Electronic Supplementary Material (ESI) for Green Chemistry.

A Protocol for Ligand Free Suzuki-Miyaura Cross-Coupling Reactions in WEB at Room Temperature

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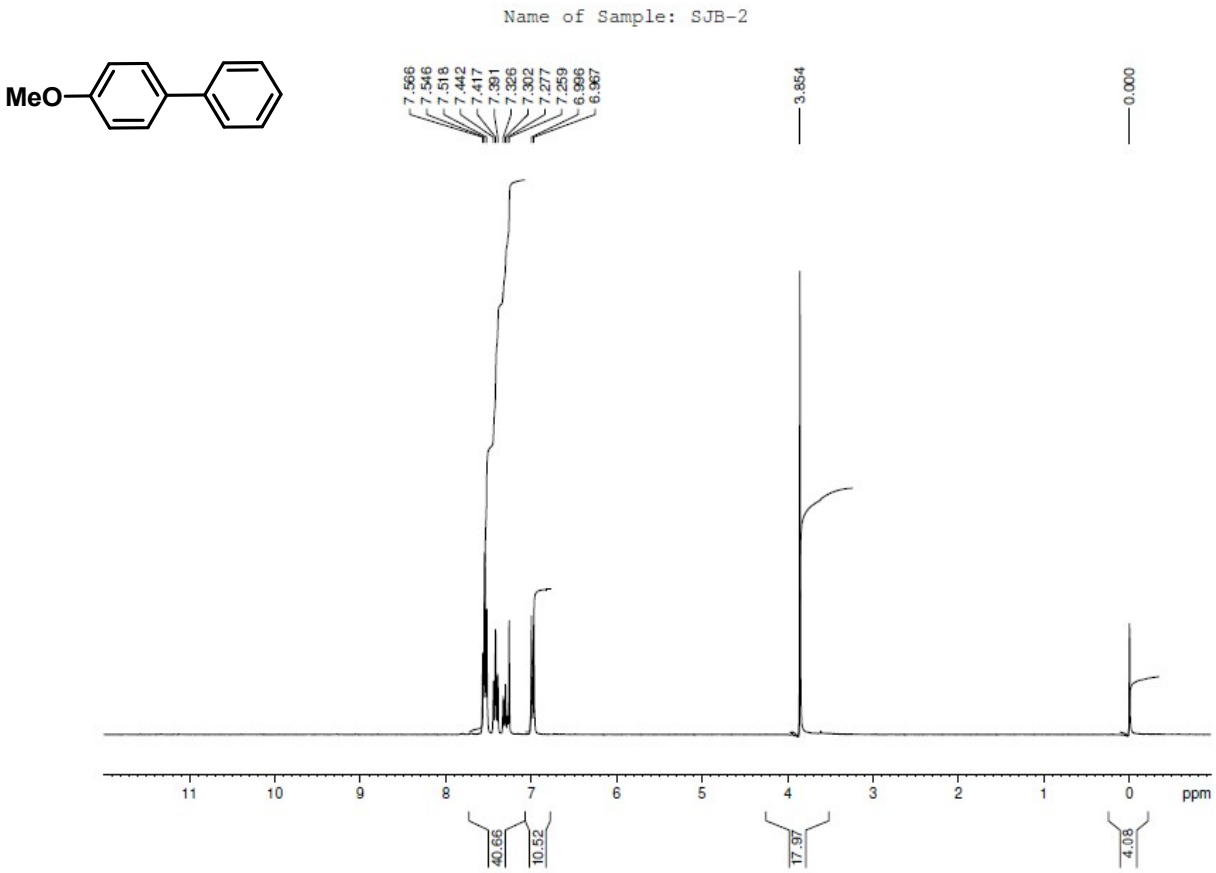
Supporting Information

Contents

General: Starting materials and solvents were purchased from common commercial sources and were used without additional purification. ^{1}H NMR spectra were recorded at 500 MHz or 300 MHz using TMS as internal standard. Infrared spectra were obtained from a FTIR spectrometer.

General experimental procedure for Suzuki reaction: A mixture of aryl halide (1 mmol), arylboronic acid (1.2 mmol) and $Pd(OAc)_2$ (0.5 mol%) in WEB (3 mL) was stirred for the indicated time at room temperature. Afterward, the reaction solution was extracted four times with diethyl ether (4 × 10 mL). The products were purified by column chromatography over silica gel using *n*-hexane/ethyl acetate (9:1 v/v) to get the desired coupling products. The products were characterized by ^{1}H NMR and GC-MS.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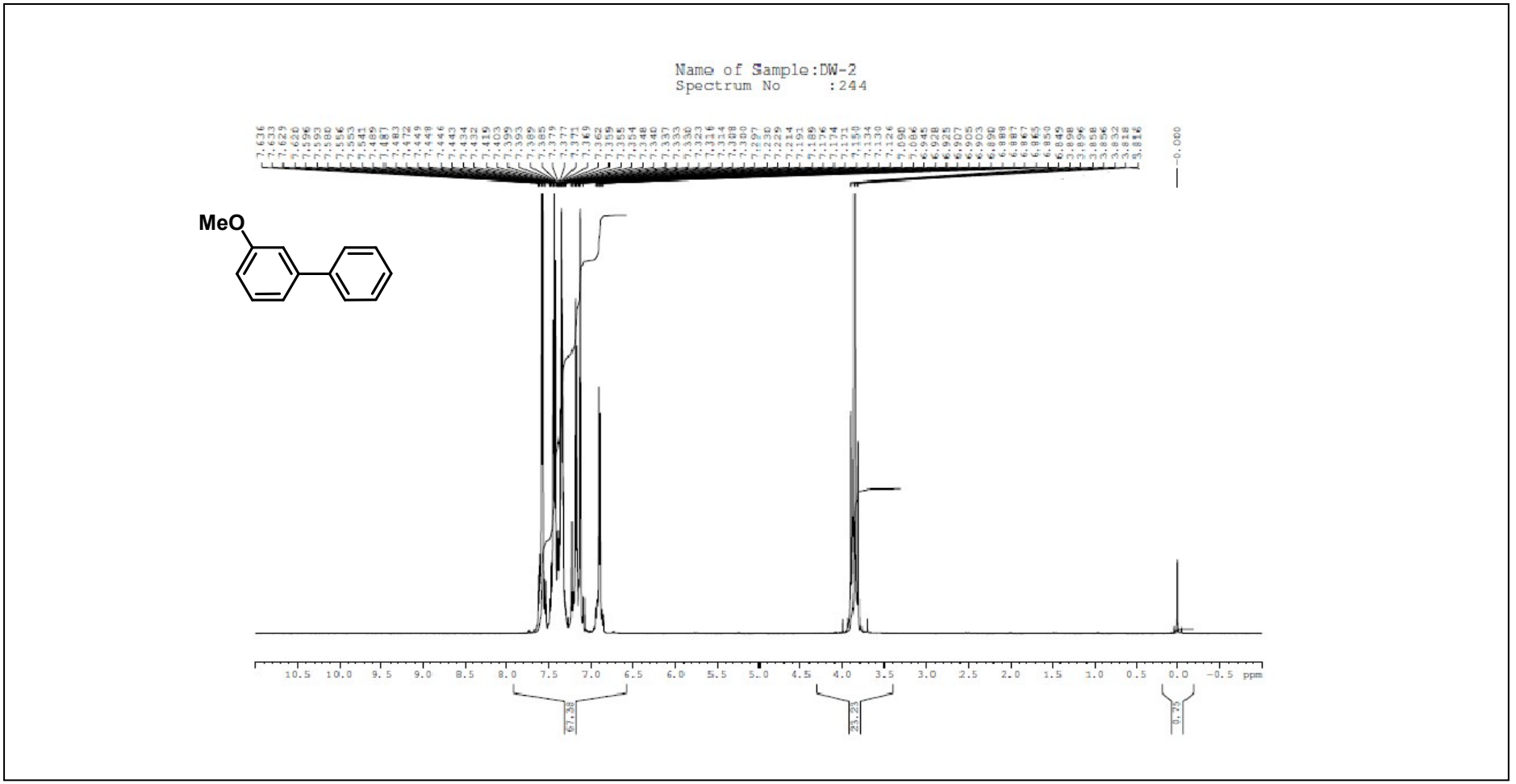
7.546
7.518
7.442
7.417
7.391
7.326
7.302
7.277
7.259
6.996
6.967
3.854
0.000
11
10
9
8
7
6
5
4
3
2
1
0
ppm
40.66
10.52
17.97
4.08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MeO
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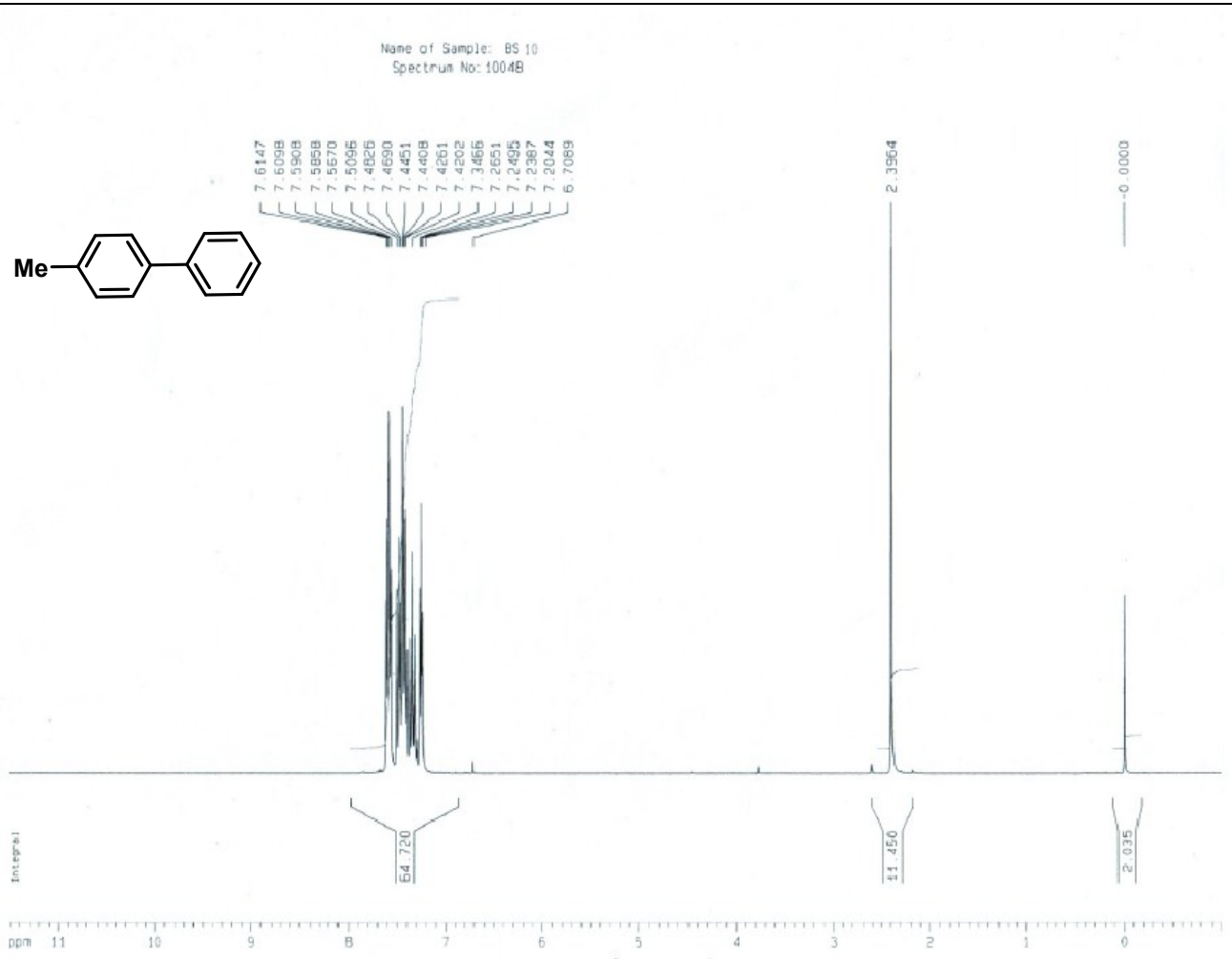
67.38
25.23
6.75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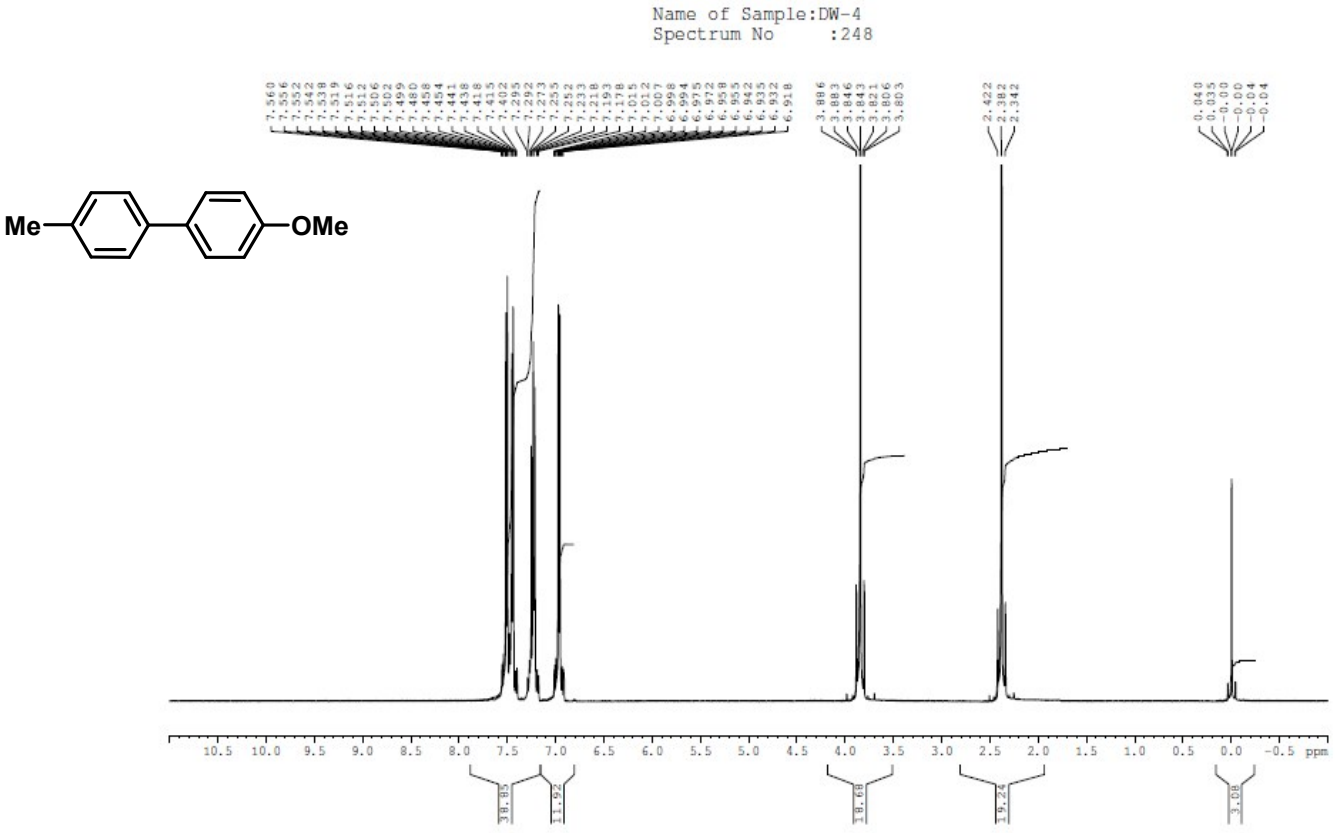
7.5908
7.5858
7.5670
7.5095
7.4826
7.4690
7.4451
7.4408
7.4261
7.4202
7.3465
7.2651
7.2495
7.2387
7.2044
6.7069
2.3964
0.0000
Integral
64.720
11.450
2.035
ppm 11 10 9 8 7 6 5 4 3 2 1 0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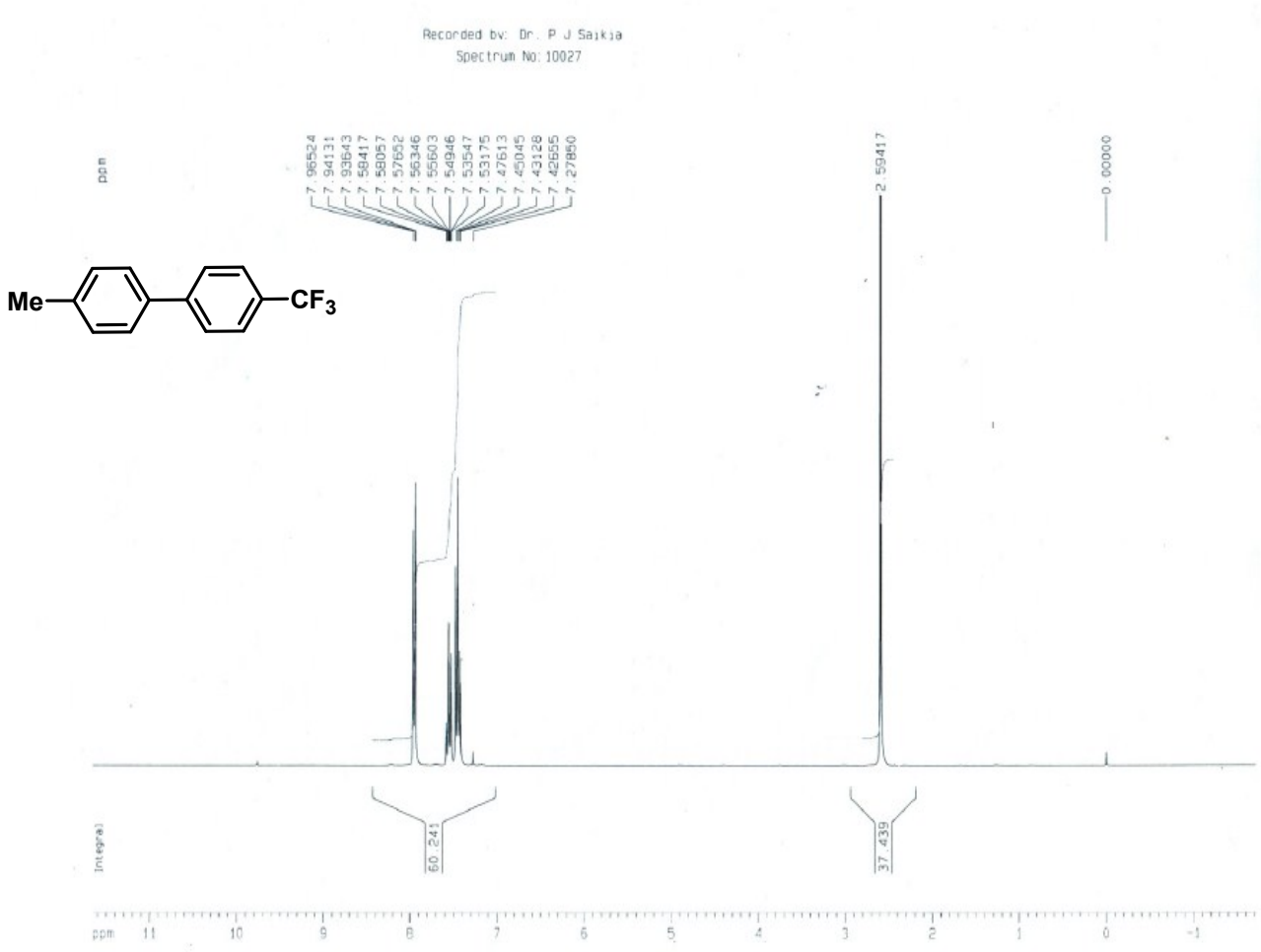
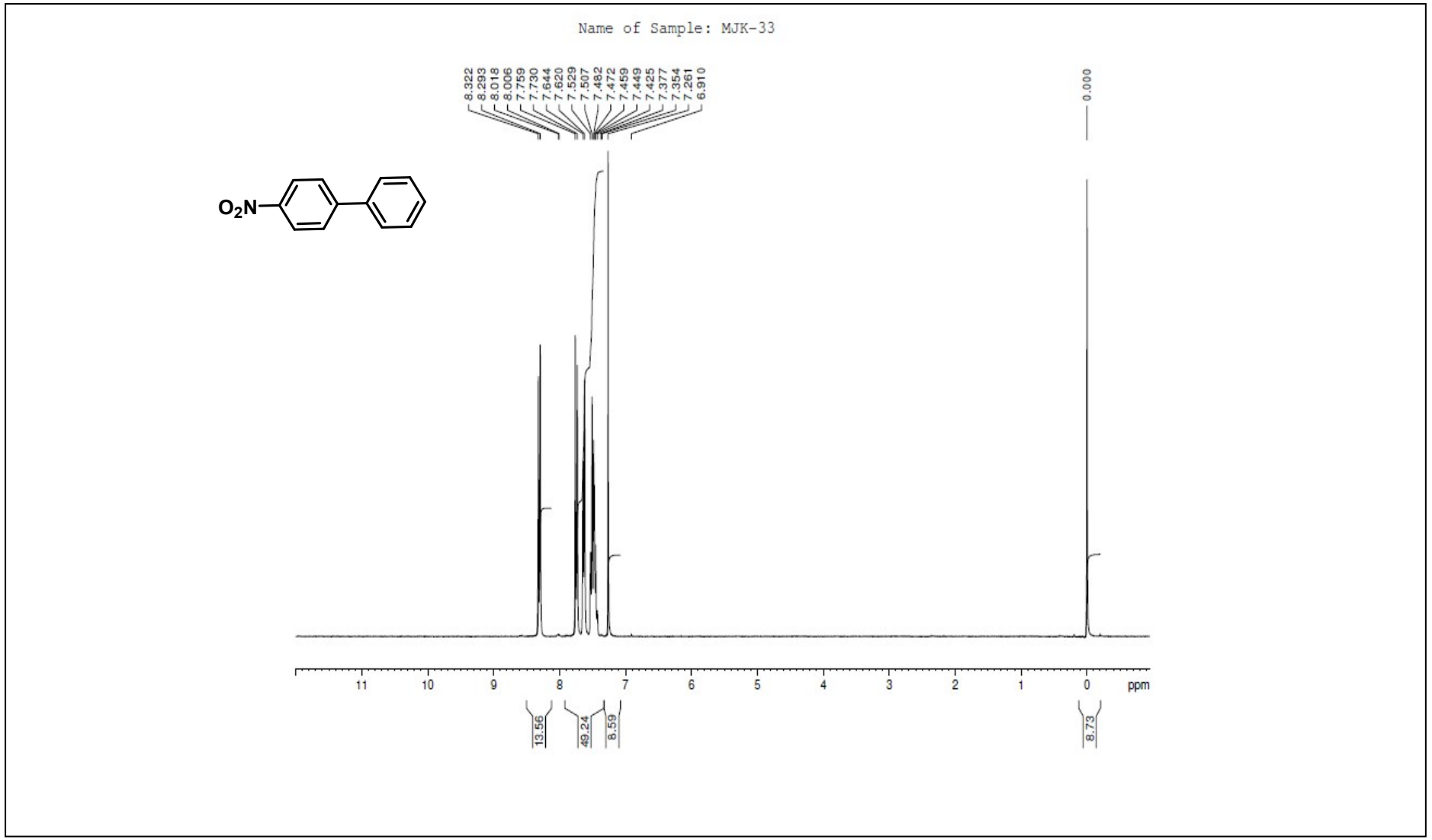
Me
OMe
7.560
7.556
7.552
7.542
7.538
7.519
7.516
7.512
7.506
7.502
7.492
7.480
7.458
7.454
7.441
7.438
7.418
7.415
7.402
7.295
7.292
7.293
7.255
7.252
7.233
7.218
7.193
7.178
7.015
7.012
7.007
6.998
6.994
6.975
6.972
6.958
6.955
6.942
6.935
6.932
6.918
3.886
3.883
3.846
3.843
3.821
3.806
3.803
2.422
2.382
2.342
0.040
0.035
0.00
0.00
-0.04
-0.04
10.5 10.0 9.5 9.0 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 0.0 -0.5 ppm
38.85
11.92
18.68
19.24
3.08


Recorded by: Dr. P J Saikia
Spectrum No: 10027
ppm
7.96524
7.94131
7.93643
7.58417
7.58057
7.57652
7.56346
7.55603
7.54946
7.53547
7.53175
7.47613
7.45045
7.43128
7.42655
7.27850
2.59417
0.00000
Me
CF3
Integral
60.241
37.439
ppm
11
10
9
8
7
6
5
4
3
2
1
0
-1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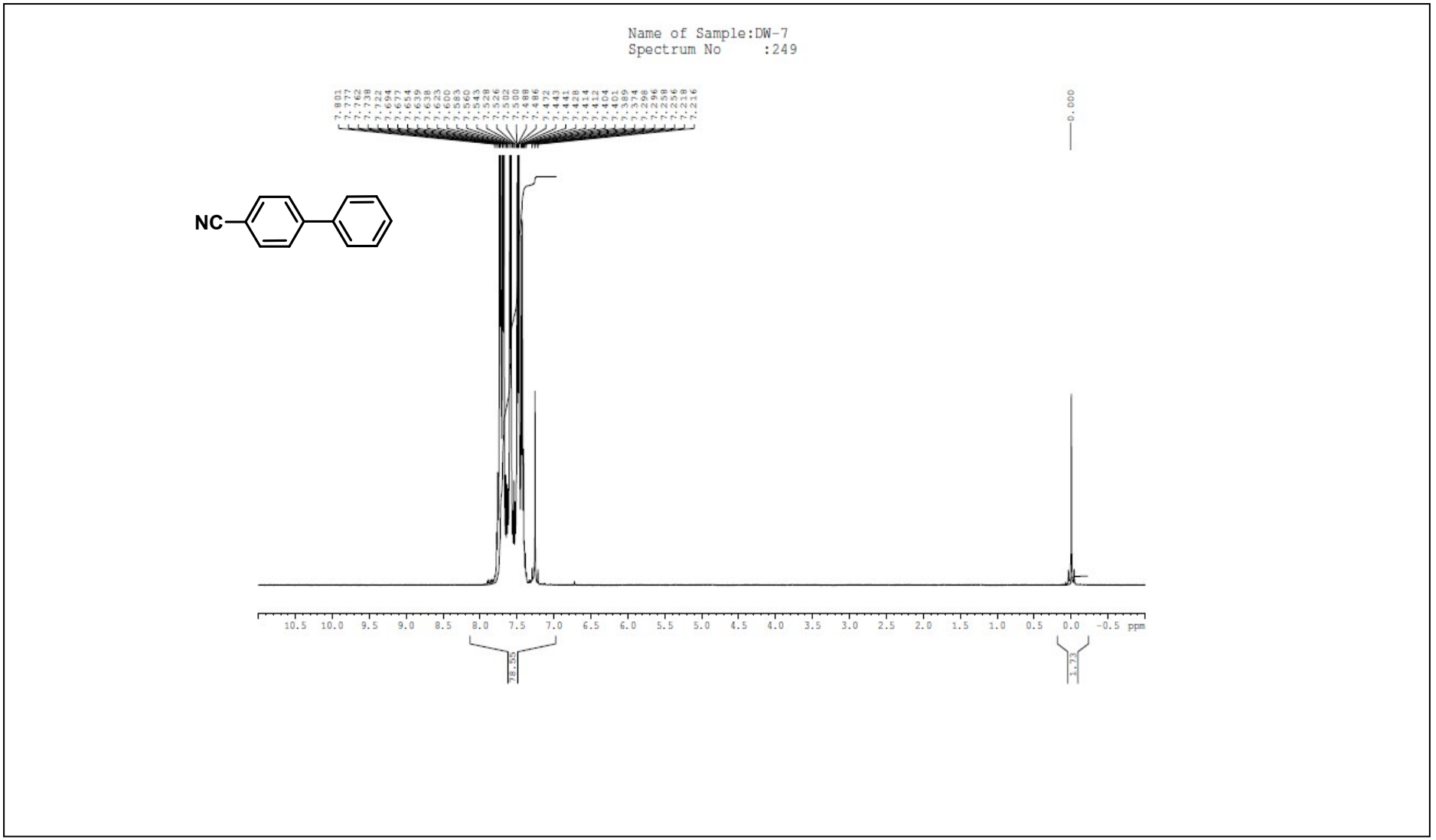
8.293
8.018
8.006
7.759
7.730
7.644
7.620
7.529
7.507
7.482
7.472
7.459
7.449
7.425
7.377
7.354
7.261
6.910
0.000
O2N
11
10
9
8
7
6
5
4
3
2
1
0
ppm
13.56
49.24
8.59
8.73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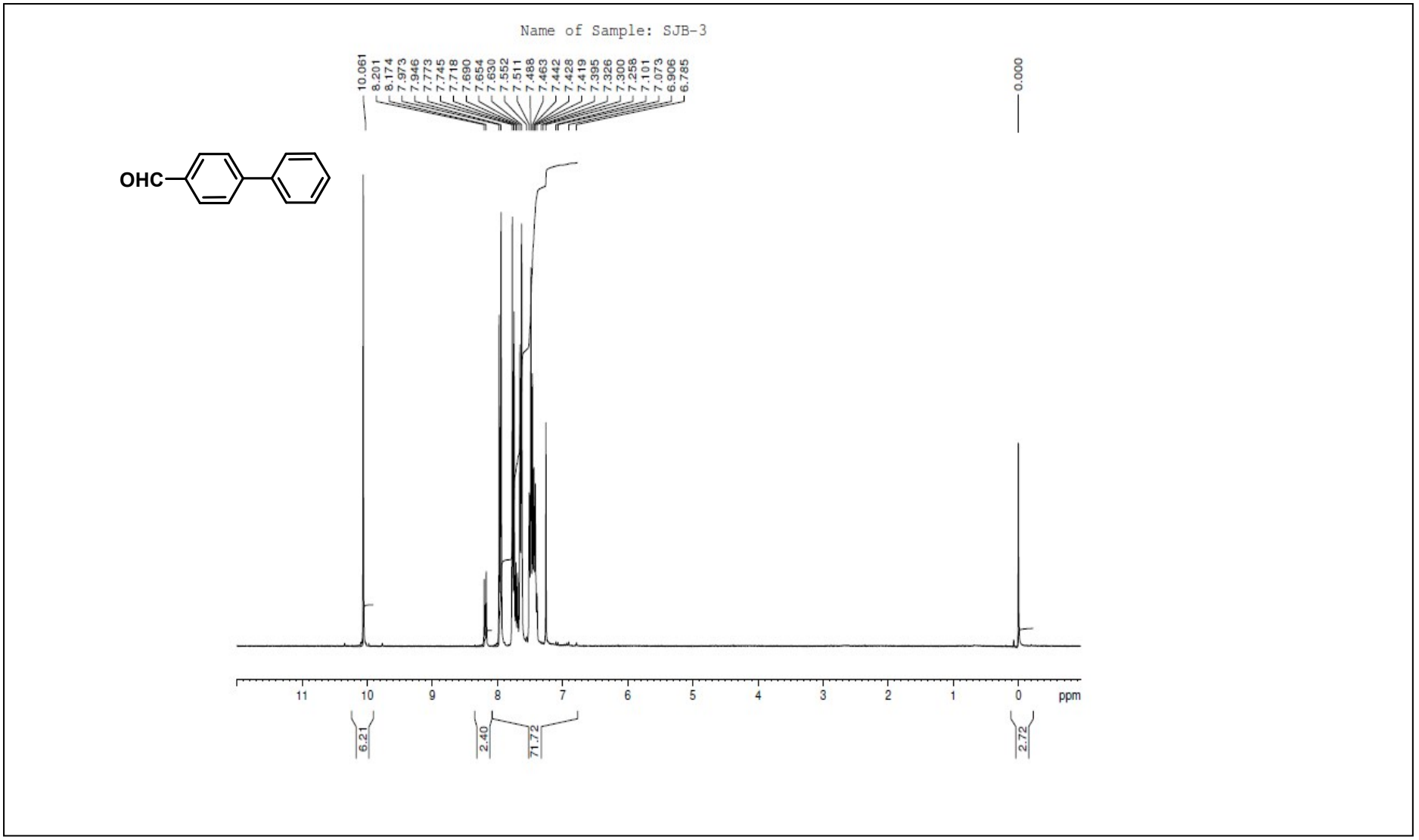
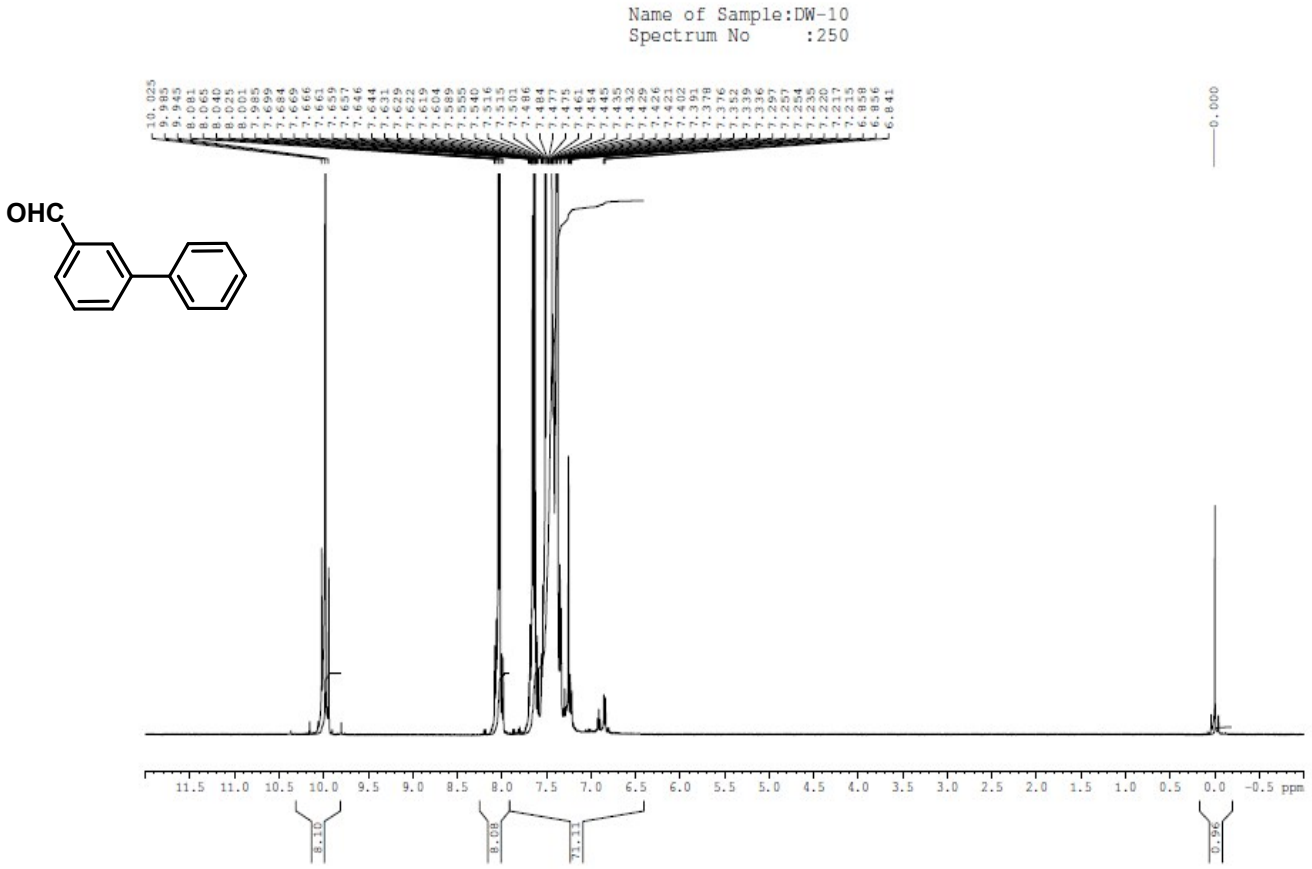
Spectrum No :249
NC
7.801
7.797
7.742
7.738
7.722
7.654
7.677
7.654
7.639
7.638
7.623
7.600
7.583
7.560
7.543
7.528
7.526
7.502
7.500
7.488
7.486
7.472
7.443
7.441
7.428
7.414
7.412
7.404
7.401
7.389
7.374
7.298
7.296
7.258
7.256
7.218
7.216
0.000
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
78.55
1.73


Name of Sample: SJB-3
10.061
8.201
8.174
7.973
7.946
7.773
7.745
7.718
7.690
7.654
7.630
7.552
7.511
7.488
7.463
7.442
7.428
7.419
7.395
7.326
7.300
7.258
7.101
7.073
6.906
6.785
0.000
OHC
11
10
9
8
7
6
5
4
3
2
1
0
ppm
6.21
2.40
71.72
2.72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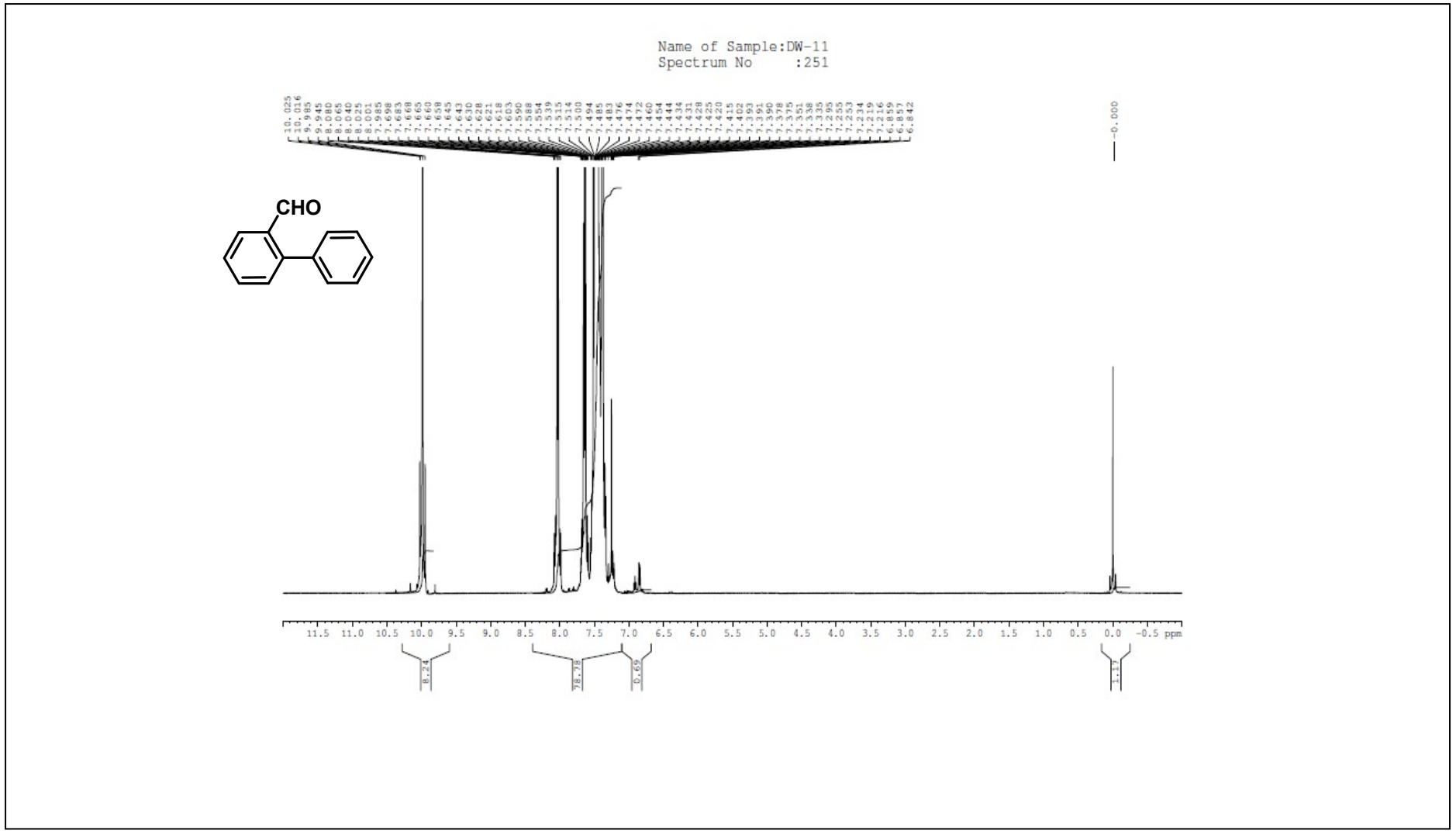
OHC
10.025
9.985
9.945
8.081
8.065
8.040
8.025
8.001
7.985
7.699
7.684
7.669
7.666
7.659
7.657
7.646
7.644
7.631
7.629
7.622
7.619
7.604
7.589
7.555
7.540
7.516
7.513
7.501
7.486
7.484
7.477
7.475
7.461
7.454
7.445
7.435
7.432
7.429
7.426
7.421
7.402
7.391
7.378
7.376
7.352
7.339
7.336
7.297
7.257
7.254
7.235
7.220
7.217
7.215
6.858
6.856
6.841
0.000
11.5 11.0 10.5 10.0 9.5 9.0 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 0.0 -0.5 ppm
8.10
8.08
13.13
0.96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CHO
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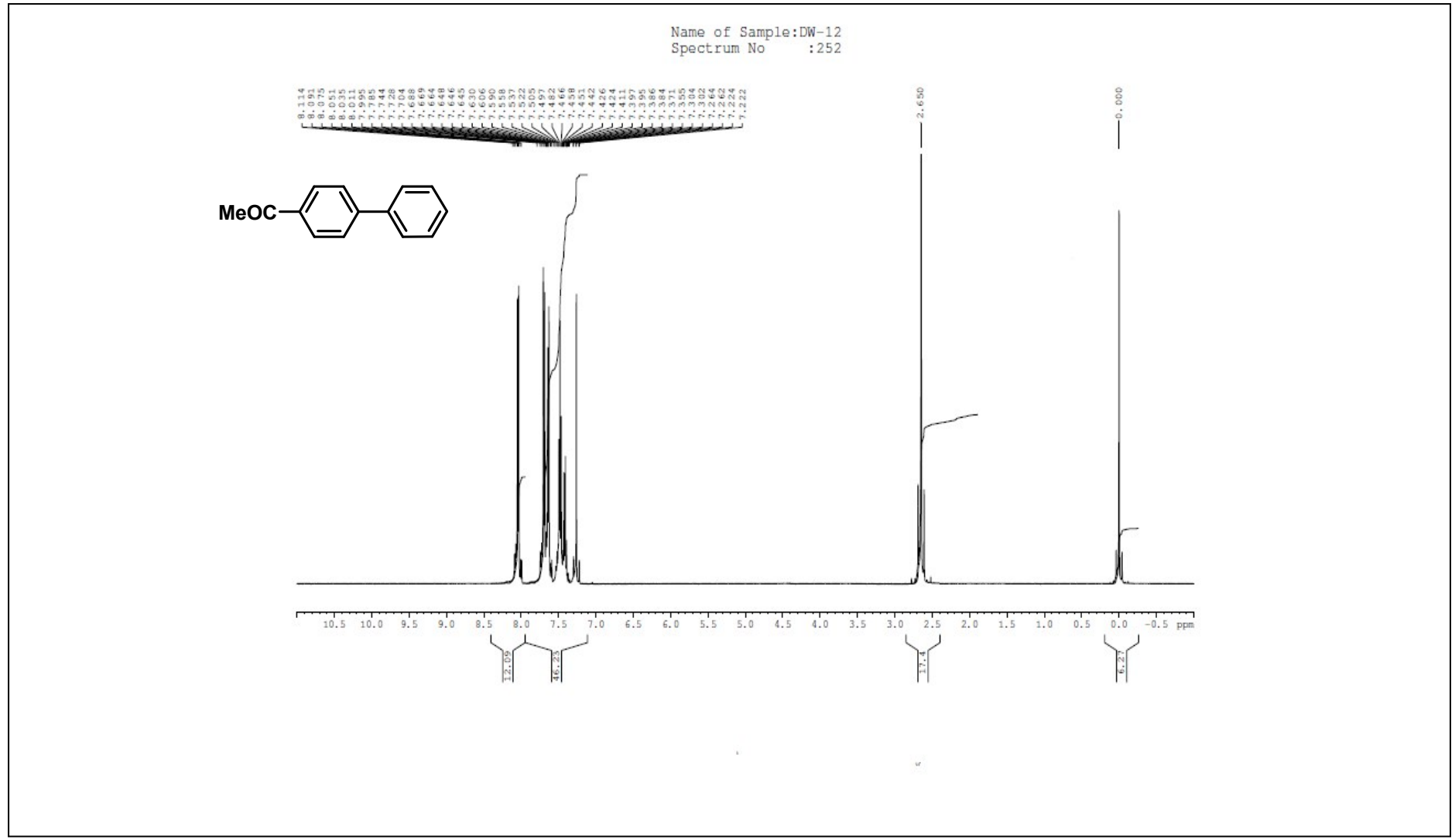
8.24
78.78
0.69
3.17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MeOC
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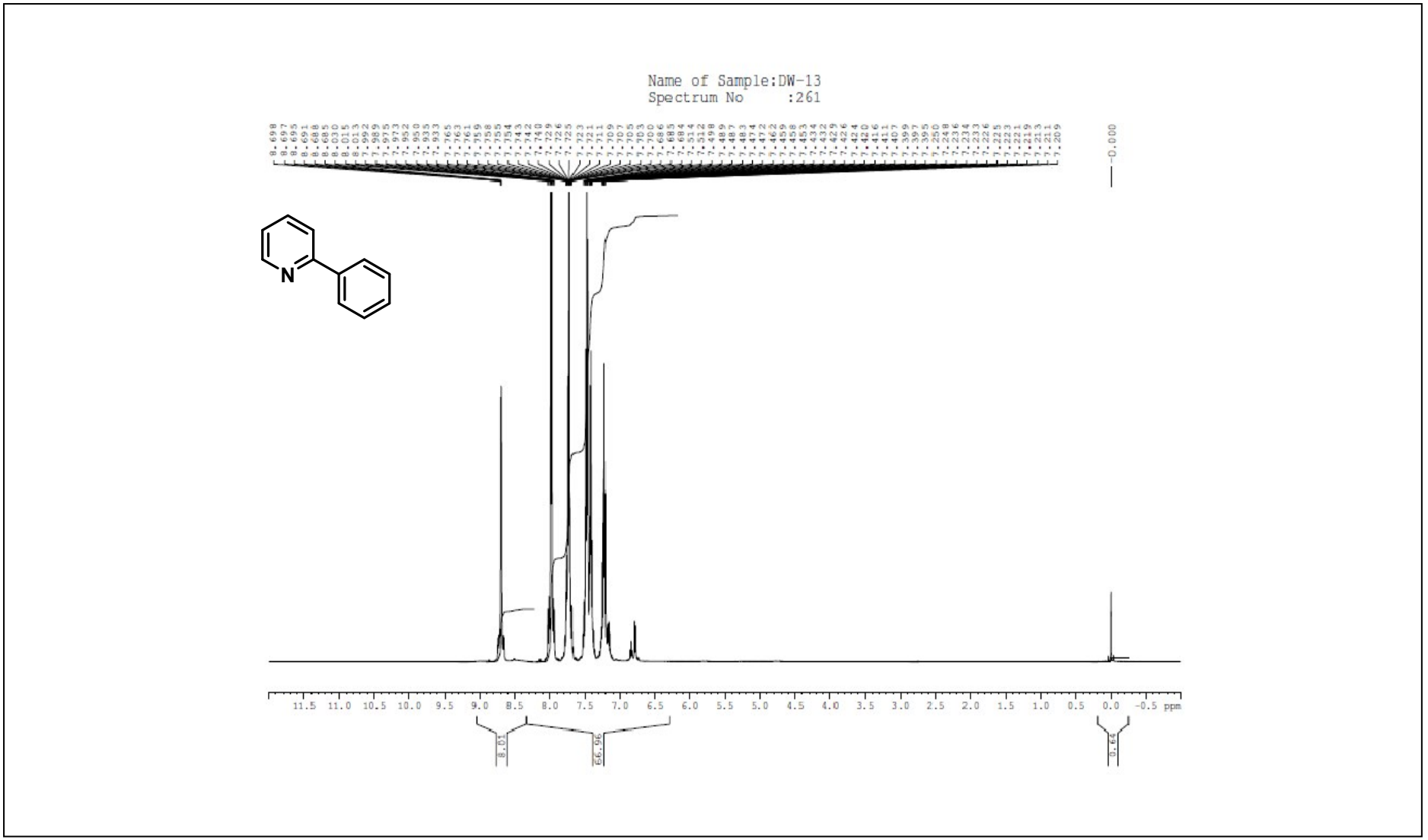
0.000
12.00
46.25
17.4
6.27
ppm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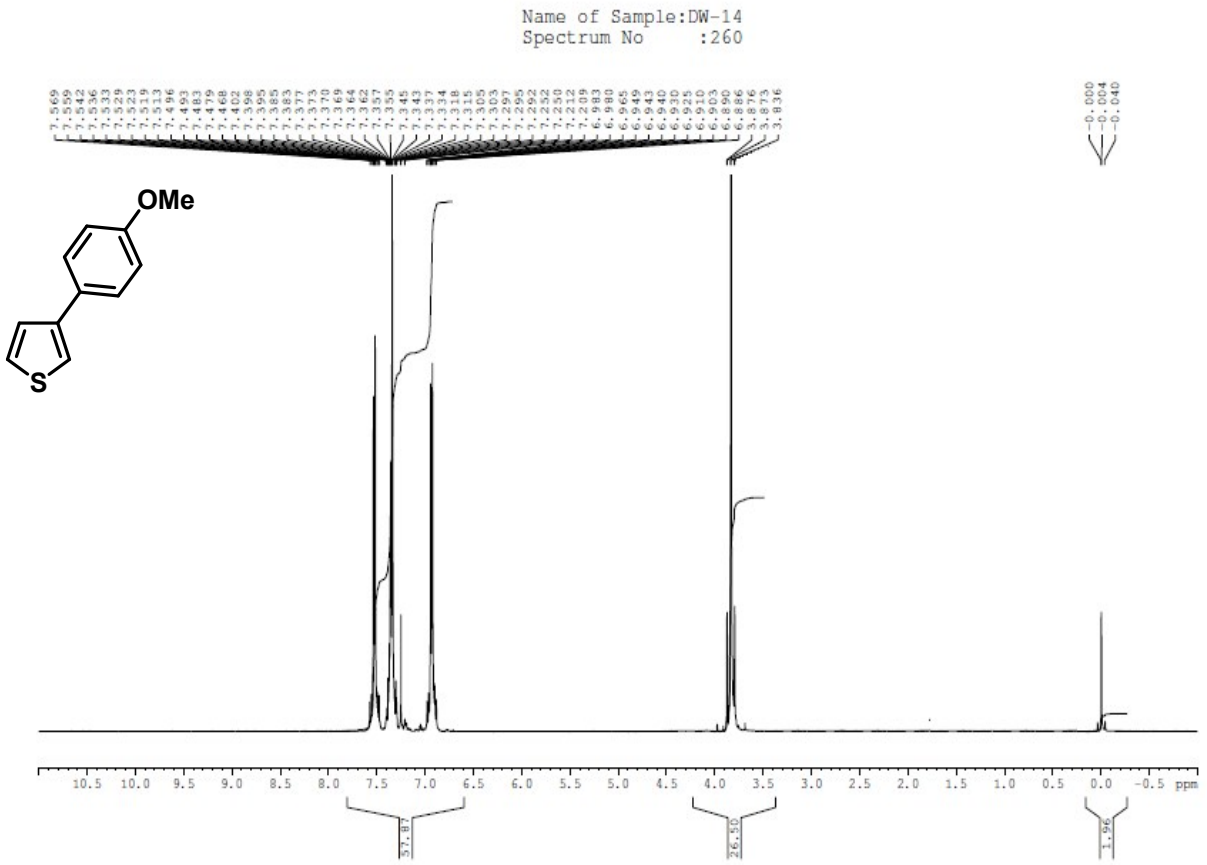
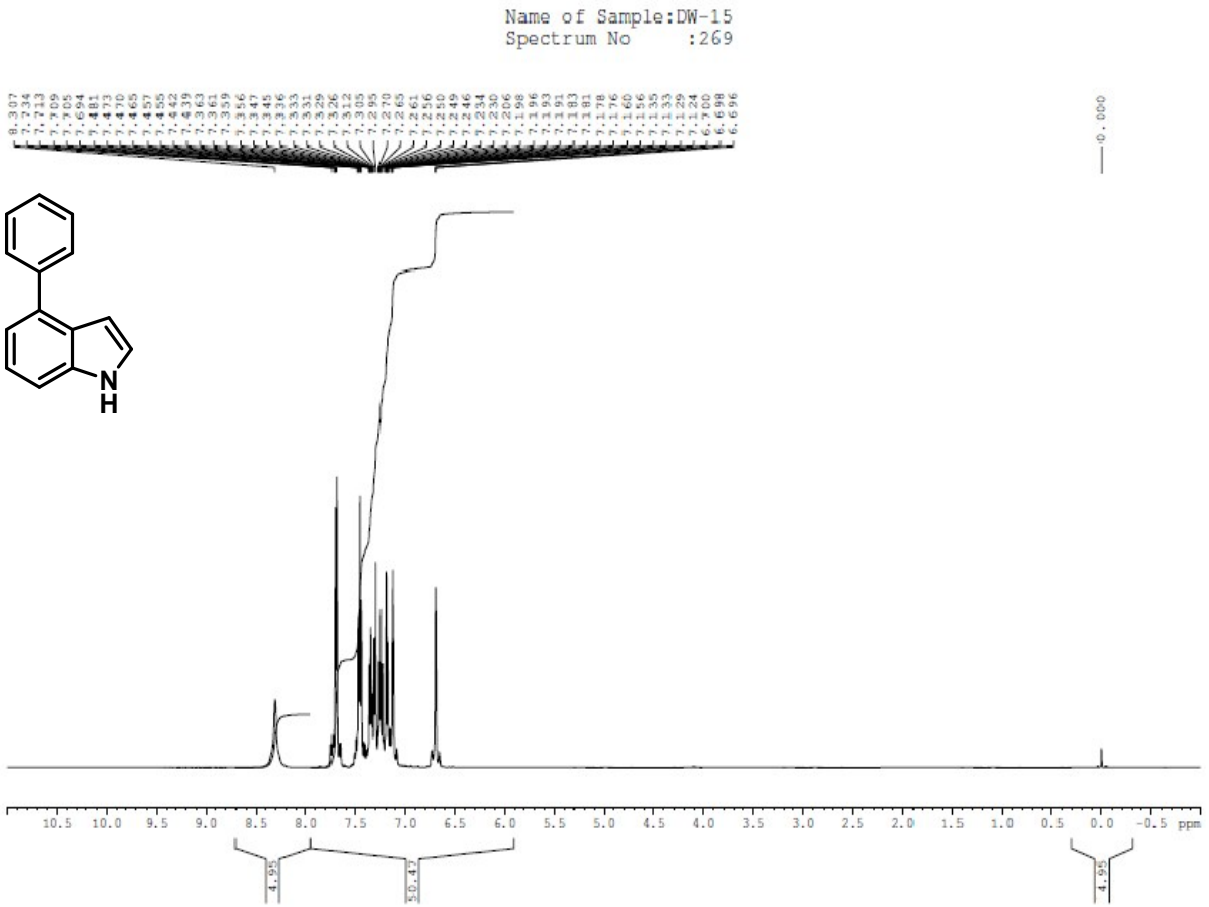
N
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 ppm
8.01
66.96
3.66


Name of Sample:DW-14
Spectrum No :260
OMe
S
ppm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7.713
7.709
7.705
7.694
7.481
7.473
7.470
7.465
7.457
7.455
7.442
7.439
7.363
7.361
7.359
7.356
7.347
7.345
7.336
7.333
7.331
7.329
7.326
7.312
7.305
7.295
7.270
7.265
7.261
7.256
7.250
7.249
7.246
7.234
7.230
7.206
7.198
7.196
7.193
7.191
7.183
7.181
7.178
7.176
7.160
7.156
7.135
7.133
7.129
7.124
6.700
6.698
6.696
0.000
N
H
10.5 10.0 9.5 9.0 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 0.0 -0.5 ppm
4.95
30.47
4.95