

Highly efficient one-pot four-component Kabachnik-Fields synthesis of novel α -amino phosphonates under solvent-free and catalyst-free conditions

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Experimental Part

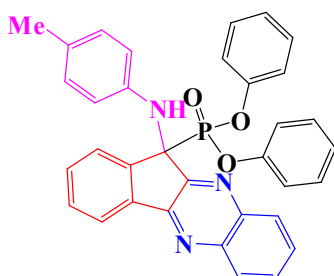
Reagents and Materials:

The chemicals used in this work were obtained from Fluka and Merck and were used without purification. Melting points were measured on an Electrothermal 9200 apparatus. IR spectra were recorded as KBr pellets on a Perkin-Elmer 781 spectrophotometer and an Impact 400 Nicolet FT-IR spectrophotometer. ^1H NMR and ^{13}C NMR spectra were recorded in $\text{DMSO}-d_6$ solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as internal reference. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

General procedure for synthesis of α -aminophosphonates

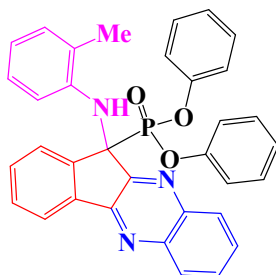
A mixture of ninhydrin **1** (1 mmol) and 1,2-phenylenediamines **2** (1 mmol) was stirred by a magnet in a test tube under solvent free conditions at room temperature for 10 min, followed by the addition of aniline derivatives (1 mmol) temperature was slowly increased until 70 °C. The reaction mixture was stirred for appropriate time, after nearly complete conversion into an intermediate presumed to be the corresponding intermediate **5** (as indicated by TLC), alkyl /aryl phosphite (1 mmol) was then added to the reaction mixture which was heated at 70 °C. After completion of the reaction (as assessed by TLC), the reaction mixture was cooled to room temperature, and EtOH (5 mL) was added to the crude products and stirred for a while. The reaction mixture was filtered and the precipitate washed with EtOH to afford the pure products. The spectroscopic and analytical data for synthesized compounds are presented below.

Diphenyl 11-(*p*-tolylamino)-11*H*-indeno[1,2-*b*]quinoxalin-11-ylphosphonate (7a):



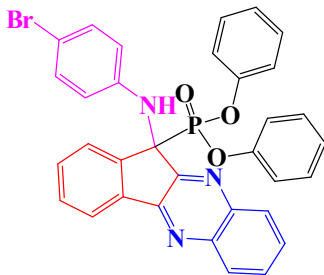
Yellow powder (Yield: 90%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3384, 1589, 1487, 1268, 1189, 930, 768. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.85 (3H, s, CH₃) 6.94 (1H, d, NH, $^3j_{\text{HP}}=11.6$ Hz), 6.08-8.28 (22 H, m, ArH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 20.2, 67.9 (1C, d, C-P, $^1j_{\text{CP}}=148$ Hz), 116.3, 120.6, 120.9, 123.3, 126.1, 127.8, 129.5, 129.8, 130.1, 130.3, 130.5, 131.3, 132.5, 137.6, 141.2, 142.4, 142.8, 143.0, 143.4, 150.2, 150.5, 153.8. Anal. Calcd for C₃₅H₂₈N₃O₃P: C, 73.80; H, 4.95; N, 7.38%;. Found C, 73.92; H, 4.89; N, 7.47%; MS: m/z 569.

Diphenyl 11-(*o*-tolylamino)-11*H*-indeno[1,2-*b*]quinoxalin-11-ylphosphonate (7b):



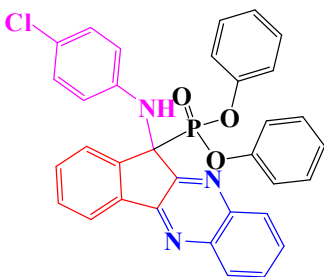
Yellow powder (Yield: 89%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3389, 1588, 1485, 1277, 1196, 927, 764. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 2.25 (3H, s, CH₃) 6.99 (1H, d, NH, $^3j_{\text{HP}}=8$ Hz), 6.39-8.32 (22 H, m, ArH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 17.8, 67.1 (1C, d, C-P, $^1j_{\text{CP}}=142$ Hz), 113.8, 119.9, 120.3, 120.4, 120.7, 120.8, 123.4, 125.9, 126.1, 126.2, 126.7, 127.4, 129.5, 129.9, 130.3, 130.4, 130.6, 130.9, 131.5, 131.9, 132.7, 142.5, 150.3, 150.6. Anal. Calcd for C₃₄H₂₆N₃O₃P: C, 73.50; H, 4.72; N, 7.56%;. Found C, 73.37; H, 4.77; N, 7.62%; MS: m/z 555.

Diphenyl 11-(4-bromophenylamino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7c):



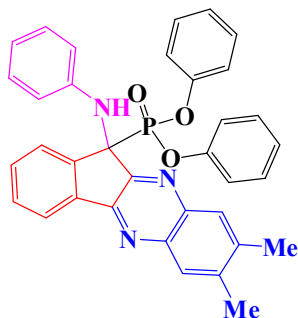
Cream powder (Yield: 93%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3335, 1589, 1487, 1265, 1205, 955, 761. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 6.20 (1H, d, NH, $^3J_{\text{HP}}=12$ Hz), 6.13-8.30 (22H, m, ArH). Anal. Calcd for $\text{C}_{33}\text{H}_{23}\text{BrN}_3\text{O}_3\text{P}$: C, 63.88; H, 3.74; N, 6.77%;. Found C, 63.75; H, 3.67; N, 6.85%; MS: m/z 619.

Diphenyl 11-(4-chlorophenylamino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7d):



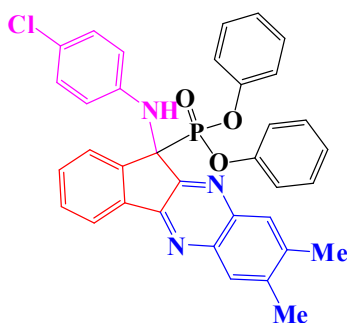
Yellow powder (Yield: 84%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3293, 1591, 1490, 1251, 1205, 955, 759. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 7.50 (1H, d, NH, $^3J_{\text{HP}}=12$ Hz), 6.16-8.30 (22H, m, ArH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 67.8 (1C, d, C-P, $^1j_{\text{CP}}=147$ Hz), 117.0, 120.5, 120.9, 123.4, 126.1, 127.4, 129.5, 129.8, 130.4, 130.6, 131.4, 132.6, 137.7, 141.2, 142.5, 142.9, 144.3, 144.5, 150.2, 150.5, 153.6, 159.9. Anal. Calcd for $\text{C}_{33}\text{H}_{23}\text{ClN}_3\text{O}_3\text{P}$: C, 68.81; H, 4.02; N, 7.30%;. Found C, 68.94; H, 4.09; N, 7.22%; MS: m/z 575.

Diphenyl 7,8-dimethyl-11-(phenylamino)-11H-indeno[1,2-*b*]quinoxalin-11-ylphosphonate (7e):



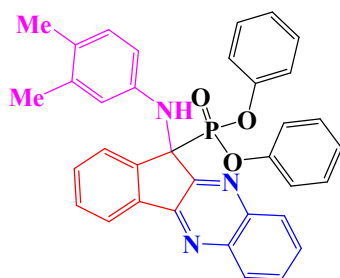
Yellow powder (Yield: 86%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3428, 1591, 1489, 1271, 1205, 945, 767. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 7.07 (1H, d, NH, $^3J_{\text{HP}}=7$ Hz), 6.14-8.24 (20H, m, ArH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 20.1, 20.3, 67.8 (1C, d, C-P, $^1j_{\text{CP}}=148$ Hz), 116.6, 120.6, 121.0, 122.9, 125.8, 126.0, 127.4, 127.7, 128.9, 129.4, 130.1, 130.3, 131.0, 132.0, 138.0, 140.1, 141.2, 141.6, 142.9, 143.1, 150.4, 152.9, 157.3. Anal. Calcd for $\text{C}_{35}\text{H}_{28}\text{N}_3\text{O}_3\text{P}$: C, 73.80; H, 4.95; N, 7.38%;. Found C, 73.71; H, 4.87; N, 7.31%; MS: m/z 569.

Diphenyl 11-(4-chlorophenylamino)-7,8-dimethyl-11H-indeno[1,2-*b*]quinoxalin-11-ylphosphonate (7f):



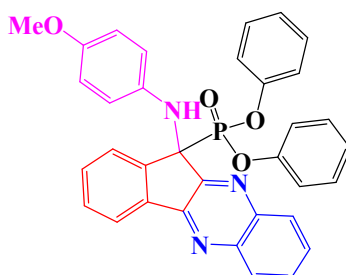
Yellow powder (Yield: 82%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3423, 1592, 1490, 1270, 1207, 949, 763. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 2.44 (3H, s, CH_3), 2.48 (3H, s, CH_3), 7.42 (1H, d, NH, $^3J_{\text{HP}}=11$ Hz), 6.14-8.24 (20H, m, ArH). Anal. Calcd for $\text{C}_{35}\text{H}_{27}\text{ClN}_3\text{O}_3\text{P}$: C, 69.59; H, 4.51; N, 6.96%;. Found C, 69.47; H, 4.56; N, 7.01%; MS: m/z 603.

Diphenyl (11-((3,4-dimethylphenyl)amino)-11H-indeno[1,2-b]quinoxalin-11-yl)phosphonate (7g):



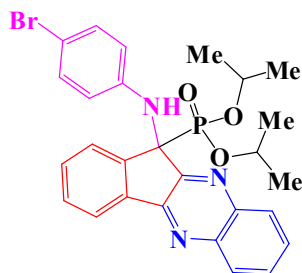
Yellow powder (Yield: 89%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3427, 1587, 1493, 1273, 1210, 953, 758. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.83 (3H, s, CH₃), 1.85 (3H, s, CH₃), 6.44 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 5.64-7.96 (21H, m, ArH). Anal. Calcd for C₃₅H₂₈N₃O₃P: C, 73.80; H, 4.96; N, 7.38%. Found C, 73.71; H, 4.88; N, 7.44%; MS: m/z 569.

Diphenyl (11-((4-methoxyphenyl)amino)-11H-indeno[1,2-b]quinoxalin-11-yl)phosphonate (7h):



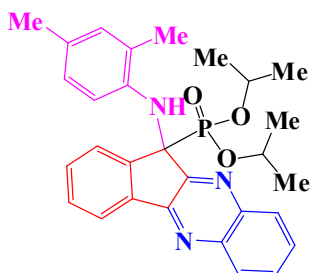
Yellow powder (Yield: 85%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3429, 1588, 1485, 1270, 1210, 953, 766. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 3.43 (3H, s, OCH₃), 6.40 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 6.20-7.94 (21H, m, ArH). Anal. Calcd for C₃₇H₂₆N₃O₄P: C, 71.45; H, 4.59; N, 7.35%. Found C, 71.37; H, 4.53; N, 7.43%; MS: m/z 571.

Diisopropyl 11-(4-bromophenylamino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7i):



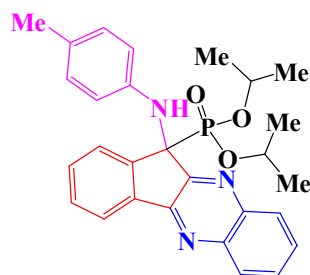
Yellow powder (Yield: 83%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3294, 1592, 1490, 1240, 1008, 757. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.04 (3H, d, CH₃), 1.05 (3H, d, CH₃), 1.14 (3H, d, CH₃), 1.16 (3H, d, CH₃), 1.88 (3H, s, CH₃), 2.29 (3H, s, CH₃), 4.38 (1H, m, OCH), 4.53 (1H, m, OCH), 6.75 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 6.01-8.23 (12H, m, ArH). Anal. Calcd for C₂₇H₂₇BrN₃O₃P: C, 58.71; H, 4.93; N, 7.61%. Found C, 58.84; H, 4.99; N, 7.54%; MS: m/z 551.

Diisopropyl 11-(2,4-dimethylphenylamino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7j):



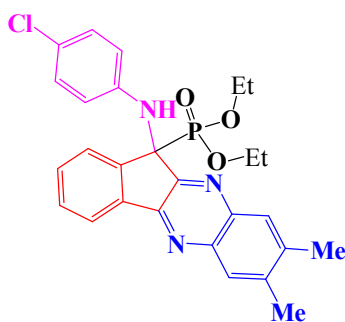
Yellow powder (Yield: 85%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3421, 1511, 1251, 1002, 759. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.03 (3H, d, CH₃), 1.10 (3H, d, CH₃), 1.13 (3H, d, CH₃), 1.19 (3H, d, CH₃), 1.88 (3H, s, CH₃), 2.29 (3H, s, CH₃), 4.38 (1H, m, OCH), 4.55 (1H, m, OCH), 5.18 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 4.98-8.24 (11H, m, ArH). Anal. Calcd for C₂₉H₃₂N₃O₃P: C, 69.45; H, 6.43; N, 8.38%. Found C, 69.32; H, 6.50; N, 8.46%; MS: m/z 501.

Diisopropyl 11-(p-tolylamino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7k):



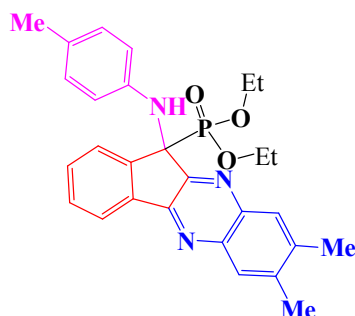
Yellow powder (Yield: 92%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3308, 1614, 1516, 1238, 1101, 1005, 758. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.17 (9H, m, 3CH₃), 1.20 (3H, s, CH₃), 3.33 (3H, s, CH₃), 3.33 (1H, m, OCH), 4.56 (1H, m, OCH), 5.96 (1H, d, NH, $^3J_{\text{HP}}$ = 10.8 Hz), 5.10-8.24 (12H, m, ArH). ^{13}C NMR (DMSO- d_6 , 100 MHz): δ_{ppm} : 16.5, 55.3, 64.3, 64.7, 67.1 (1C, d, C-P, $^1j_{\text{CP}}$ =143 Hz), 114.3, 117.6, 122.9, 127.1, 129.4, 129.8, 130.2, 130.5, 130.9, 132.1, 137.4, 139.2, 141.2, 142.2, 144.7, 152.7, 153.8, 159.7. Anal. Calcd for C₂₈H₃₀N₃O₃P: C, 68.98; H, 6.20; N, 8.62%;. Found C, 68.86; H, 6.25; N, 8.71%; MS: m/z 487.

Diethyl 11-(4-chlorophenylamino)-7,8-dimethyl-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7l):



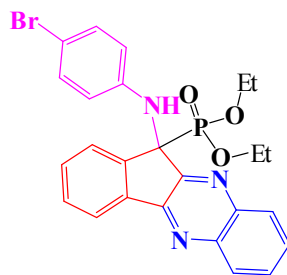
Orange powder (Yield: 80%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3427, 1602, 1494, 1233, 1025, 752. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 0.99 (3H, t, CH₃), 1.08 (3H, t, CH₃), 2.43 (3H, s, CH₃), 2.47 (3H, s, CH₃), 3.83 (2H, q, OCH₂), 3.98 (2H, q, OCH₂), 6.80 (1H, d, NH, $^3J_{\text{HP}}$ = 12 Hz), 6.03-7.95 (10H, m, ArH). Anal. Calcd for C₂₇H₂₇ClN₃O₃P: C, 63.84; H, 5.36; N, 8.27%;. Found C, 63.70; H, 5.41; N, 8.18%; MS: m/z 508.

Diethyl 7,8-dimethyl-11-(*p*-tolylamino)-11*H*-indeno[1,2-*b*]quinoxalin-11-ylphosphonate (7m):



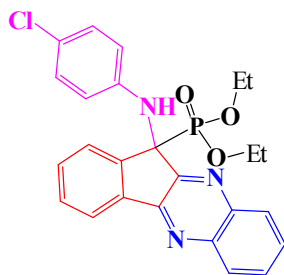
Yellow powder (Yield: 86%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3431, 1618, 1520, 1236, 1025. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.00 (3H, t, CH₃), 1.09 (3H, t, CH₃), 2.44 (3H, s, CH₃), 2.48 (3H, s, CH₃), 3.83 (2H, q, OCH₂), 3.98 (2H, q, OCH₂), 6.21 (1H, d, NH, $^3J_{\text{HP}} = 11.2$ Hz), 5.94-8.16 (10H, m, ArH). Anal. Calcd for C₂₈H₃₀N₃O₃P: C, 68.98; H, 6.20; N, 8.62%. Found C, 69.11; H, 6.13; N, 8.71%; MS: m/z 487.

Diethyl 11-(4-bromophenylamino)-11*H*-indeno[1,2-*b*]quinoxalin-11-ylphosphonate (7n):



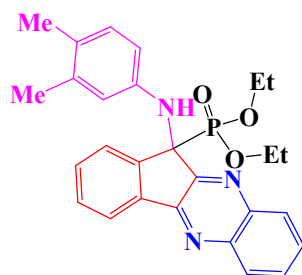
Orange powder (Yield: 83%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3279, 1593, 1490, 1238, 1034, 756. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 0.98 (3H, t, CH₃), 1.09 (3H, t, CH₃), 3.77 (2H, q, OCH₂), 4.00 (2H, q, OCH₂), 6.94 (1H, d, NH, $^3J_{\text{HP}} = 10.4$ Hz), 6.00-8.24 (12H, m, ArH). Anal. Calcd for C₂₅H₂₃BrN₃O₃P: C, 57.27; H, 4.42; N, 8.01%. Found C, 57.20; H, 4.46; N, 8.09%; MS: m/z 524.

Diethyl 11-(4-chlorophenylamino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7o):



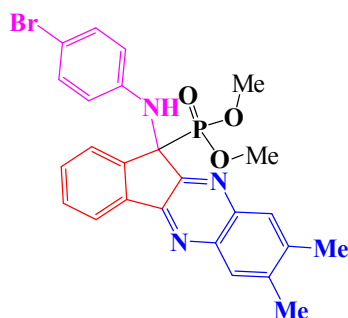
Cream powder (Yield: 87%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3410, 1591, 1493, 1256, 1009, 757. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.09 (3H, t, CH₃), 1.11 (3H, t, CH₃), 4.01 (2H, q, OCH₂), 4.02 (2H, q, OCH₂), 6.93 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 6.04-8.25 (12H, m, ArH). Anal. Calcd for C₂₅H₂₃ClN₃O₃P: C, 62.57; H, 4.83; N, 8.76%. Found C, 62.41; H, 4.77; N, 8.85%; MS: m/z 479.

Diethyl 11-((3,4-dimethylphenyl)amino)-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7p):



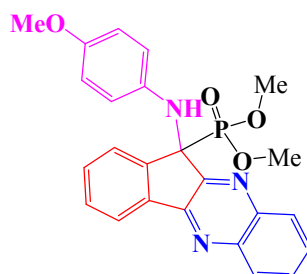
Yellow powder (Yield: 89%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3415, 1585, 1487, 1250, 1020, 757. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 1.04 (3H, t, CH₃), 1.10 (3H, t, CH₃), 1.80 (3H, s, CH₃), 1.82 (3H, s, CH₃), 3.60 (2H, q, OCH₂), 4.03 (2H, q, OCH₂), 6.38 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 5.49-8.25 (11H, m, ArH). Anal. Calcd for C₂₇H₂₈N₃O₃P: C, 68.49; H, 5.96; N, 8.87%. Found C, 68.41; H, 5.88; N, 8.79%; MS: m/z 573.

Dimethyl 11-(4-bromophenylamino)-7,8-dimethyl-11H-indeno[1,2-b]quinoxalin-11-ylphosphonate (7q):

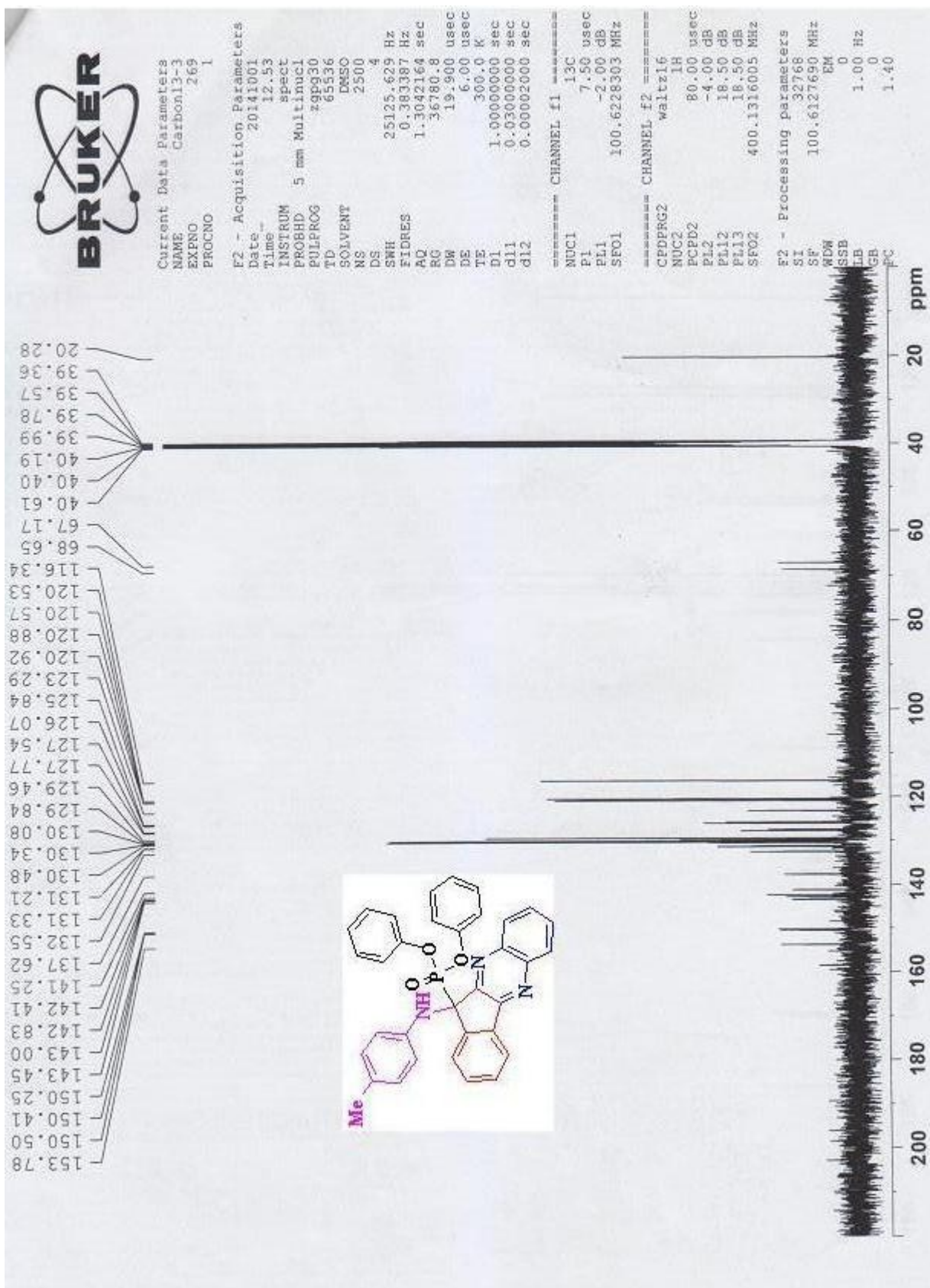


Pale yellow powder (Yield: 92%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3283, 1549, 1492, 1242, 1038, 767. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 2.47 (3H, s, CH₃), 2.48 (3H, s, CH₃), 3.51 (3H, d, OCH₃), 3.64 (3H, d, OCH₃), 6.94 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 5.97-8.20 (10H, m, ArH). Anal. Calcd for C₂₅H₂₃BrN₃O₃P: C, 57.27; H, 4.42; N, 8.01%. Found C, 57.38; H, 4.47; N, 8.10%; MS: m/z 524.

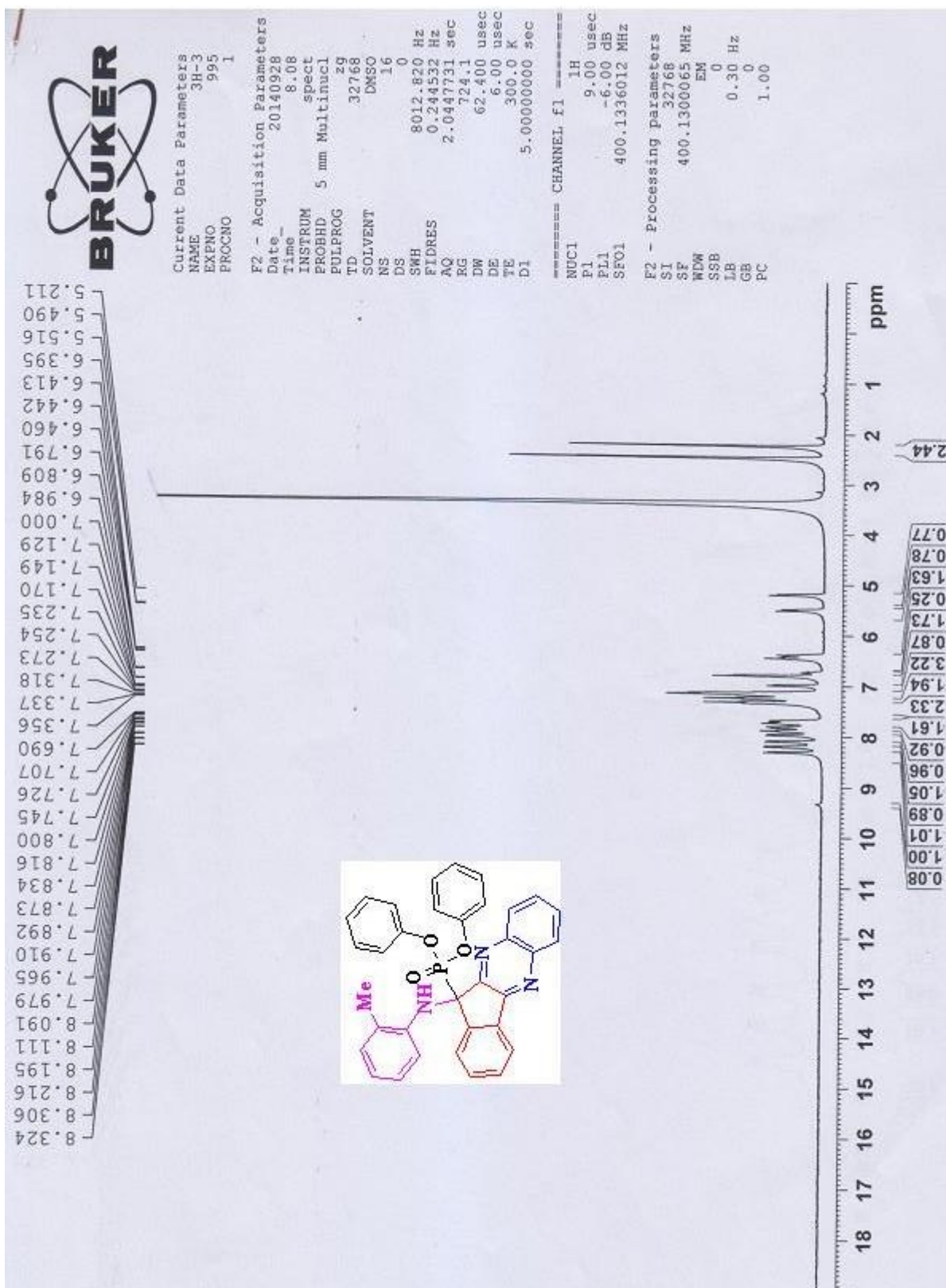
Dimethyl (11-((4-methoxyphenyl)amino)-11H-indeno[1,2-b]quinoxalin-11-yl)phosphonate (7r):



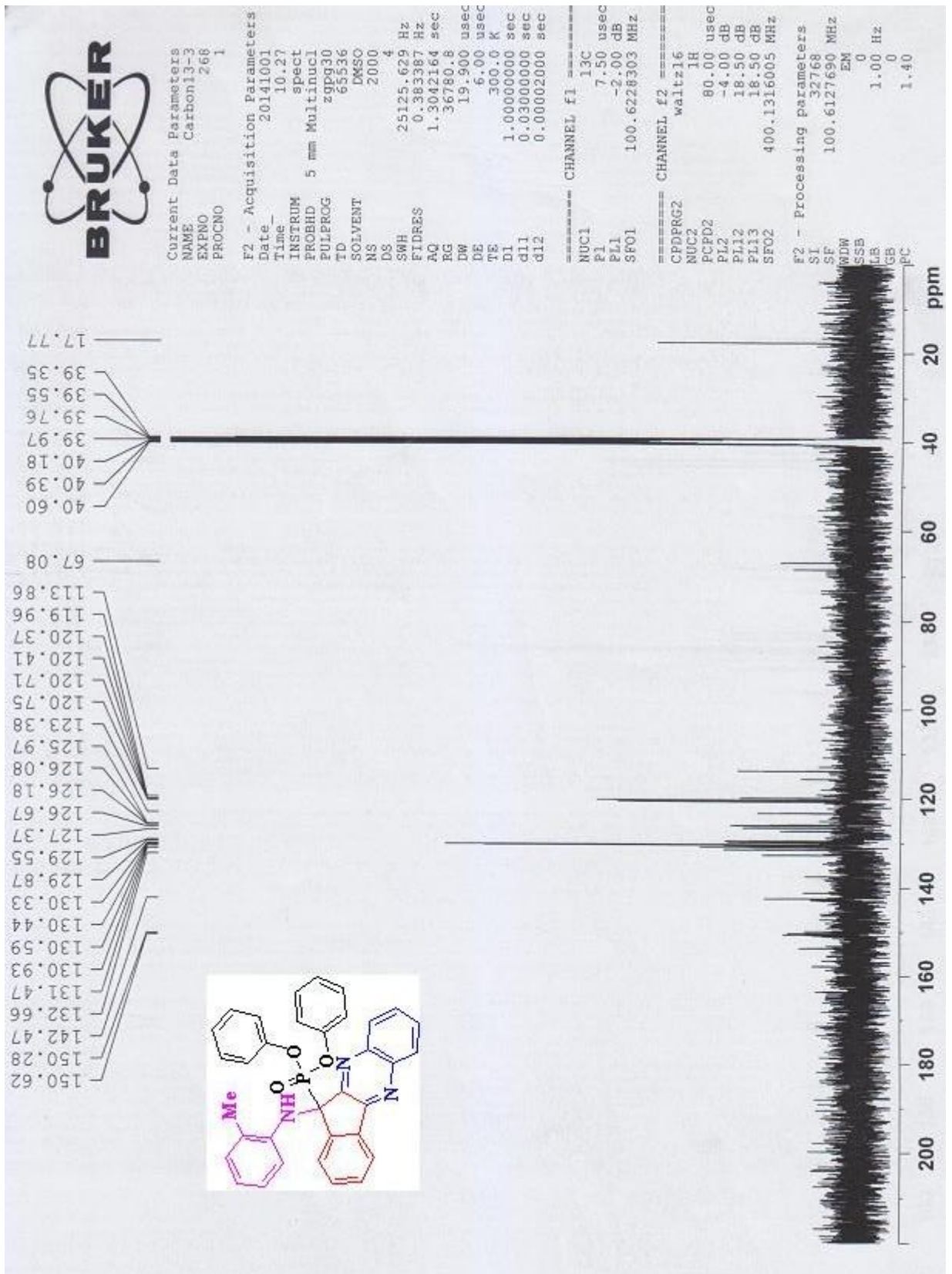
Yellow powder (Yield: 92%). mp>300°C. IR (KBr) (ν_{\max} / cm^{-1}): 3278, 1540, 1485, 1252, 1020, 763. ^1H NMR (DMSO- d_6 , 400 MHz): δ_{ppm} : 3.41 (3H, s, OCH₃), 3.53 (3H, d, OCH₃), 3.68 (3H, d, OCH₃), 6.23 (1H, d, NH, $^3J_{\text{HP}} = 12$ Hz), 6.04-8.21 (12H, m, ArH). Anal. Calcd for C₂₄H₂₂N₃O₄P: C, 64.43; H, 4.96; N, 9.39%. Found C, 64.35; H, 4.84; N, 9.30%; MS: m/z 447.



¹³C NMR of 7a



¹H NMR of 7b



¹³C NMR of 7b



Current Data Parameters
NAME 3H-3
EXPNO 2048
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141105
Time_ 15.02
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 322.5
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

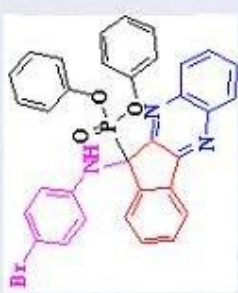
==== CHANNEL f1 =====
NUC1 1H
PI 9.00 usec
PL1 -6.00 dB
SF01 400.1336012 MHz

F2 - Processing parameters
SI 32768
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

8.305
8.287
8.214
8.193
8.162
8.145
8.115
8.095
8.074
7.920
7.904
7.892
7.871
7.840
7.821
7.803
7.751
7.733
7.711
7.690
7.672
7.542
7.505
7.333
7.314
7.296
7.253
7.207
7.189
7.168
7.142
7.122
7.087
7.068
7.000
6.952
6.931
6.880
6.859
6.754
6.735
6.675
6.656



1.00
1.09
1.12
2.32
1.24
2.29
0.83
0.61
2.36
1.92
1.70
1.36
2.99
1.84
1.89
2.09
2.04
0.46



¹H NMR of 7c



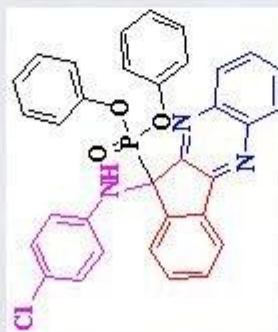
Current Data Parameters
NAME JH-J
EXPNO 965
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140921
Time_ 14.32
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 574.7
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 5.0000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SF01 400.1336012 MHz

F2 - Processing parameters
SI 32768
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

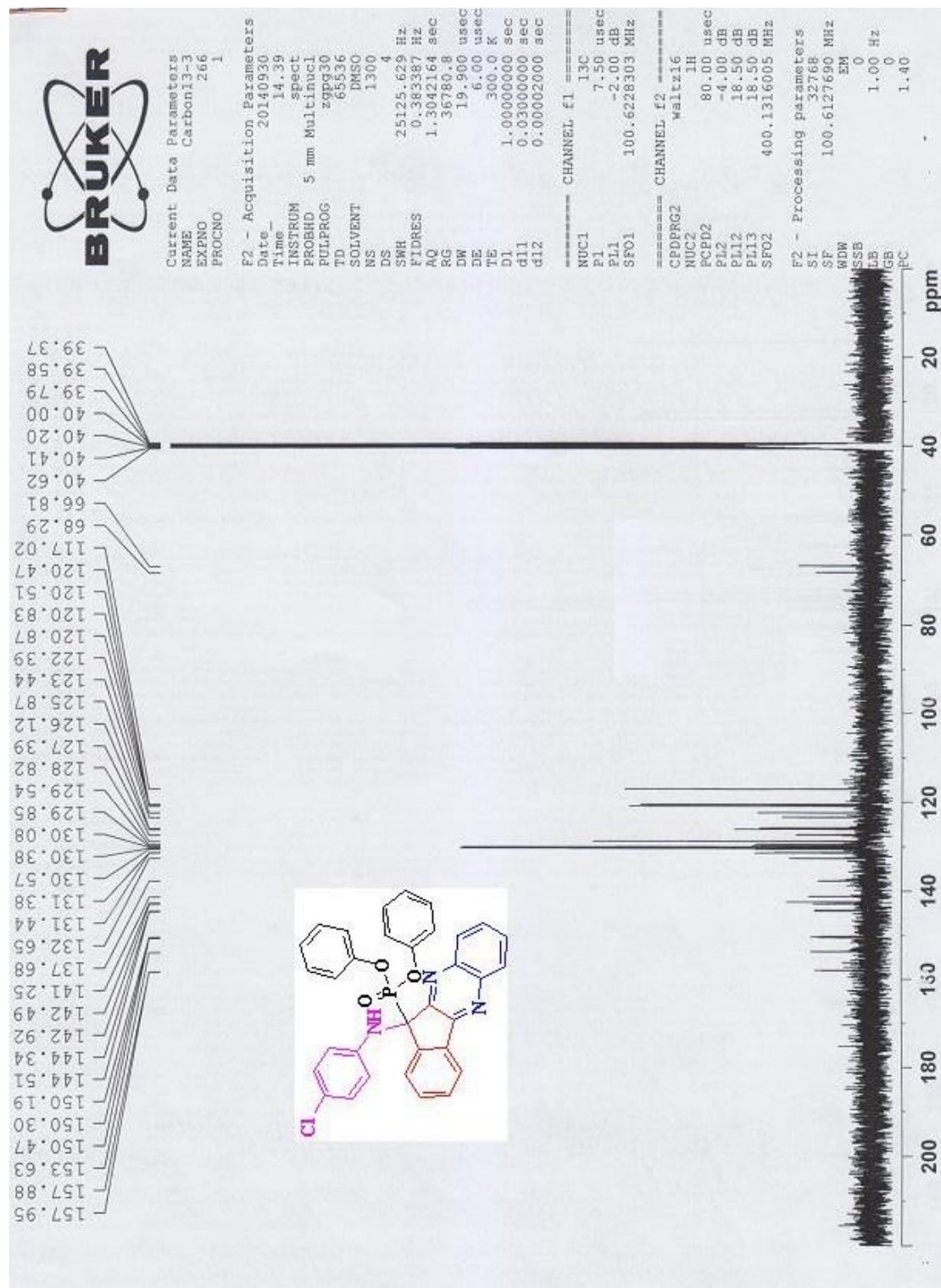
8.303
8.286
8.215
8.194
8.117
8.097
7.899
7.877
7.845
7.827
7.807
7.736
7.715
7.692
7.518
7.487
7.335
7.317
7.298
7.211
7.192
7.172
7.083
7.073
7.066
6.836
6.814
6.670
6.651
6.190
6.168
3.421
2.486
2.450



1.00
1.03
1.01
1.13
2.06
1.06
2.10
3.14
2.96
2.00
2.02
2.02

8 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm

¹H NMR of 7d



¹³C NMR of 7d



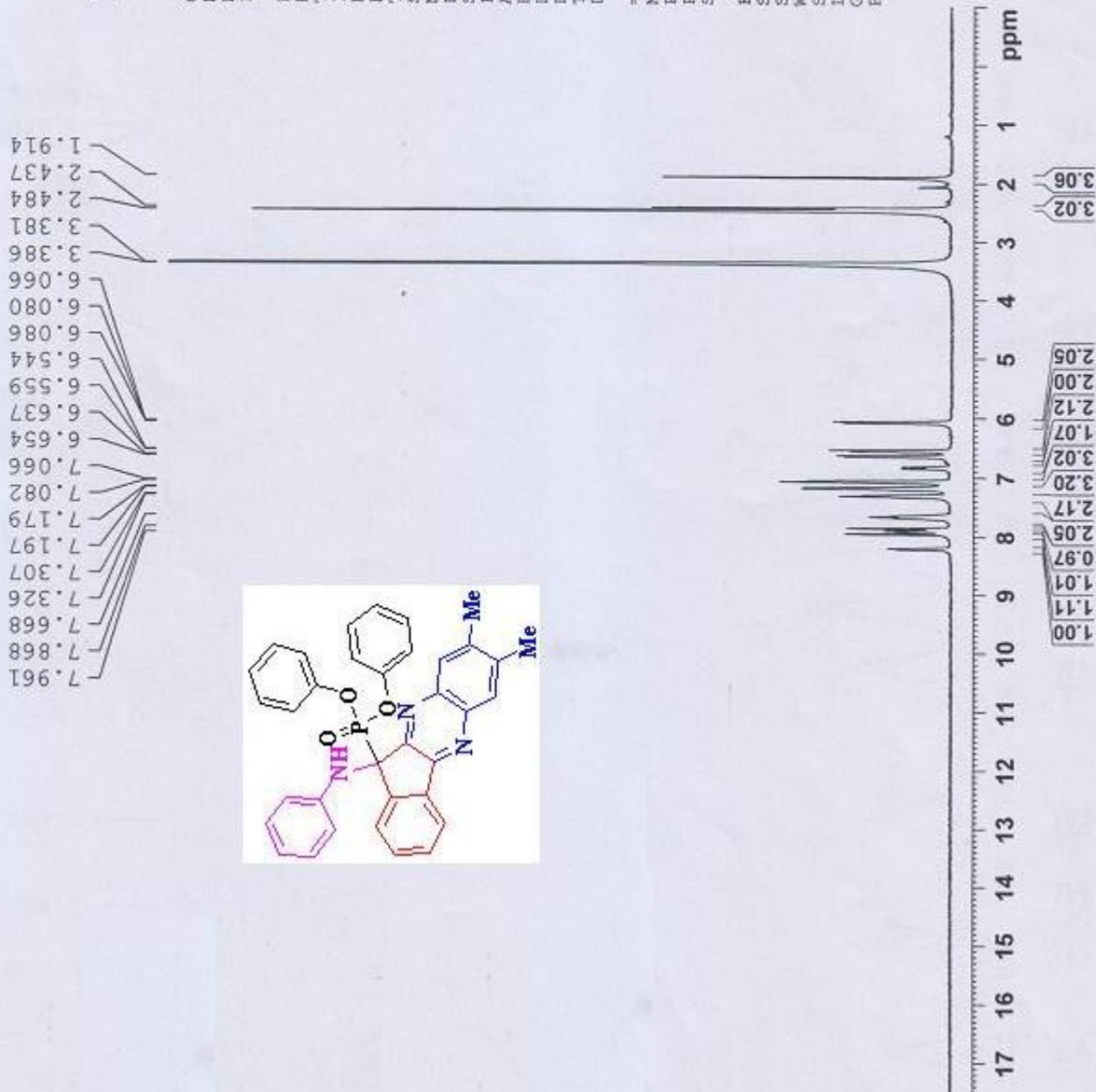
Current Data Parameters
NAME 3H-3
EXPNO 988
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140927
Time 9.35
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 728.1
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

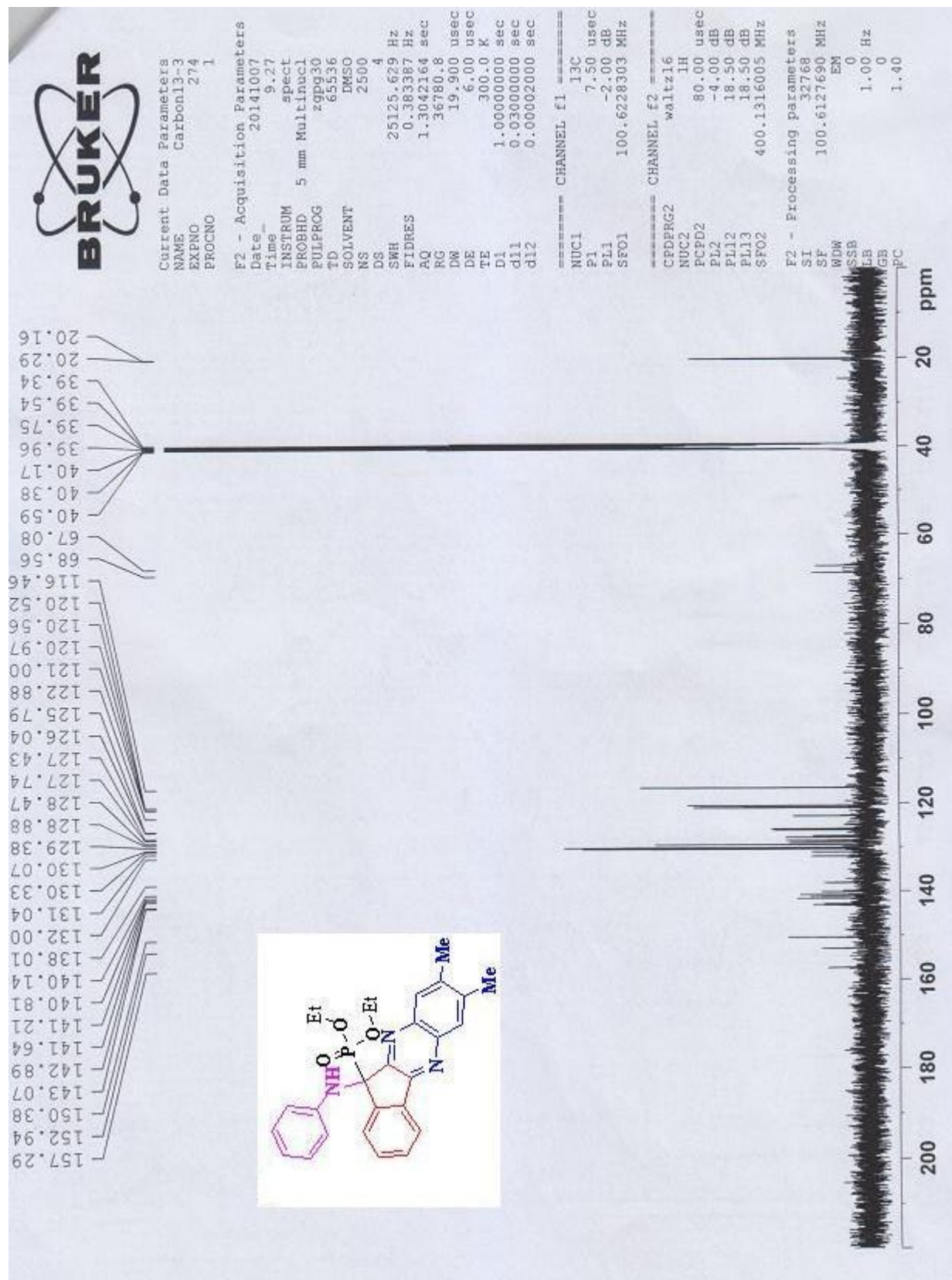
===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1336012 MHz

F2 - Processing parameters
SI 32768
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

(7e)



¹H NMR of 7e



¹³C NMR of 7e

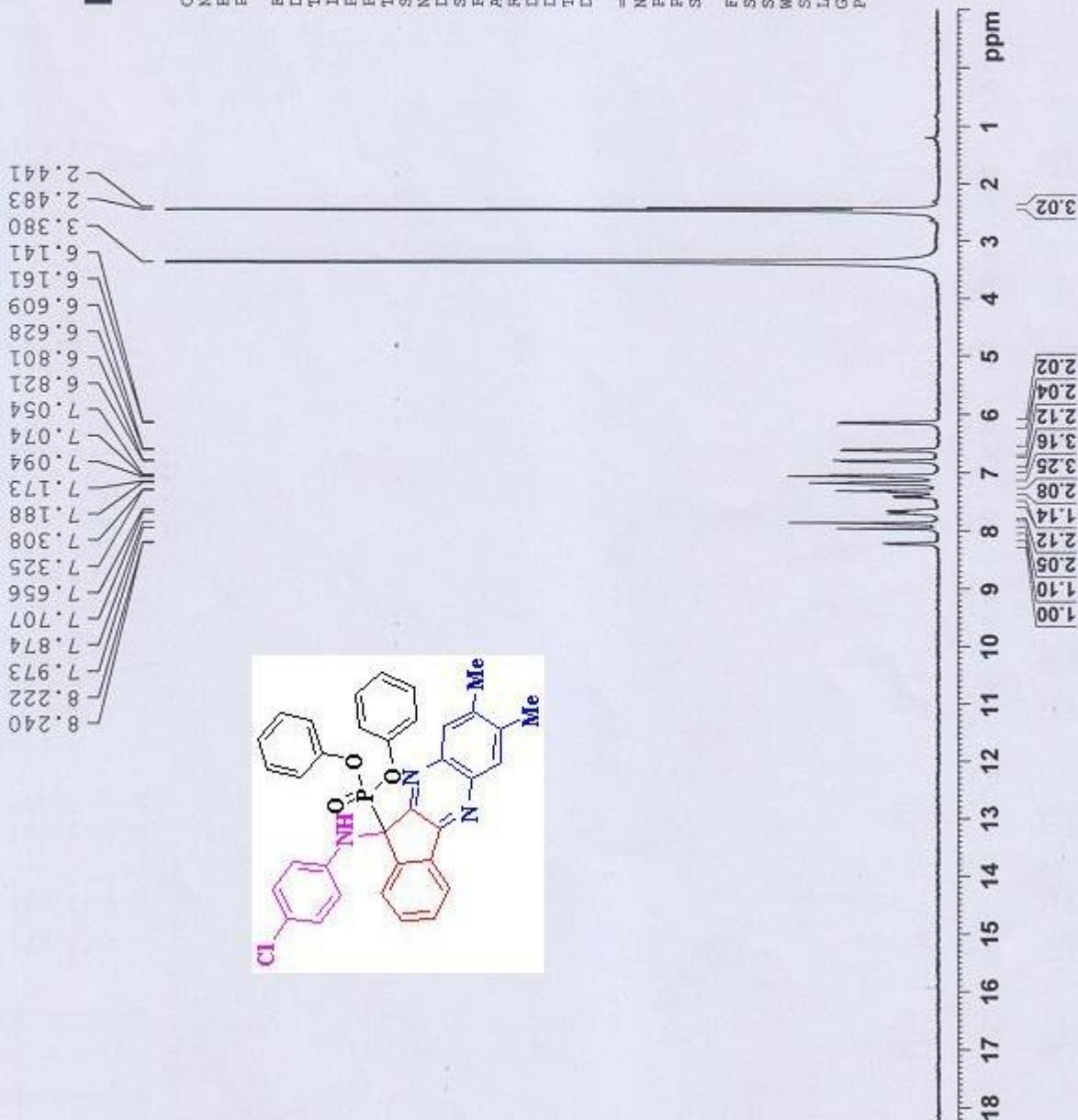


Current Data Parameters
NAME 3H-3
EXPNO 966
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140921
Time_ 14.38
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 1149.4
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 5.0000000 sec

==== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 400.1336012 MHz

F2 - Processing parameters
SI 32768
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹H NMR of 7f

ZR2
1H NMR

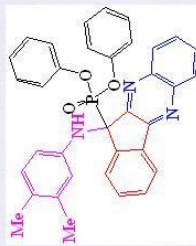
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Current Data Parameters
NAME      Dr.ghahramanzade
EXPNO     4
PROCNO    1

F2 - Acquisition Parameters
Date_     20151018
Time      11.19
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         32768
SOLVENT   DMSO
NS         32
DS         1
SWH        7812.500 Hz
FIDRES     0.238419 Hz
AQ         2.0972021 sec
RG         228.1
DW         64.000 usec
DE         6.00 usec
TE         380.0 K
D1         2.00000000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.80 usec
PL1        -2.00 dB
SFO1       300.1323986 MHz

F2 - Processing parameters
SI          65536
SF          300.1300000 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
  
```



¹H NMR of 7g

ZR1
1H NMR

Current Data Parameters
 NAME Dr.ghahramanzade
 EXPNO 3
 PROCNO 1

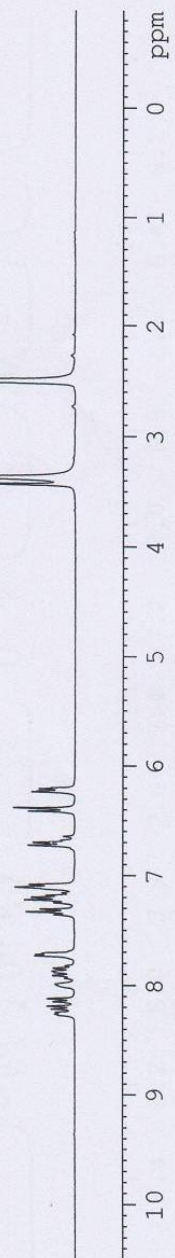
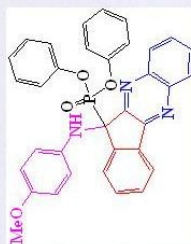
F2 - Acquisition Parameters

Date_ 20151018
 Time 11.15
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 32768
 SOLVENT DMSO
 NS 13
 DS 1
 SWH 7812.500 Hz
 FIDRES 0.238419 Hz
 AQ 2.0972021 sec
 RG 228.1
 DW 64.000 usec
 DE 6.00 usec
 TE 380.0 K
 D1 2.0000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 7.80 usec
 PL1 -2.00 dB
 SFO1 300.1323986 MHz

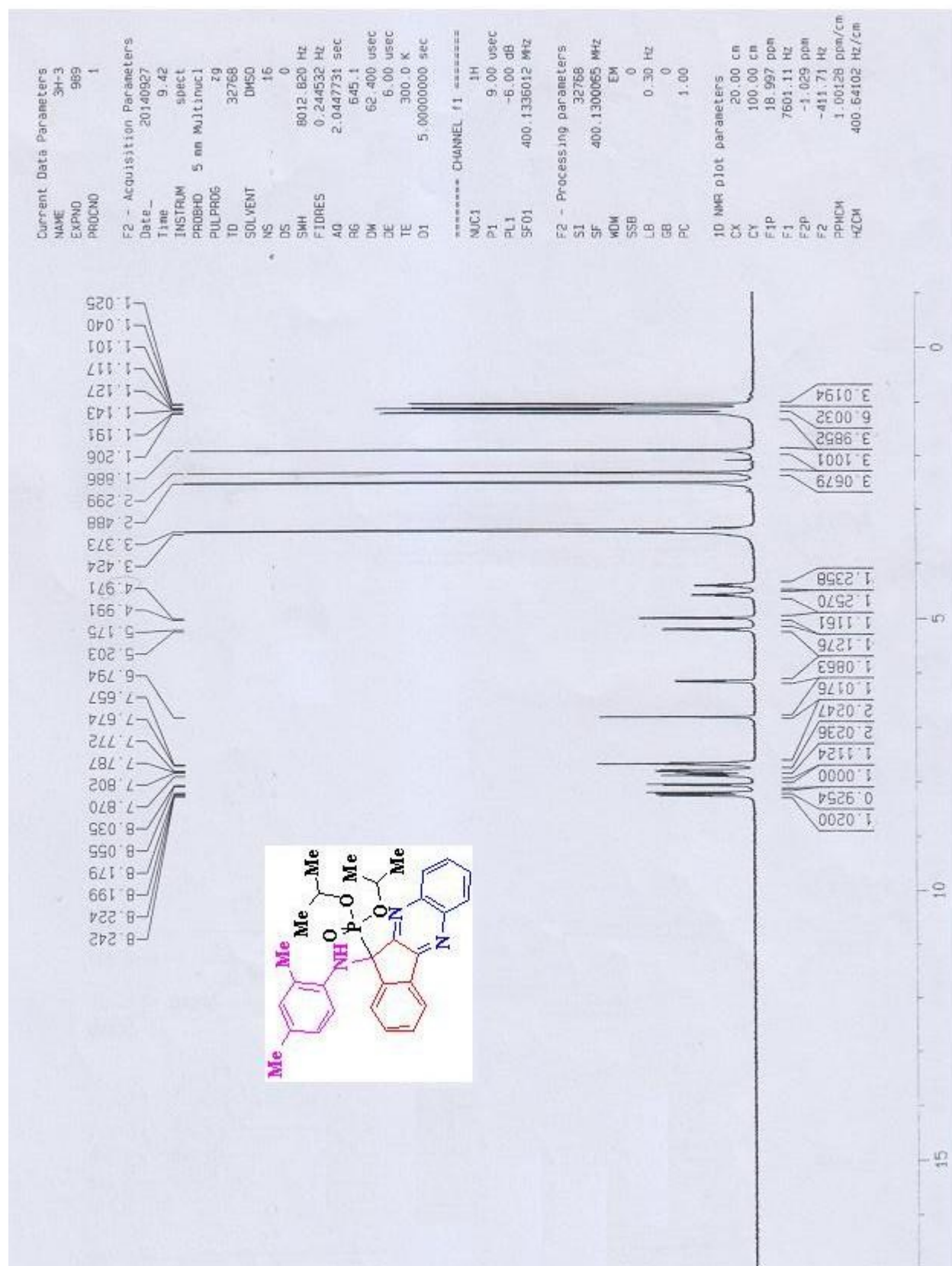
F2 - Processing parameters
 SI 65536
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

7.941
7.918
7.891
7.885
7.879
7.852
7.829
7.829
7.740
7.728
7.728
7.717
7.366
7.341
7.314
7.257
7.232
7.210
7.188
7.164
7.118
7.093
6.736
6.708
6.688
6.652
6.422
6.392
6.244
6.235
6.214
6.205
3.437
3.378
2.510
2.505

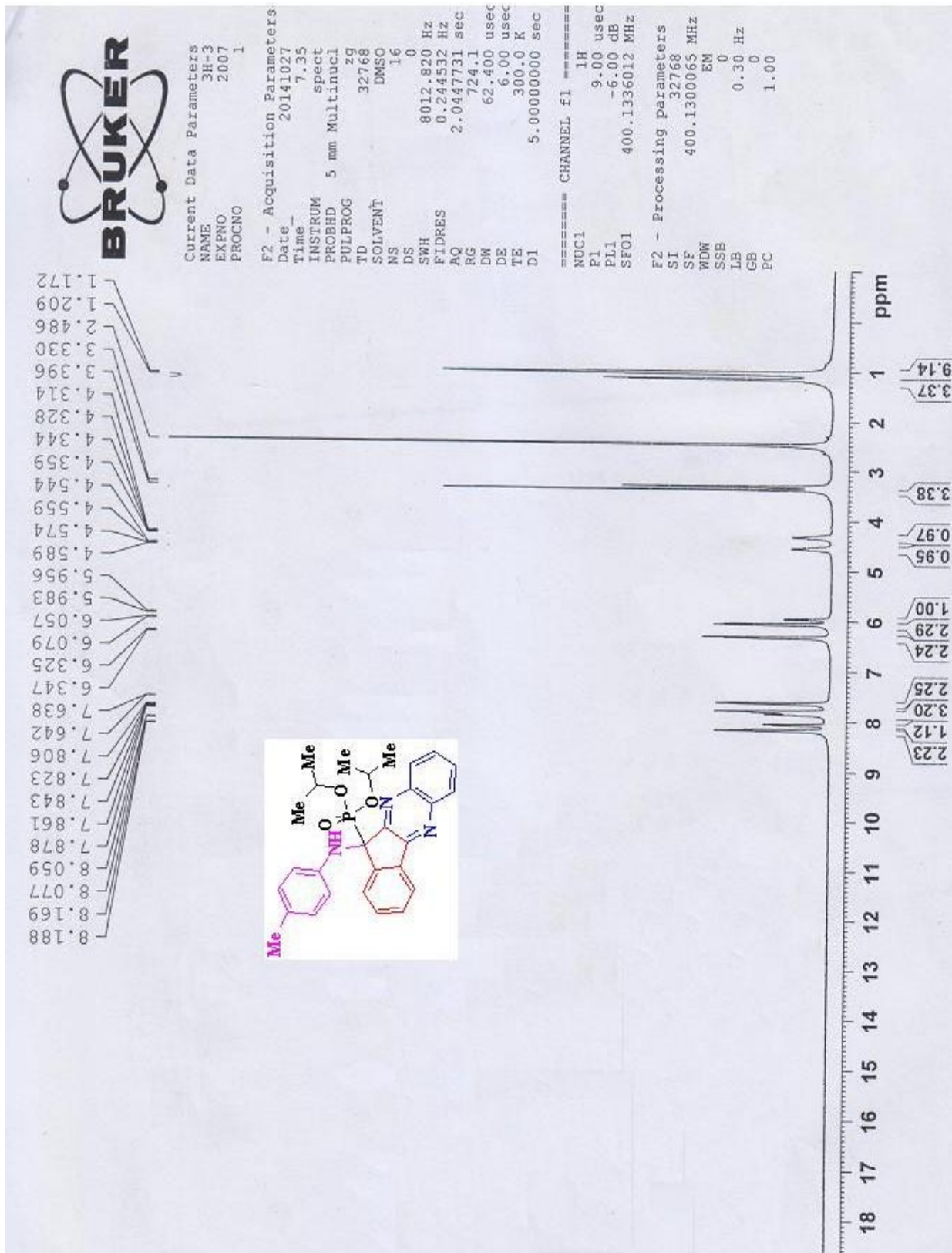


3.08
1.00
1.03
1.03
0.97
2.11
1.98
2.00
2.93
2.86
2.22
1.98
1.99

¹H NMR of 7h



¹H NMR of 7j



¹H NMR of 7k



Current Data Parameters
NAME Carbon13-3
EXPNO 313
PROCNO 1

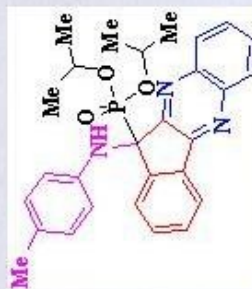
F2 - Acquisition Parameters
Date_ 20141117
Time 15.23
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 2500
DS 4
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 14596.5
DW 19.900 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

=====
CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 -2.00 dB
SFO1 100.6228303 MHz

=====
CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.50 dB
PL13 18.50 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

159.67
153.79
152.73
144.74
144.68
142.20
141.17
139.18
137.36
132.12
130.96
130.53
130.22
129.78
129.42
127.08
122.90
117.56
114.35
68.89
67.46
64.72
64.65
64.40
64.33
55.31
40.55
40.34
40.13
39.92
39.72
39.51
39.30
16.49



200 180 160 140 120 100 80 60 40 20 ppm

¹³C NMR of 7k



Current Data Parameters
NAME 3H-3
EXPNO 999
PROCNO 1

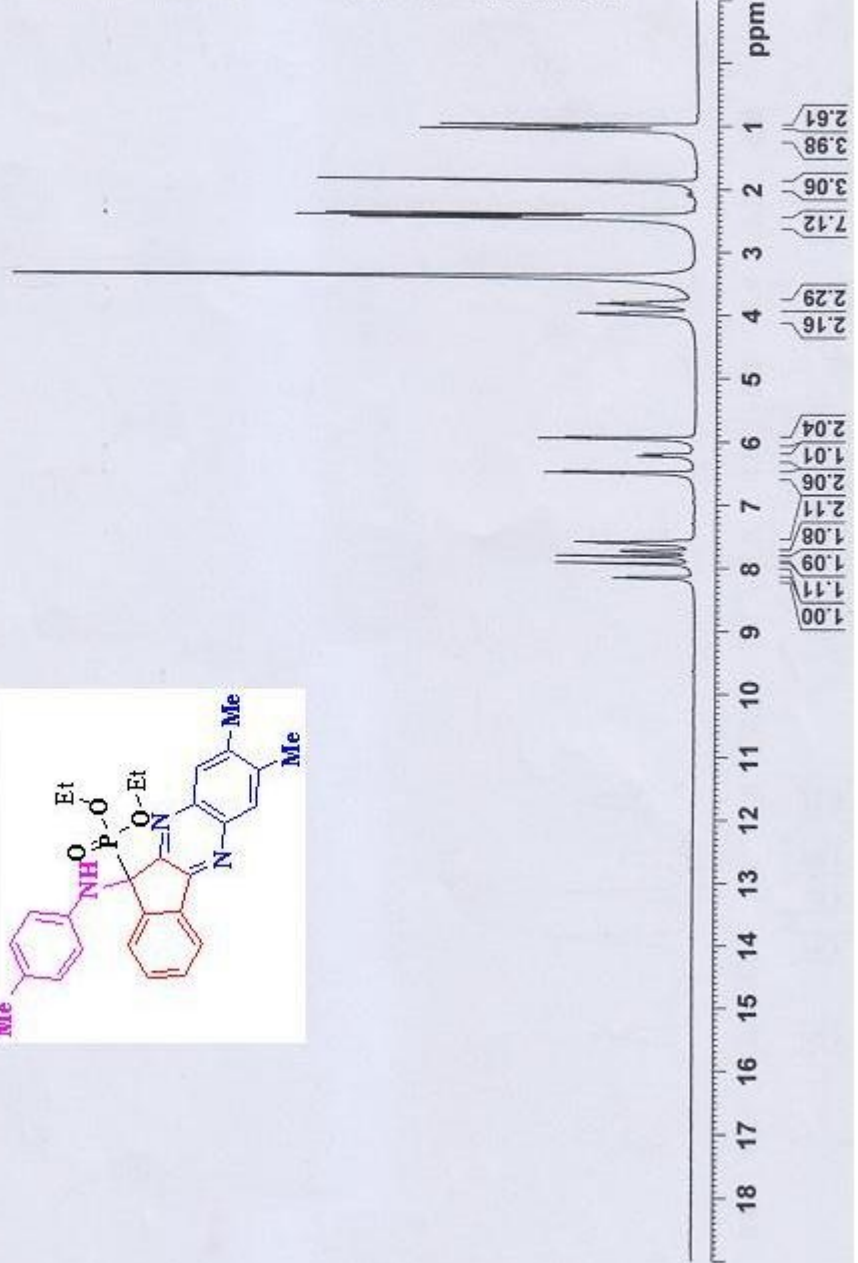
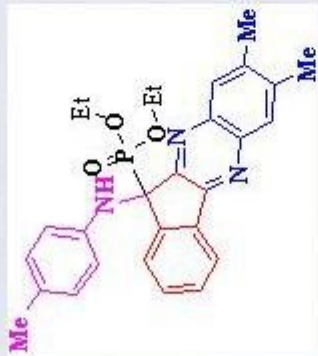
F2 - Acquisition Parameters

Date_ 20140928
Time_ 8.40
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 512
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SF01 400.1336012 MHz

F2 - Processing parameters
SI 32768
SF 400.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1.170
1.878
2.410
2.446
2.481
3.406
3.795
3.814
3.833
3.850
3.964
3.982
3.999
5.940
5.948
5.960
6.203
6.231
6.483
6.502
7.565
7.583
7.601
7.614
7.733
7.747
7.821
7.931
8.149
8.165



¹H NMR of 7m

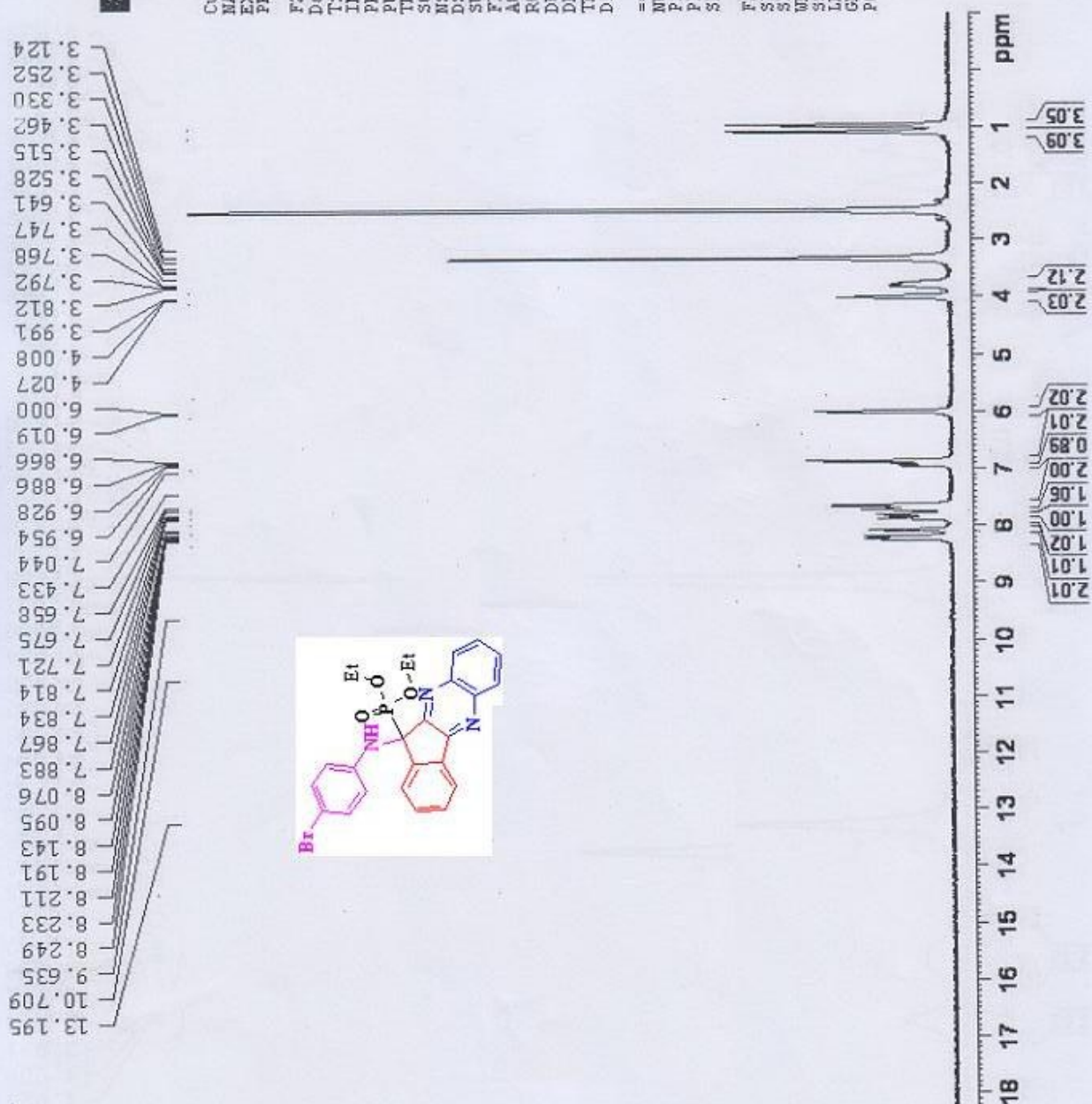


Current Data Parameters
NAME 3H-3
EXPNO 2049
PROCNO 1

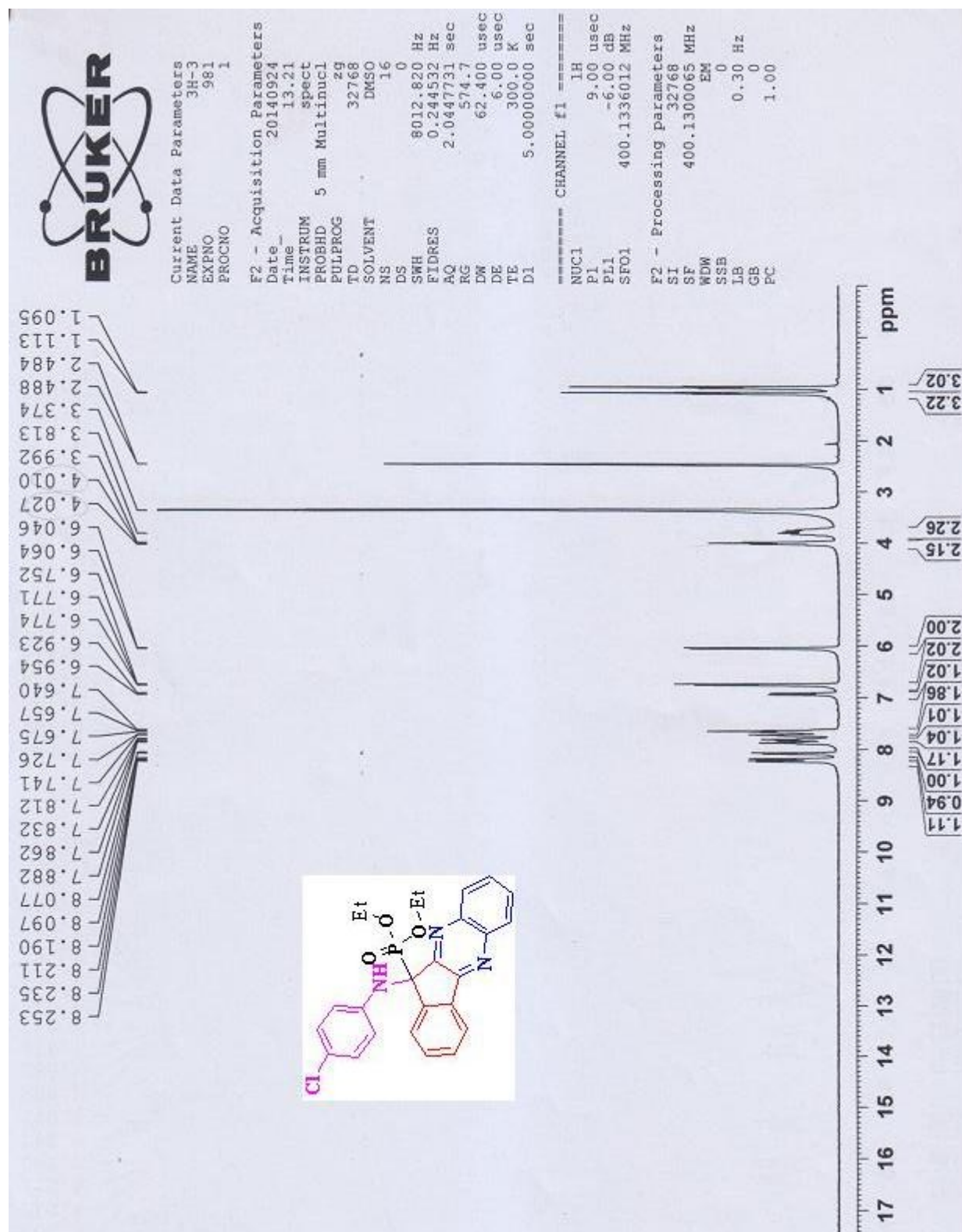
F2 - Acquisition Parameters
Date_ 20141105
Time 15:08
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 32768
SOLVENT DMSO
MS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.244532 Hz
AQ 2.0447731 sec
RG 1024
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 5.00000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SF01 400.1336012 MHz

F2 - Processing parameters
SI 32768
SF 400.1300065 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹H NMR of 7n



¹H NMR of 7o

ZR3
1H NMR

```

Current Data Parameters
NAME      Dr. ghahramanzade
EXPNO     7
PROCNO    1

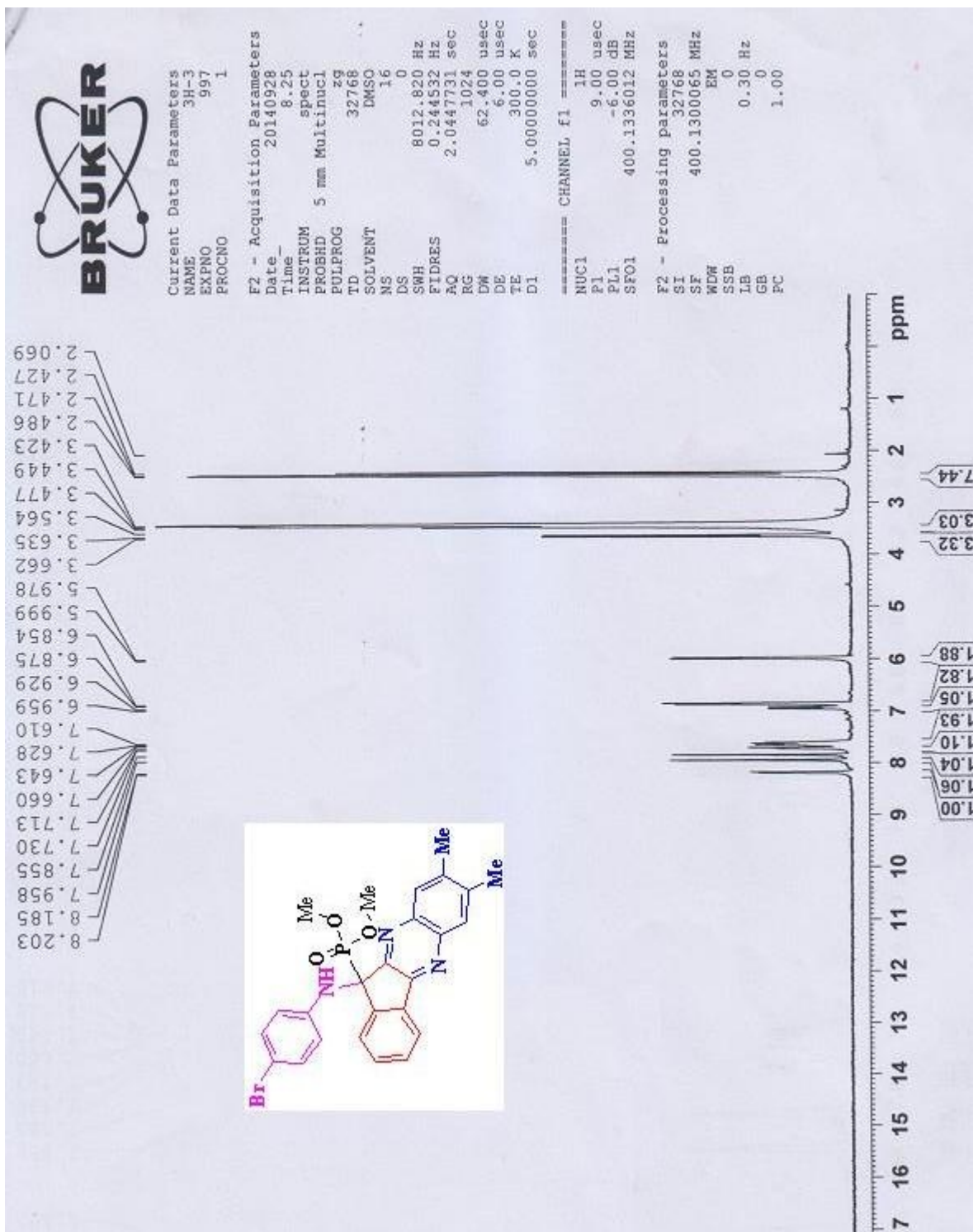
F2 - Acquisition Parameters
Date_     20151018
Time      11.34
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zg30
TD         32768
SOLVENT   DMSO
NS         32
DS         1
SWH        7812.500 Hz
FIDRES     0.238419 Hz
AQ         2.0972021 sec
RG         228.1
DE         64.000 usec
TE         380.0 K
D1         2.00000000 sec

===== CHANNEL f1 =====
NUC1       1H
P1         7.80 usec
PL1        -2.00 dB
SFO1       300.1323986 MHz

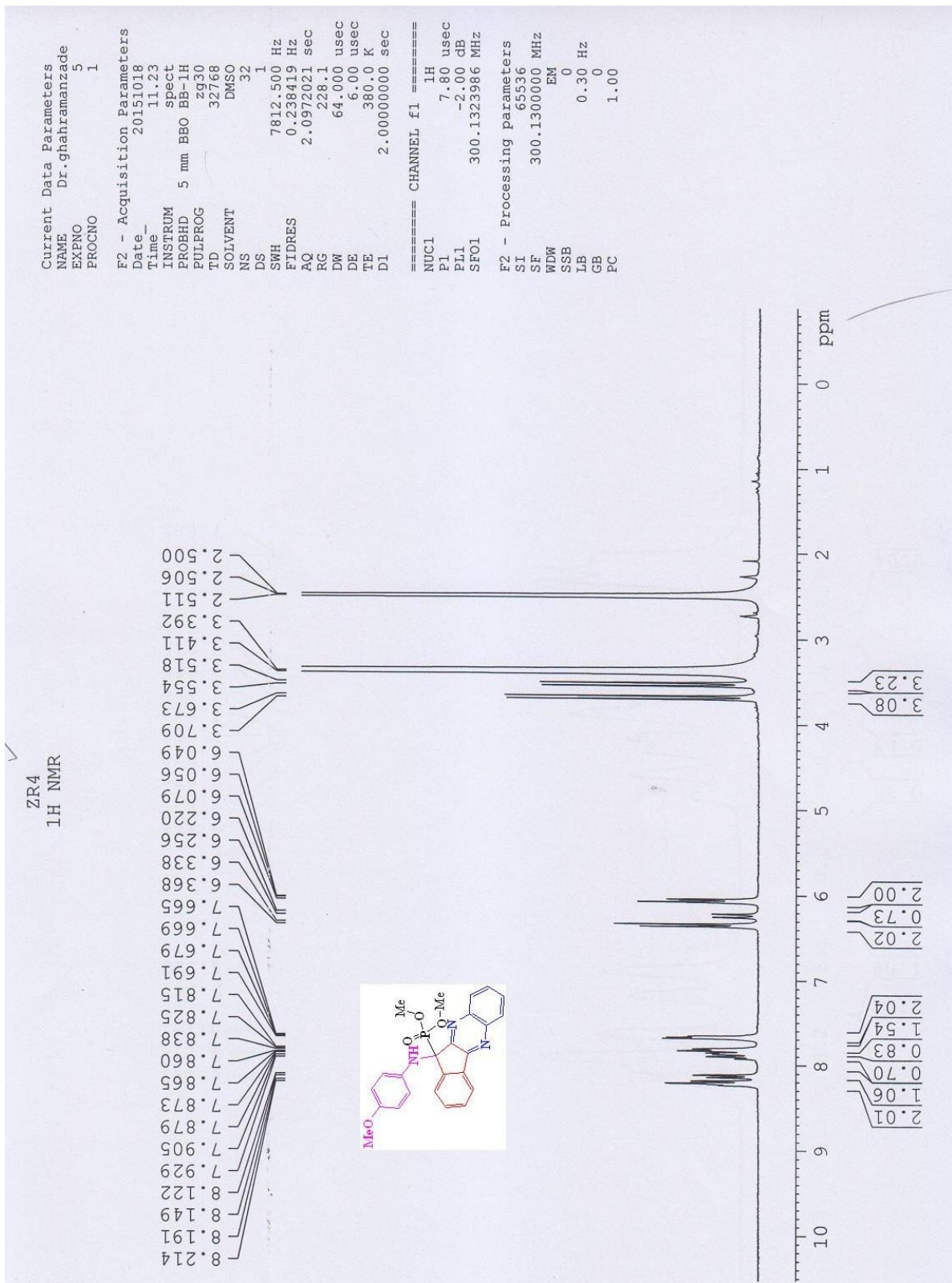
F2 - Processing parameters
SI         65536
SF         300.1300000 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



¹H NMR of 7p



^1H NMR of 7q



¹H NMR of 7r