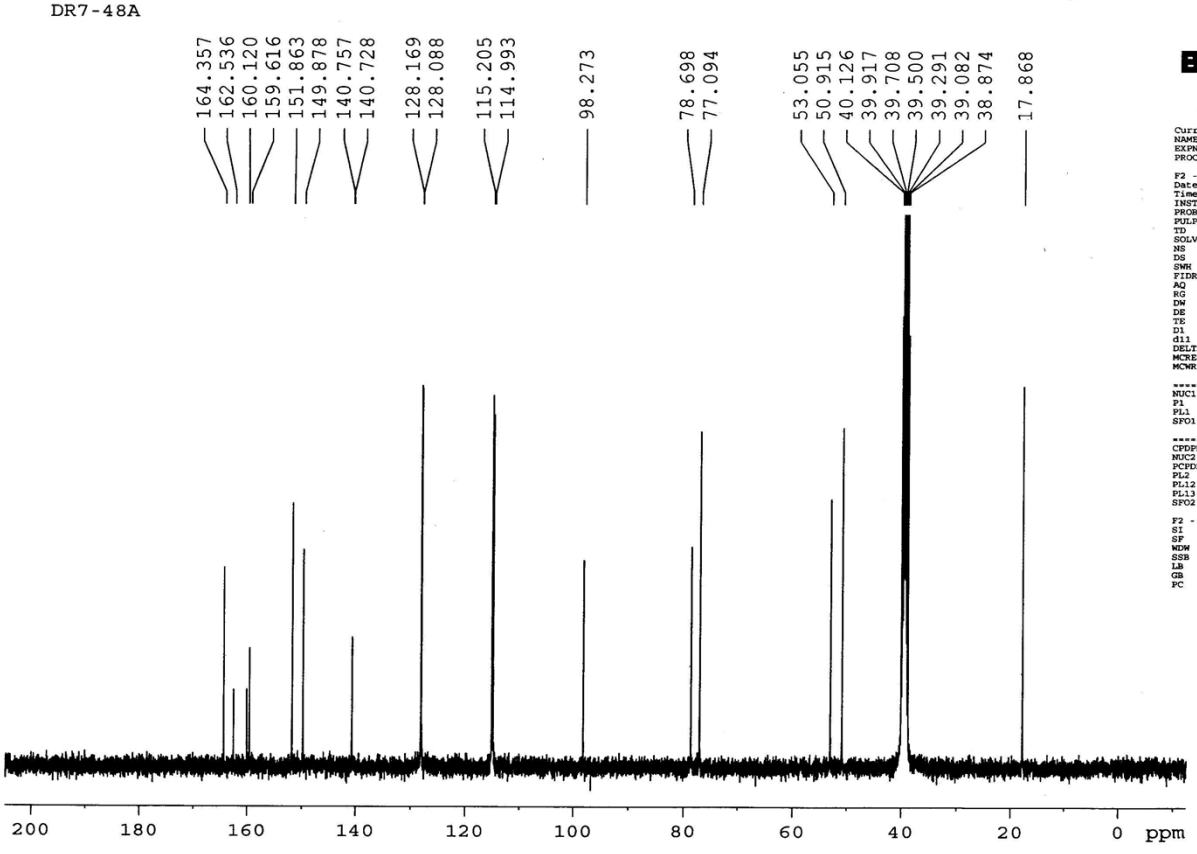
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128194 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DR7-48A



DR7-48A



BRUKER

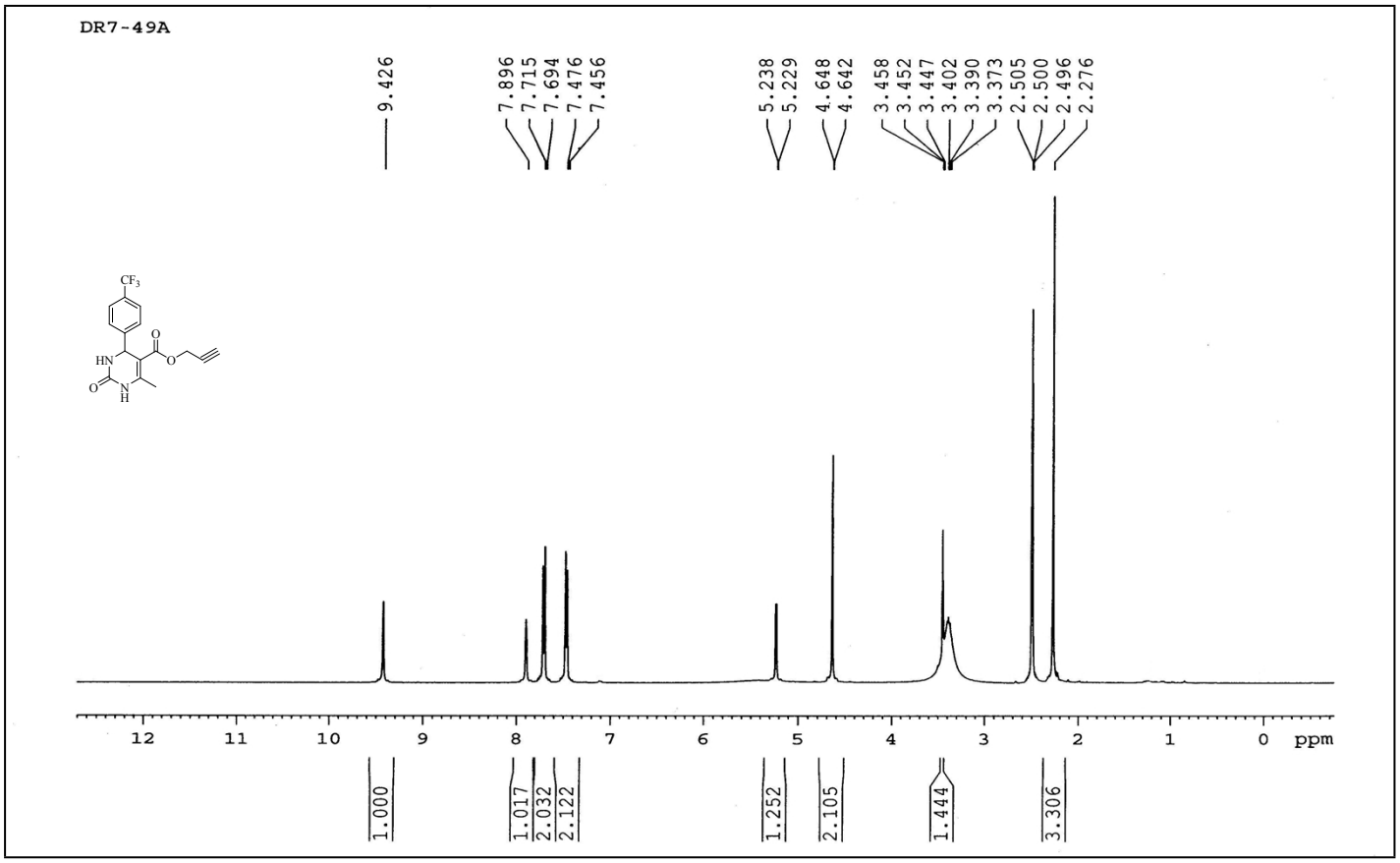
Current Data Parameters
NAME CARBON
EXPNO 421
PROCNO 1

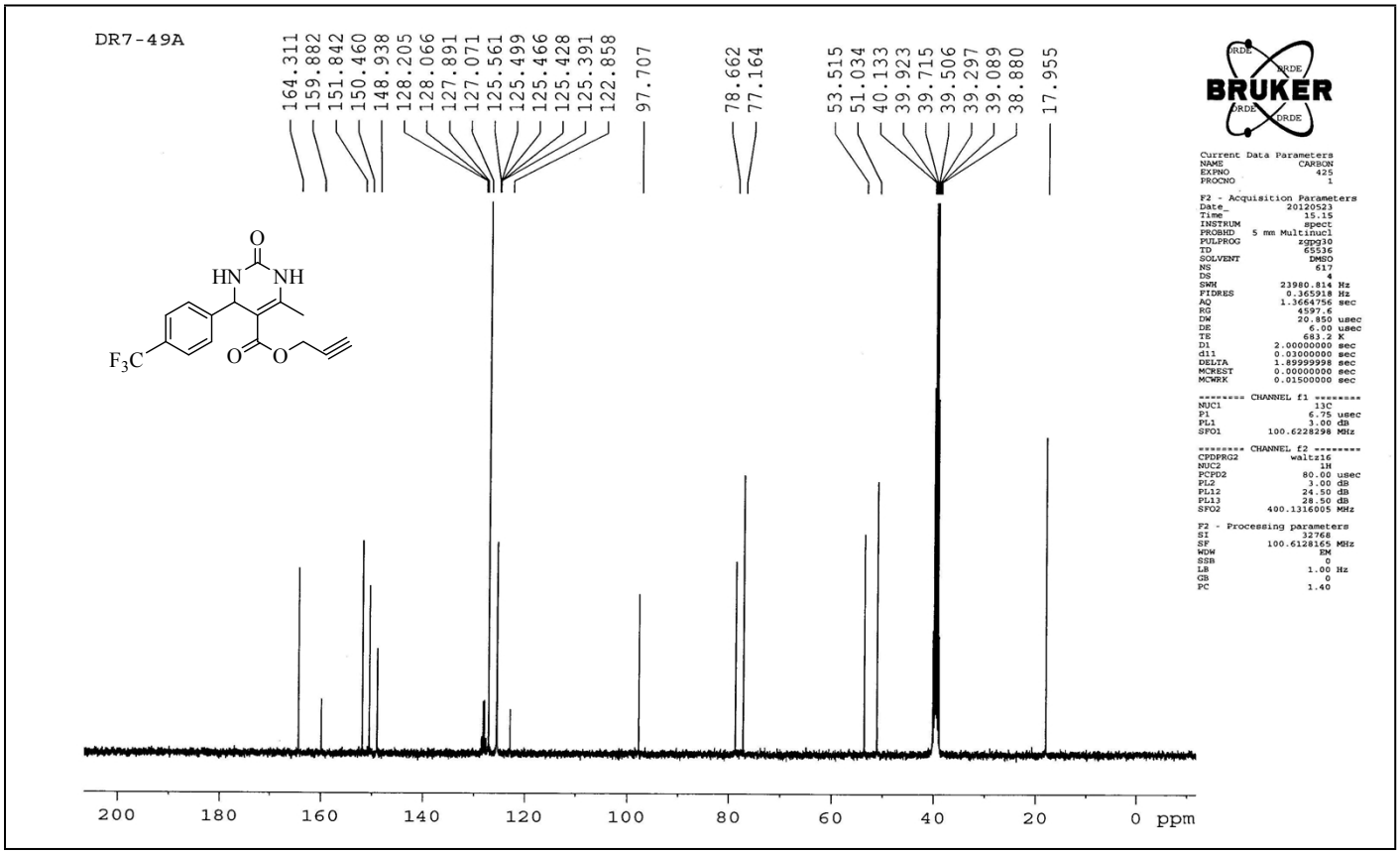
F2 - Acquisition Parameters
Date_ 20120521
Time 16.18
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 574.7
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

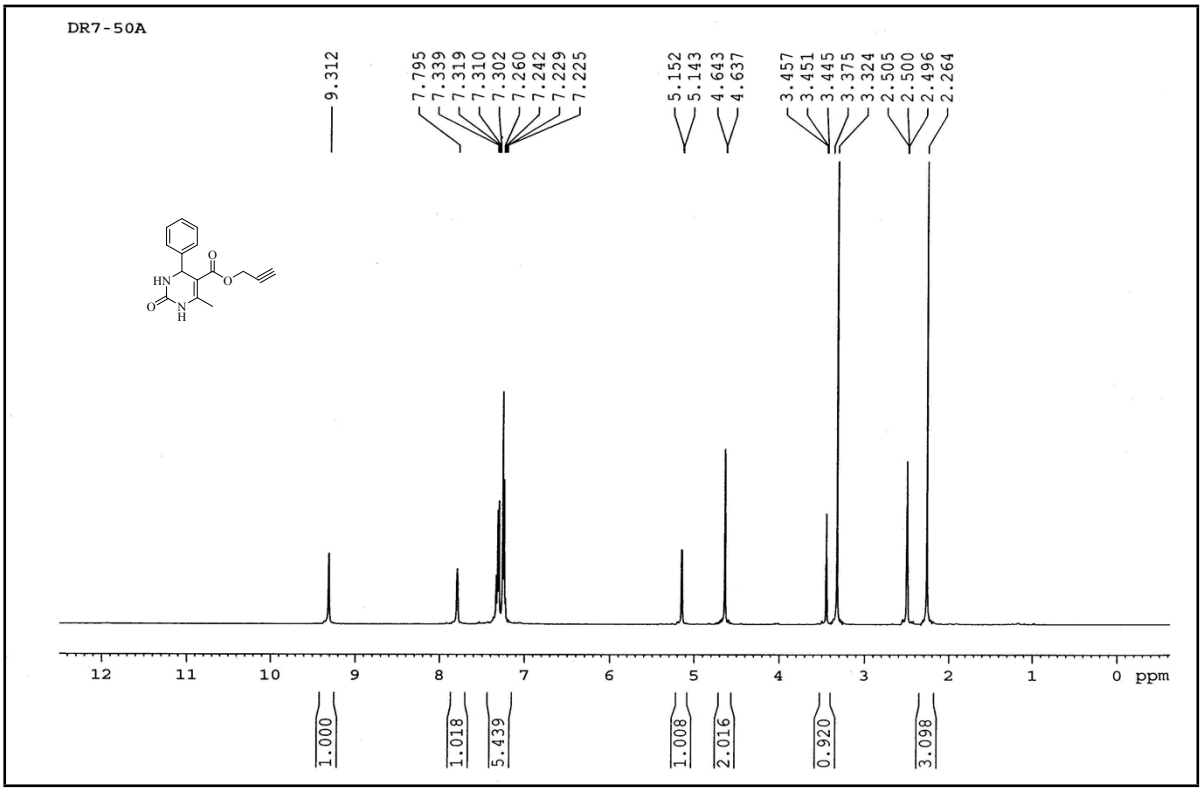
-------- CHANNEL f1 --------
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz

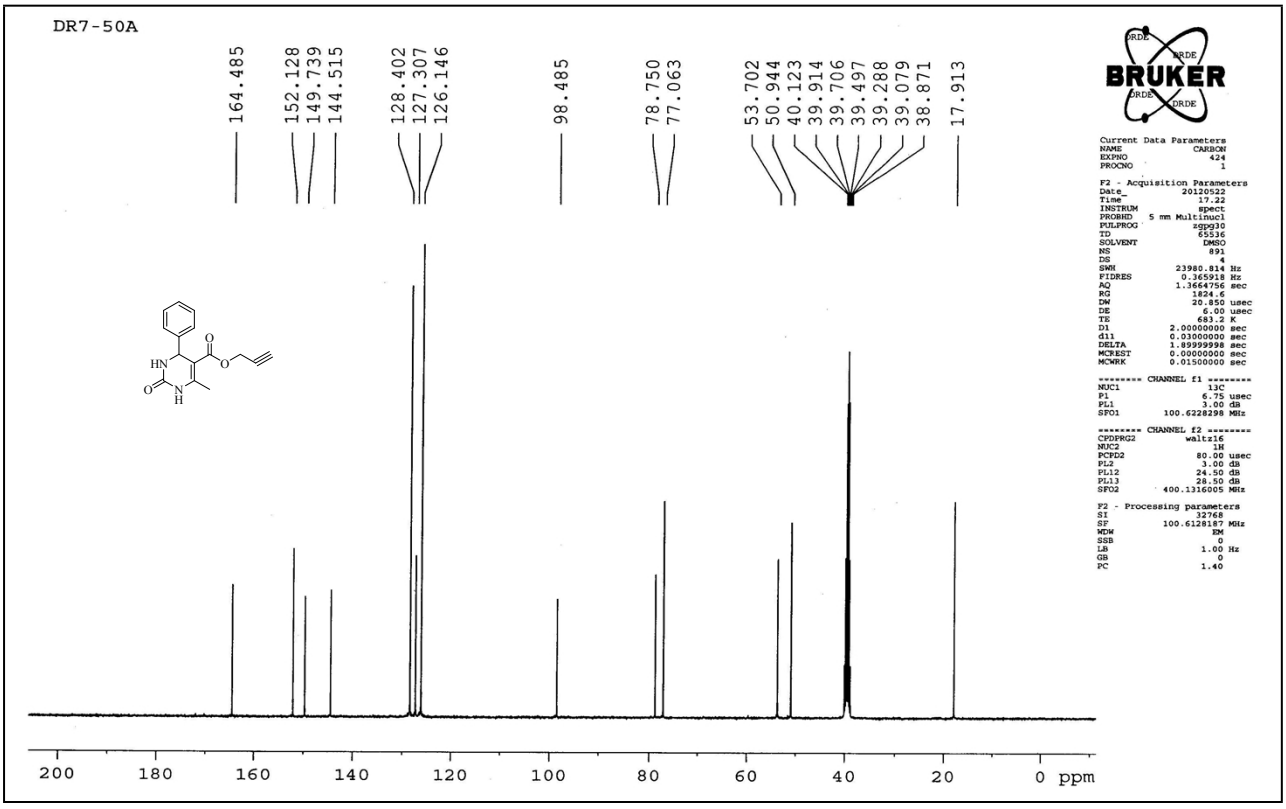
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz

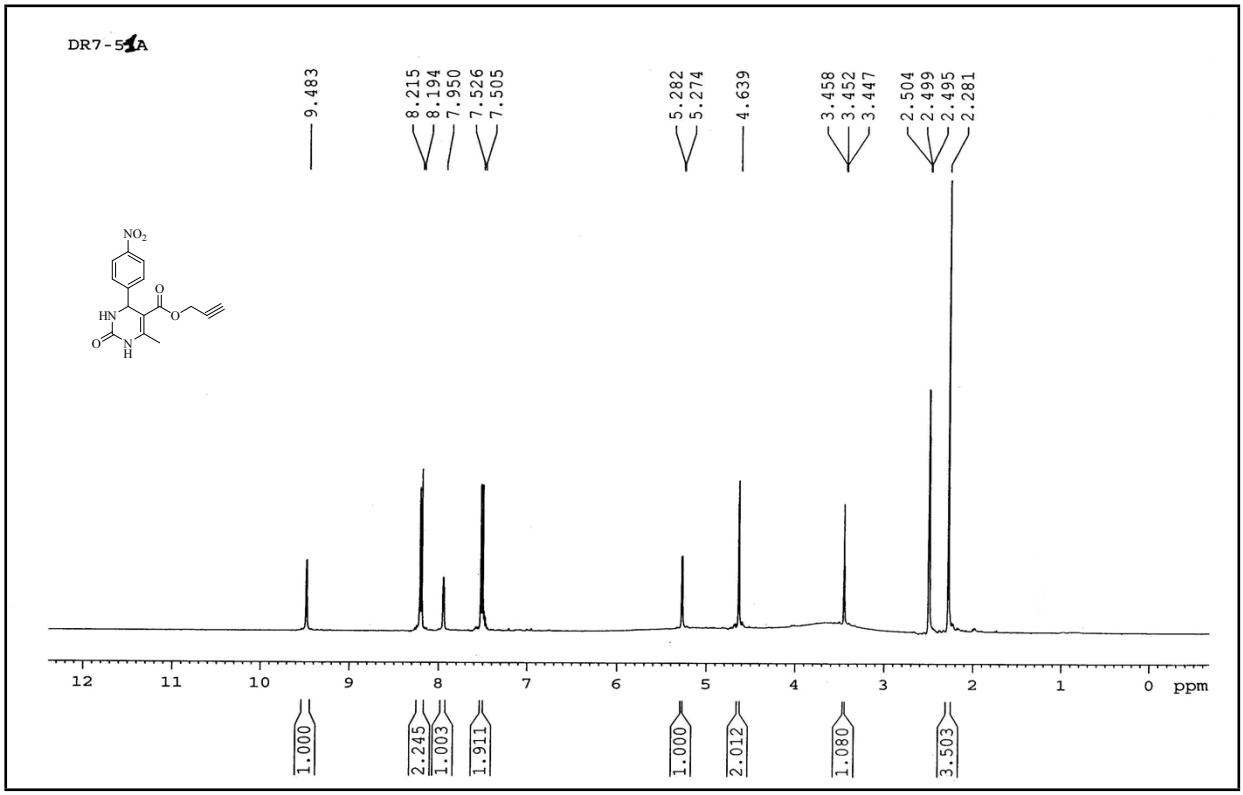
F2 - Processing parameters
SI 32768
SF 100.6128209 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

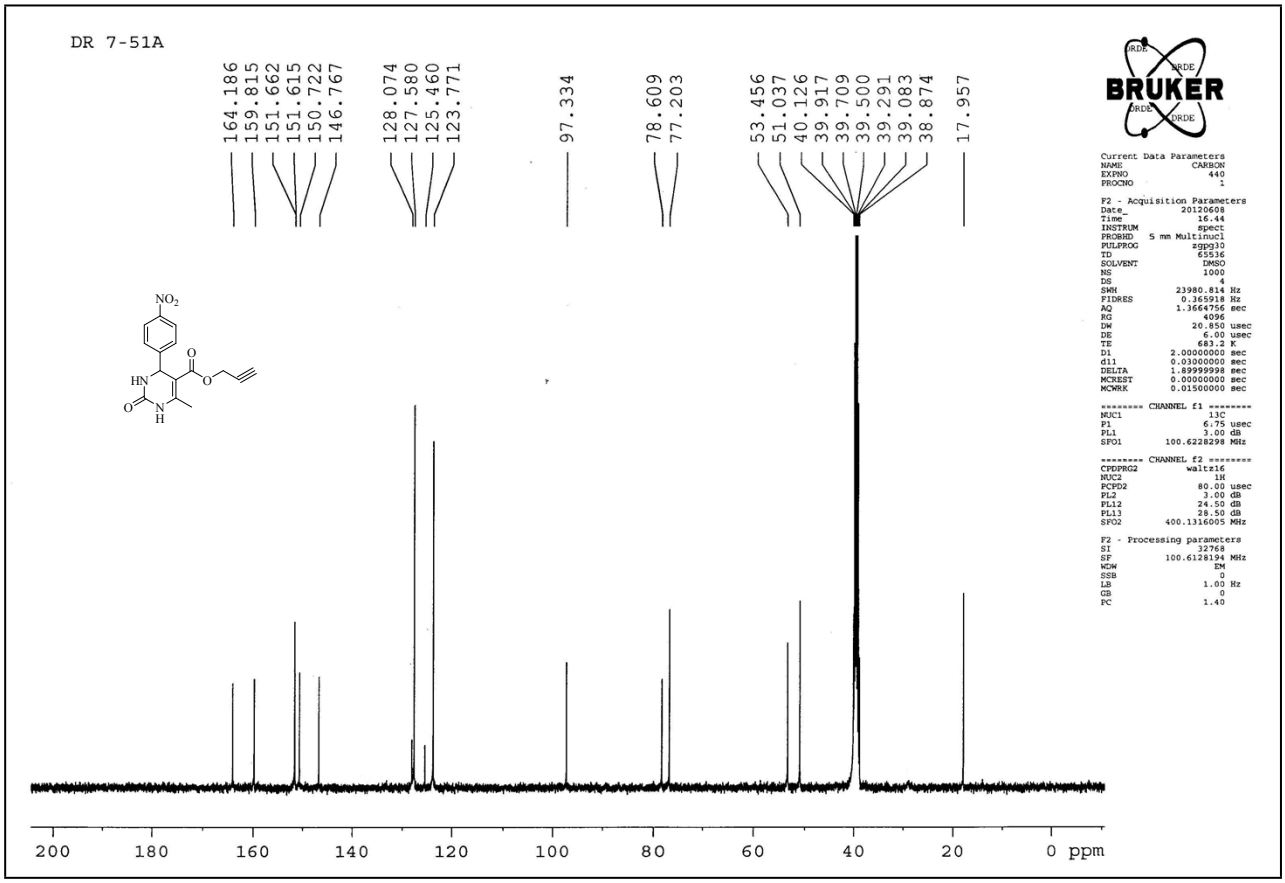

DR7-49A
9.426
7.896
7.715
7.694
7.476
7.456
5.238
5.229
4.648
4.642
3.458
3.452
3.447
3.402
3.390
3.373
2.505
2.500
2.496
2.276
1.000
1.017
2.032
2.122
1.252
2.105
1.444
3.306
ppm

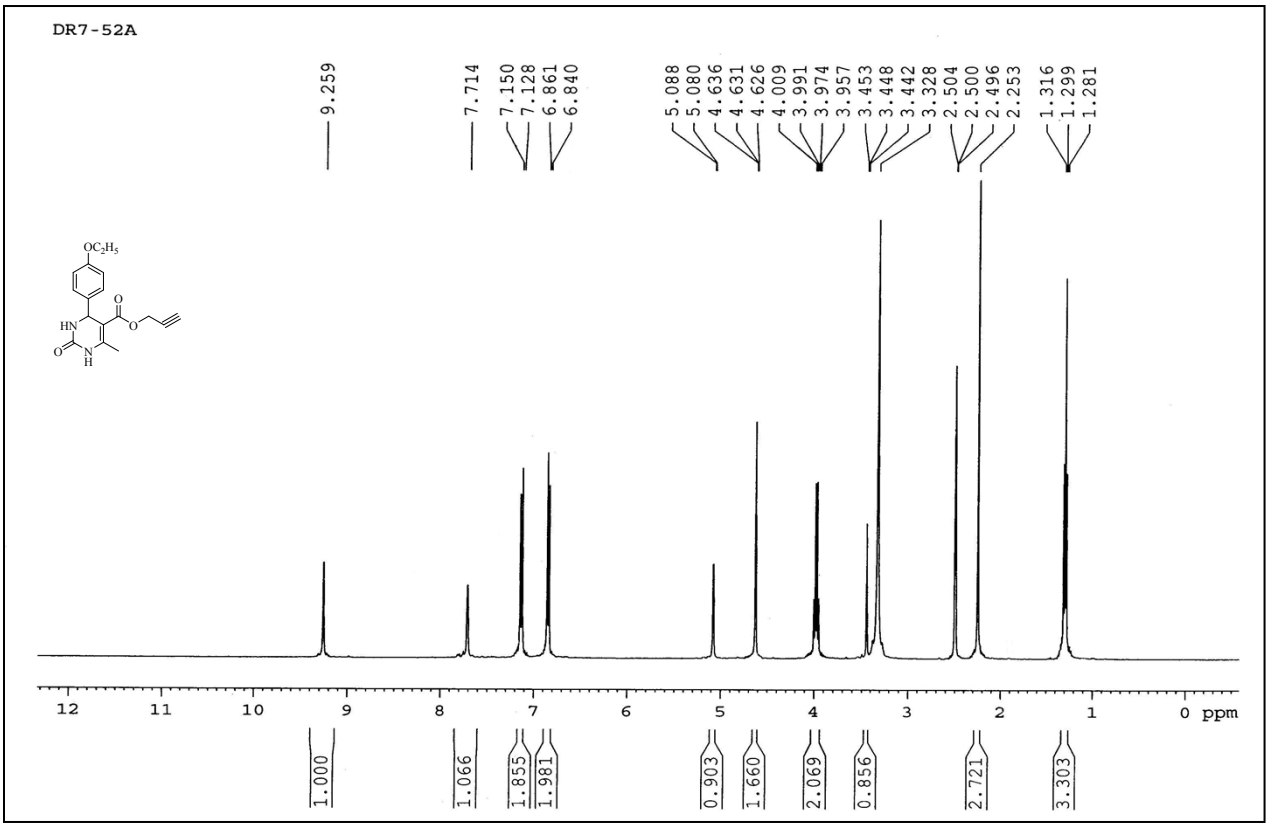

DR7-49A
164.311
159.882
151.842
150.460
148.938
128.205
128.066
127.891
127.071
125.561
125.499
125.466
125.428
125.391
122.858
97.707
78.662
77.164
53.515
51.034
40.133
39.923
39.715
39.506
39.297
39.089
38.880
17.955
BRUKER
Current Data Parameters
NAME CARBON
EXPNO 425
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120523
Time 15.15
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 617
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4597.6
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128165 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

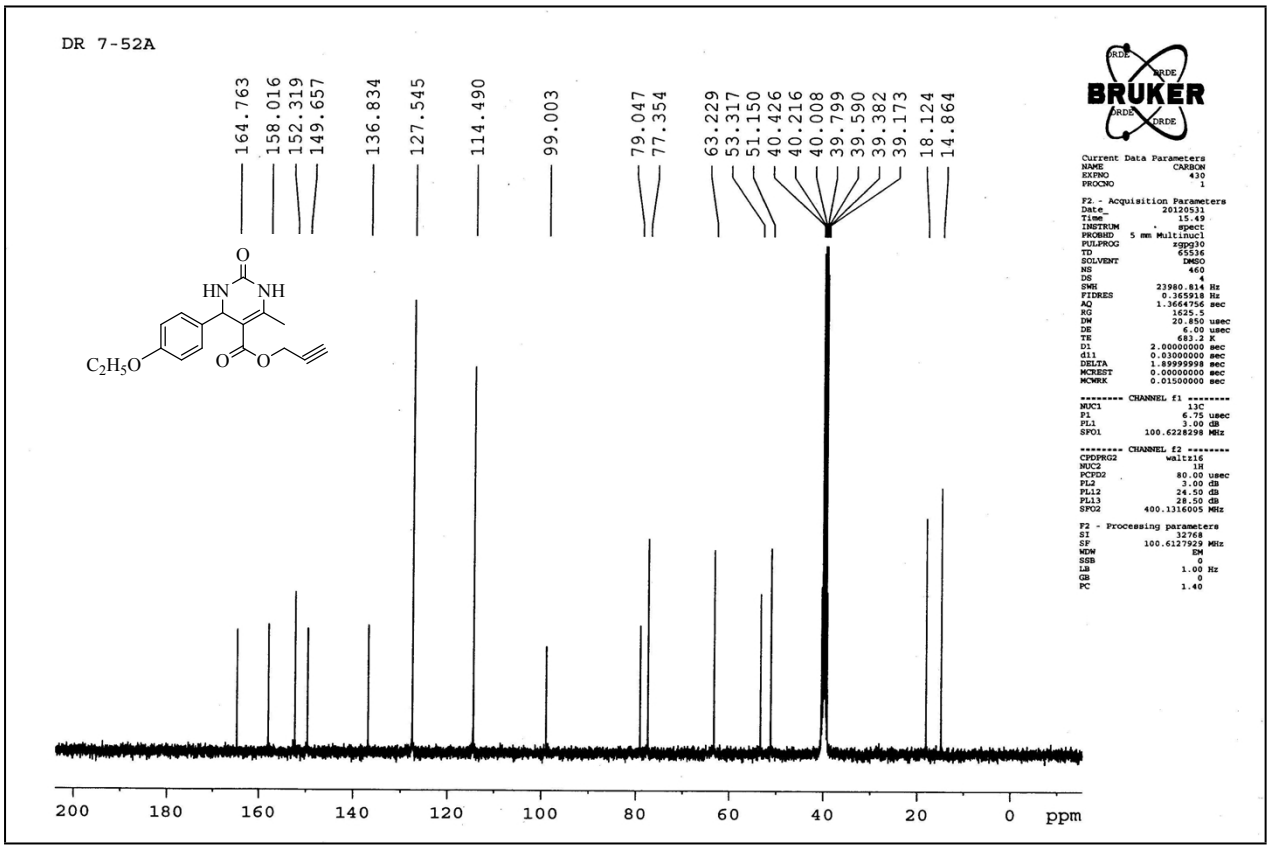

DR7-50A
9.312
7.795
7.339
7.319
7.310
7.302
7.260
7.242
7.229
7.225
5.152
5.143
4.643
4.637
3.457
3.451
3.445
3.375
3.324
2.505
2.500
2.496
2.264
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
1.000
1.018
5.439
1.008
2.016
0.920
3.098

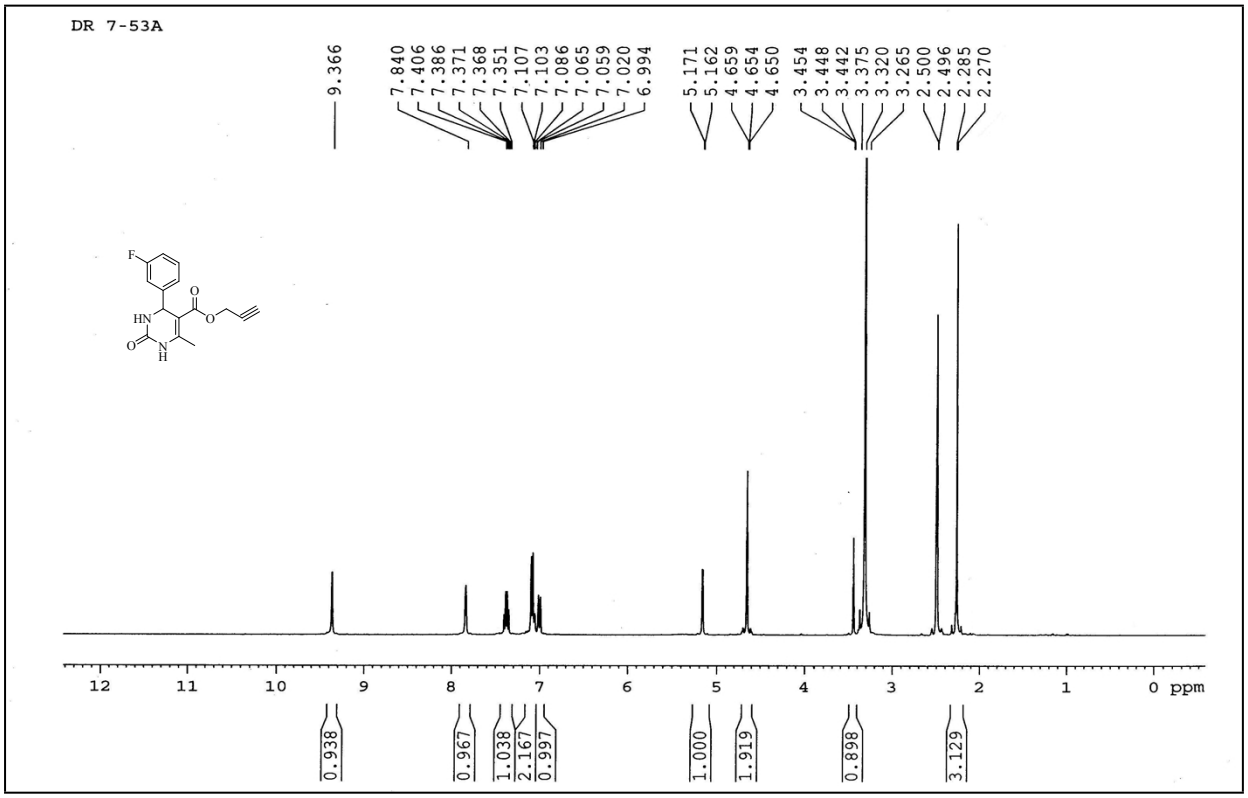

DR7-50A
164.485
152.128
149.739
144.515
128.402
127.307
126.146
98.485
78.750
77.063
53.702
50.944
40.123
39.914
39.706
39.497
39.288
39.079
38.871
17.913
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
Current Data Parameters
NAME CARBON
EXPNO 424
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120522
Time 17.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 891
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1824.6
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128187 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

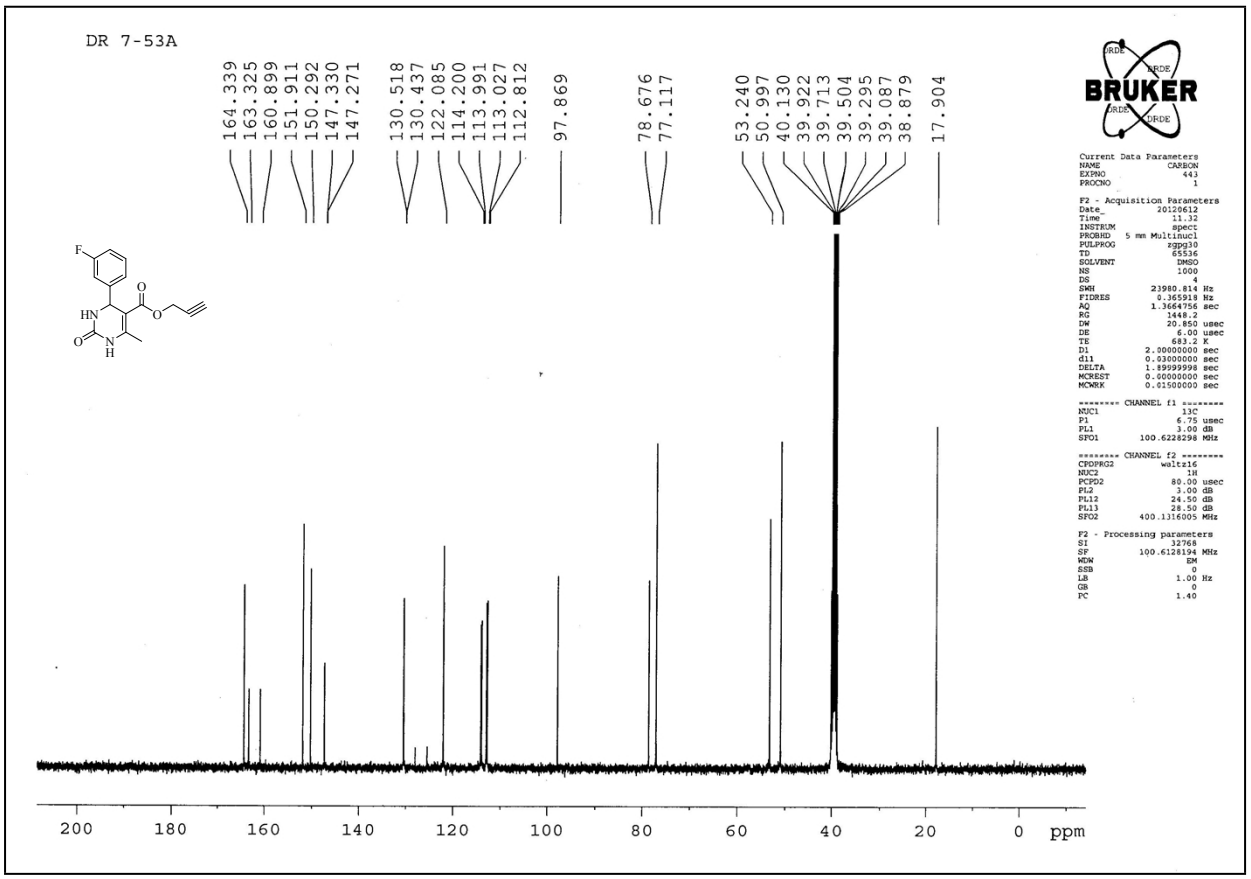

DR7-51A
9.483
8.215
8.194
7.950
7.526
7.505
5.282
5.274
4.639
3.458
3.452
3.447
2.504
2.499
2.495
2.281
1.000
2.245
1.003
1.911
1.000
2.012
1.080
3.503
ppm

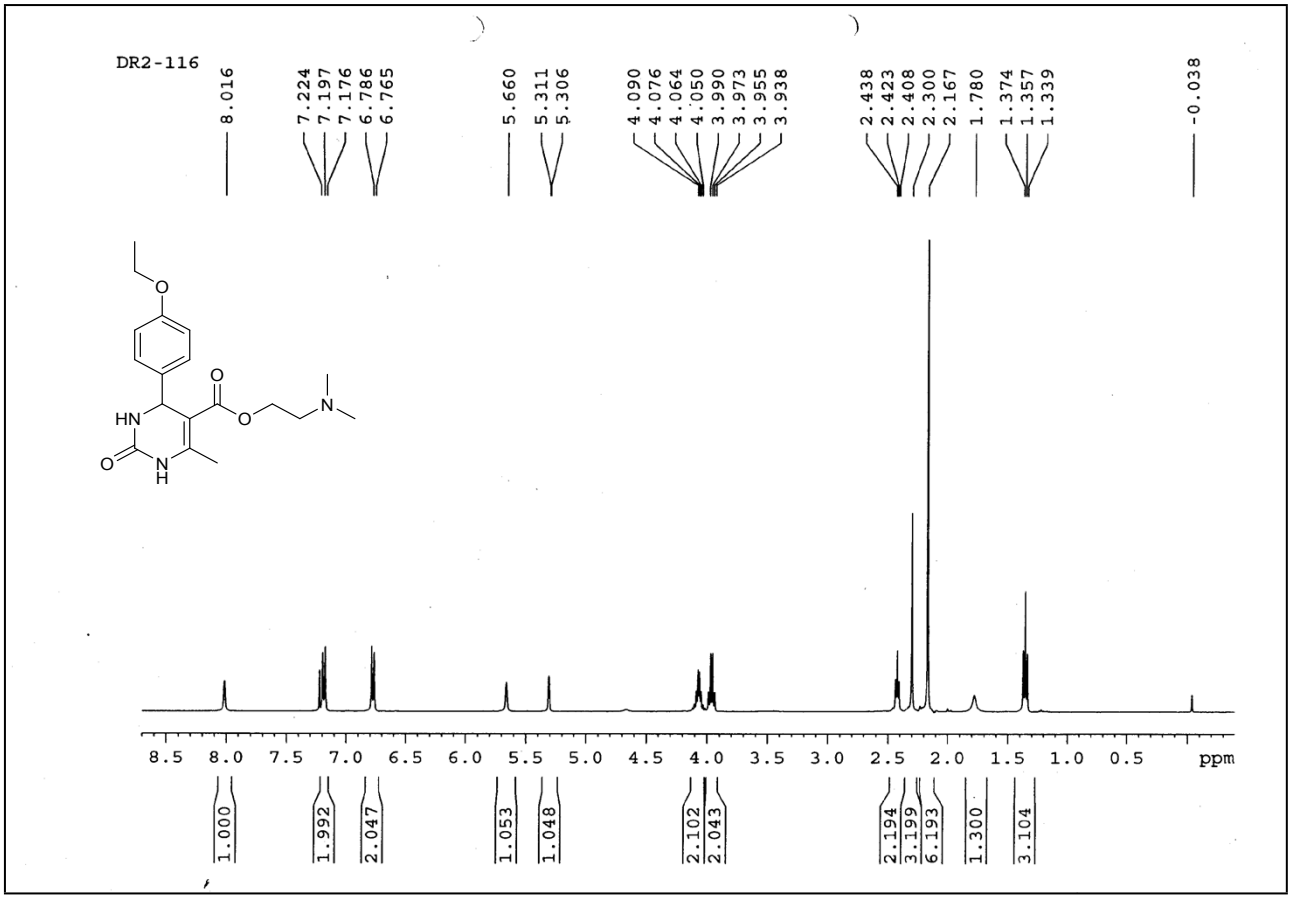

DR 7-51A
164.186
159.815
151.662
151.615
150.722
146.767
128.074
127.580
125.460
123.771
97.334
78.609
77.203
53.456
51.037
40.126
39.917
39.709
39.500
39.291
39.083
38.874
17.957
ppm
BRUKER
Current Data Parameters
NAME CARBON
EXPNO 440
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120608
Time 16.44
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 4096
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128194 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

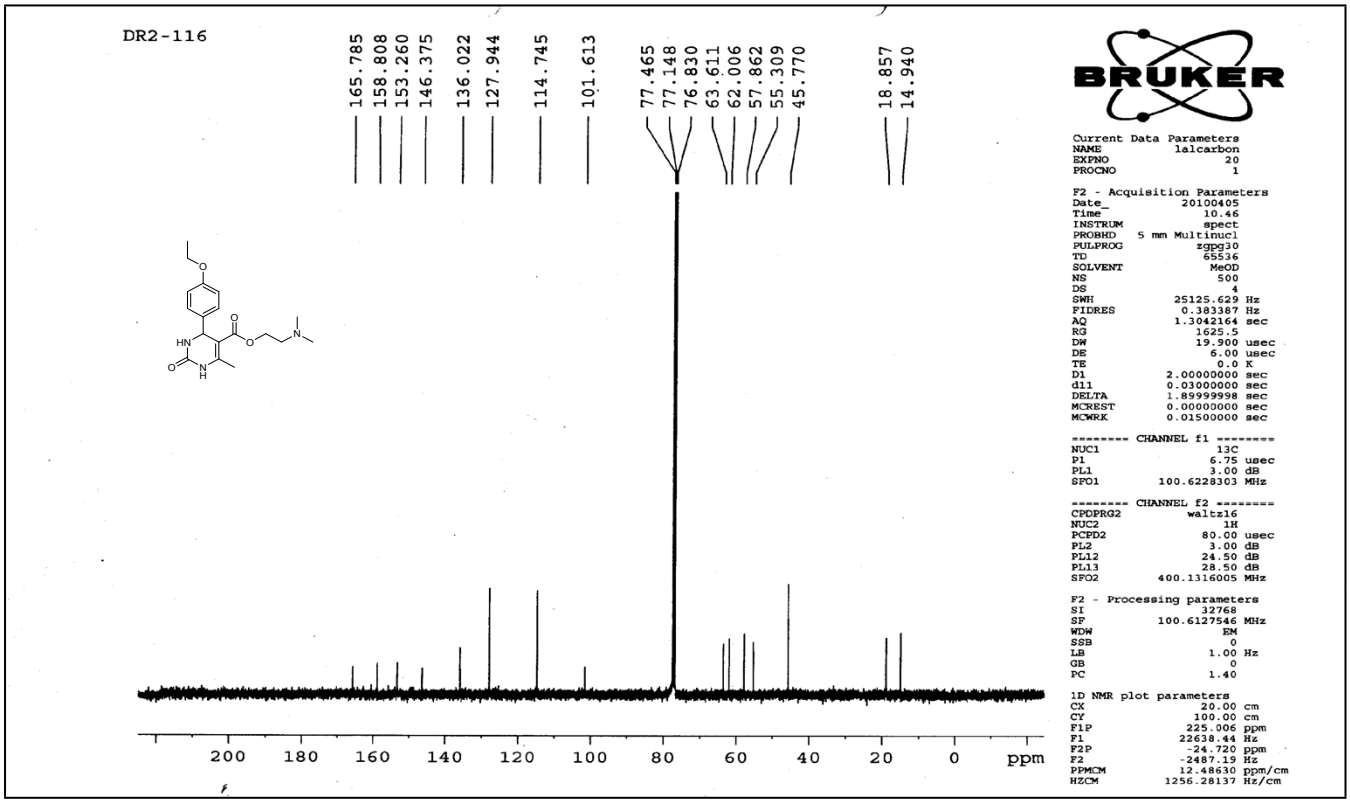

DR7-52A
9.259
7.714
7.150
7.128
6.861
6.840
5.088
5.080
4.636
4.631
4.626
4.009
3.991
3.974
3.957
3.453
3.448
3.442
3.328
2.504
2.500
2.496
2.253
1.316
1.299
1.281
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.000
1.066
1.855
1.981
0.903
1.660
2.069
0.856
2.721
3.303

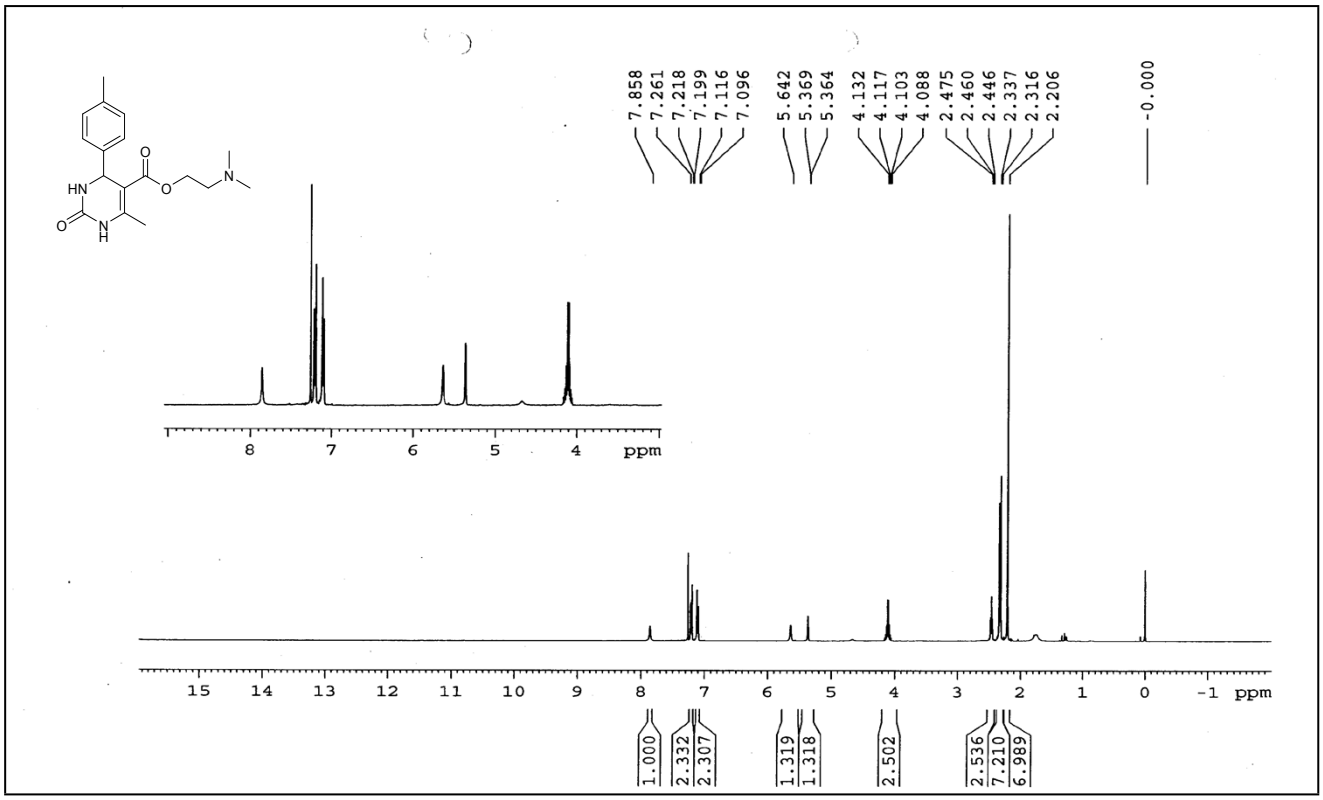

DR 7-52A
164.763
158.016
152.319
149.657
136.834
127.545
114.490
99.003
79.047
77.354
63.229
53.317
51.150
40.426
40.216
40.008
39.799
39.590
39.382
39.173
18.124
14.864
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME CARBON
EXPNO 430
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120531
Time 15.49
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 460
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1625.5
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127929 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

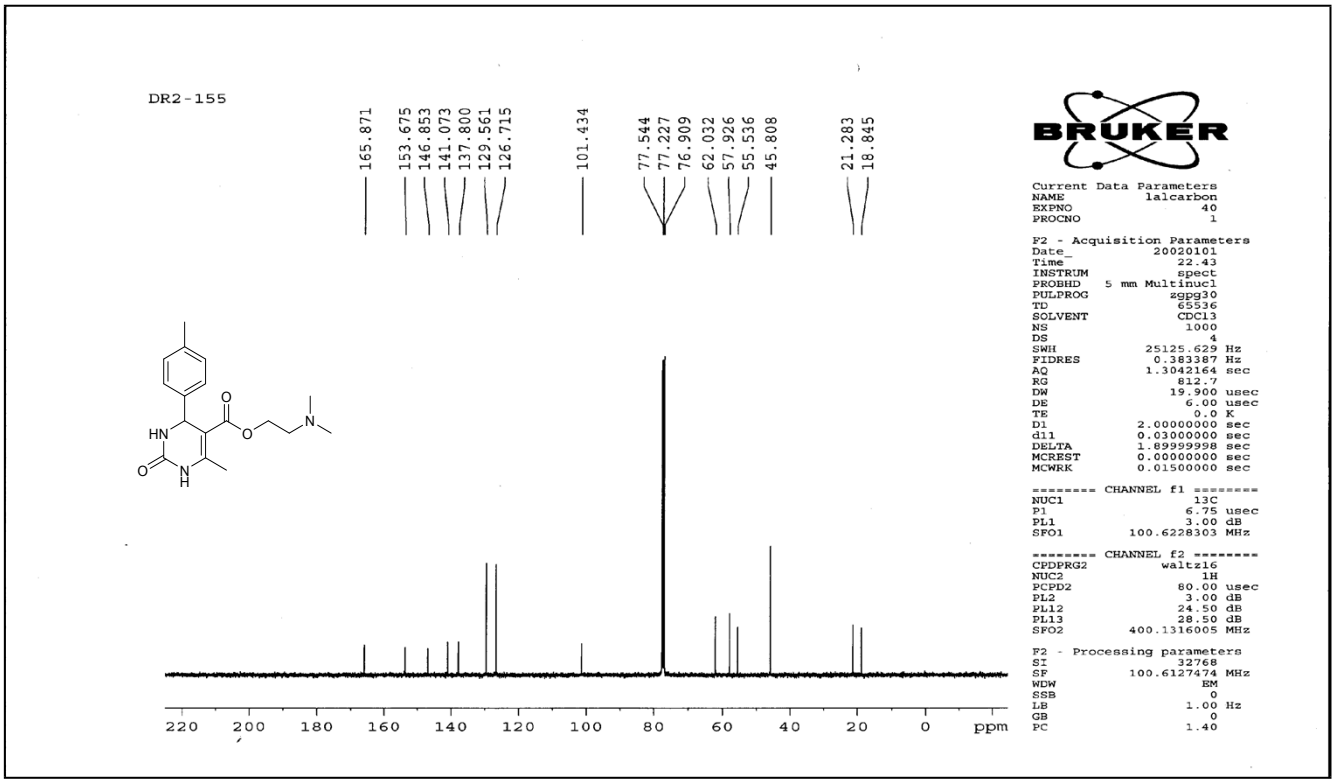

DR 7-53A
9.366
7.840
7.406
7.386
7.371
7.368
7.351
7.107
7.103
7.086
7.065
7.059
7.020
6.994
5.171
5.162
4.659
4.654
4.650
3.454
3.448
3.442
3.375
3.320
3.265
2.500
2.496
2.285
2.270
0.938
0.967
1.038
2.167
0.997
1.000
1.919
0.898
3.129
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm


DR 7-53A
164.339
163.325
160.899
151.911
150.292
147.330
147.271
130.518
130.437
122.085
114.200
113.991
113.027
112.812
97.869
78.676
77.117
53.240
50.997
40.130
39.922
39.713
39.504
39.295
39.087
38.879
17.904
BRUKER
Current Data Parameters
NAME CARBON
EXPNO 443
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120612
Time 11.32
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1000
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 1448.2
DW 20.850 usec
DE 6.00 usec
TE 683.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128194 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


DR2-116
8.016
7.224
7.197
7.176
6.786
6.765
5.660
5.311
5.306
4.090
4.076
4.064
4.050
3.990
3.973
3.955
3.938
2.438
2.423
2.408
2.300
2.167
1.780
1.374
1.357
1.339
-0.038
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
1.992
2.047
1.053
1.048
2.102
2.043
2.194
3.199
6.193
1.300
3.104


DR2-116
165.785
158.808
153.260
146.375
136.022
127.944
114.745
101.613
77.465
77.148
76.830
63.611
62.006
57.862
55.309
45.770
18.857
14.940
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
Current Data Parameters
NAME lalcarbon
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100405
Time 10.46
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 500
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 1625.5
DW 19.900 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
CHANNEL f1
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228303 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 20.00 cm
CY 100.00 cm
F1P 225.006 ppm
F1 22638.44 Hz
F2P -24.720 ppm
F2 -2487.19 Hz
PPMCM 12.48630 ppm/cm
HZCM 1256.28137 Hz/cm


7.858
7.261
7.218
7.199
7.116
7.096
5.642
5.369
5.364
4.132
4.117
4.103
4.088
2.475
2.460
2.446
2.337
2.316
2.206
-0.000
ppm
1.000
2.332
2.307
1.319
1.318
2.502
2.536
7.210
6.989


DR2-155
165.871
153.675
146.853
141.073
137.800
129.561
126.715
101.434
77.544
77.227
76.909
62.032
57.926
55.536
45.808
21.283
18.845
ppm
BRUKER
Current Data Parameters
NAME lalcarbon
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20020101
Time 22.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1000
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 812.7
DW 19.900 usec
DE 6.00 usec
TE 0.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 6.75 usec
PL1 3.00 dB
SFO1 100.6228303 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 24.50 dB
PL13 28.50 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127474 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40