

Supporting Information

for

## A Facile and Practical Synthesis of *N*-Acetyl Enamides

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**General Methods.** All reactions were carried out under a nitrogen atmosphere. Acetic anhydride and acetic acid were purchased from Aldrich and used directly. THF (<0.02% water content) was purchased from J. T. Baker and used directly without further purifications. Anhydrous ferrous acetate was purchased from Strem Chemicals, Inc. All the ketoximes substrates **1a-o** were prepared from corresponding ketones and hydroxylamine hydrochloride following reported procedures.<sup>1,8</sup> <sup>1</sup>H and <sup>13</sup>C NMR data were recorded on a Bruker-Biospin DRX500 or DRX400 NMR Spectrometer with DMSO-*d*<sub>6</sub> as the solvent. Melting points were determined on a Fisher-Johns melting-point apparatus.

**General procedure for the synthesis of *N*-acetyl arylenamides:** Acetic anhydride (8 mmol, 2 equiv) and acetic acid (12 mmol, 3 equiv) were added to a solution of ketoxime **1a-o** (4 mmol, 1 equiv, 0.2 M) in THF (20 mL). The solution was purged with nitrogen for 15 minutes. Iron (II) acetate (8 mmol, 2 equiv) was then added and the resulting mixture was stirred at reflux under nitrogen for 5-12 hours and then cooled to room temperature. The reaction mixture was diluted with water (20 mL) and neutralized with 10% NaHCO<sub>3</sub> solution (~ 10 mL) until the pH was approximately 5.0. The mixture was then extracted twice with EtOAc (2 × 20 mL). The combined organic solution was washed sequentially with 10% NaHCO<sub>3</sub> solution (20 mL) and brine (20 mL), dried over sodium sulfate, and concentrated. The pure desired compound **2a-o** was obtained by silica gel column chromatography (EtOAc/hexanes as eluent).

*N*-(1-(4-Bromophenyl)vinyl)acetamide (**2a**):<sup>1</sup> 90% yield; mp 112-113 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 9.38 (s, 1H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 5.56 (s, 1H), 5.03 (s, 1H), 2.00 (s, 3H); <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>) δ 169.0, 140.6, 137.1, 131.1, 128.3, 121.3, 102.9, 23.6.

*N*-(1-Phenylvinyl)acetamide (**2b**):<sup>2,3</sup> 78% yield; mp 82-84 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 9.32 (s, 1H), 7.45-7.42 (m, 2H), 7.39-7.33 (m, 3H), 5.62 (s, 1H), 4.98 (s, 1H), 2.01 (s, 3H).

*N*-(1-(4-Methoxyphenyl)vinyl)acetamide (**2c**):<sup>3,4</sup> 82% yield; mp 94-95 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 9.22 (s, 1H), 7.37 (d, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.7 Hz, 2H), 5.52 (s, 1H), 4.90 (s, 1H), 3.77 (s, 3H), 2.00 (s, 3H).

*N*-(1-(4-(Trifluoromethyl)phenyl)vinyl)acetamide (**2d**):<sup>3,4</sup> 87% yield; mp 113-115 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 9.51 (s, 1H), 7.73 (d, *J* = 8.3 Hz, 2H), 7.64 (d, *J* = 8.3 Hz, 2H), 5.61 (s, 1H), 5.13 (s, 1H), 2.02 (s, 3H).

*N*-(1-(2-Methoxyphenyl)vinyl)acetamide (**2e**):<sup>5</sup> 77% yield; mp 124-125 °C; <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 9.15 (s, 1H), 7.33 (td, *J* = 8.0, 1.8 Hz, 1H), 7.19 (dd, *J* = 7.4, 1.8 Hz, 1H), 7.02 (d, <sup>3</sup>*J* = 8.2 Hz, 1H), 6.94 (t, *J* = 7.6 Hz, 1H), 5.82 (s, 1H), 4.54 (s, 1H), 3.77 (s, 3H), 1.92 (s, 3H).

*N*-(1-(4-Nitrophenyl)vinyl)acetamide (**2f**):<sup>6</sup> 45% yield; mp 148-149 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.22 (d, *J* = 8.5 Hz, 2H), 7.58 (d, *J* = 8.5 Hz, 2H), 6.87 (brs, 1H), 5.83 (s, 1H), 5.26 (s, 1H), 2.17 (s, 3H).

*N*-(1-(Naphthalen-2-yl)vinyl)acetamide (**2g**):<sup>2</sup> 77% yield; mp 123-124 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.87-7.82 (m, 5H), 7.52-7.48 (m, 2H), 6.91 (br s, 1H), 5.96 (s, 1H), 5.24 (s, 1H), 2.19 (s, 3H).

*N*-(1-(4-Methoxyphenyl)prop-1-enyl)acetamide (**2h**, 1/1.5 *E/Z* mixtures):<sup>3</sup> 80% yield; *Z* isomer: <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>) δ 9.03-8.99 (m, 1H), 7.19 (d, *J* = 8.5 Hz, 2H), 6.94 (d, *J* = 8.5 Hz, 2H), 5.97 (q, *J* = 7.1 Hz, 1H), 3.77-3.73 (m, 3H), 1.89 (s, 3H), 1.65-

1.58 (m, 3H); *E* isomer:  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  9.03-8.99 (m, 1H), 7.29 (d,  $J$  = 8.7 Hz, 2H), 6.87 (d,  $J$  = 8.7 Hz, 2H), 5.77 (q,  $J$  = 6.8 Hz, 1H), 3.77-3.73 (m, 3H), 1.99 (s, 3H), 1.65-1.58 (m, 3H).

*N*-(2-Methyl-1-phenylprop-1-enyl)acetamide (**2i**):<sup>1,7</sup> 66% yield; mp 82-83 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  9.00 (s, 1H), 7.33-7.29 (m, 2H), 7.24-7.19 (m, 3H), 1.89 (s, 3H), 1.71 (s, 3H), 1.69 (s, 3H).

*N*-(Cyclohexylidene(phenyl)methyl)acetamide (**2j**):<sup>1,7</sup> 67% yield; mp 130-131 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  8.98 (s, 1H), 7.31 (m, 2H), 7.22 (m, 1H), 7.18 (m, 2H), 2.17-2.11 (m, 4H), 1.87 (s, 3H), 1.57-1.52 (m, 4H), 1.50-1.46 (m, 2H).

*N*-(3,4-Dihydronaphthalen-1-yl)acetamide (**2k**):<sup>1,7,8</sup> 80% yield; mp 133-134 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  9.11 (s, 1H), 7.22-7.14 (m, 4H), 6.16 (t,  $J$  = 4.6 Hz, 1H), 2.69 (t,  $J$  = 7.9 Hz, 2H), 2.29-2.23 (m, 2H), 2.02 (s, 3H).

*N*-(1*H*-inden-3-yl)acetamide (**2l**):<sup>1,7,8</sup> 50% yield; mp 126-127 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  9.72 (s, 1H), 7.77 (d,  $J$  = 7.6 Hz, 1H), 7.47 (d,  $J$  = 7.4 Hz, 1H), 7.33 (t,  $J$  = 7.3 Hz, 1H), 7.23 (t,  $J$  = 7.2 Hz, 1H), 6.75 (t,  $J$  = 2.2 Hz, 1H), 3.37 (d,  $J$  = 2.2 Hz, 2H), 2.13 (s, 3H).

*N*-(2-Methyl-3,4-dihydronaphthalen-1-yl)acetamide (**2m**):<sup>7,8</sup> 55% yield; mp 153-154 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  8.93 (s, 1H), 7.15-7.07 (m, 3H), 7.04 (d,  $J$  = 7.6 Hz, 1H), 2.73 (t,  $J$  = 8.1 Hz, 2H), 2.29 (t,  $J$  = 8.1 Hz, 2H), 2.03 (s, 3H), 1.77 (s, 3H).

*N*-(6-Methoxy-3,4-dihydronaphthalen-1-yl)acetamide (**2n**):<sup>7</sup> 82% yield; mp 126-128 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  9.03 (s, 1H), 7.13 (d,  $J$  = 8.4 Hz, 1H), 6.79-6.71 (m, 2H), 6.03 (t,  $J$  = 4.5 Hz, 1H), 3.74 (s, 3H), 2.68-2.63 (m, 2H), 2.26-2.20 (m, 2H), 2.00 (s, 3H).

*N*-(5,7-Dimethyl-3,4-dihydronaphthalen-1-yl)acetamide (**2o**):<sup>7</sup> 74% yield; mp 162-163 °C;  $^1\text{H}$  NMR (500 MHz, DMSO- $d_6$ )  $\delta$  9.01 (s, 1H), 6.89 (s, 1H), 6.87 (s, 1H), 6.14 (t,  $J$  = 4.6 Hz, 1H), 2.57 (t,  $J$  = 7.8 Hz, 2H), 2.25-2.18 (m, 8H), 2.01 (s, 3H).

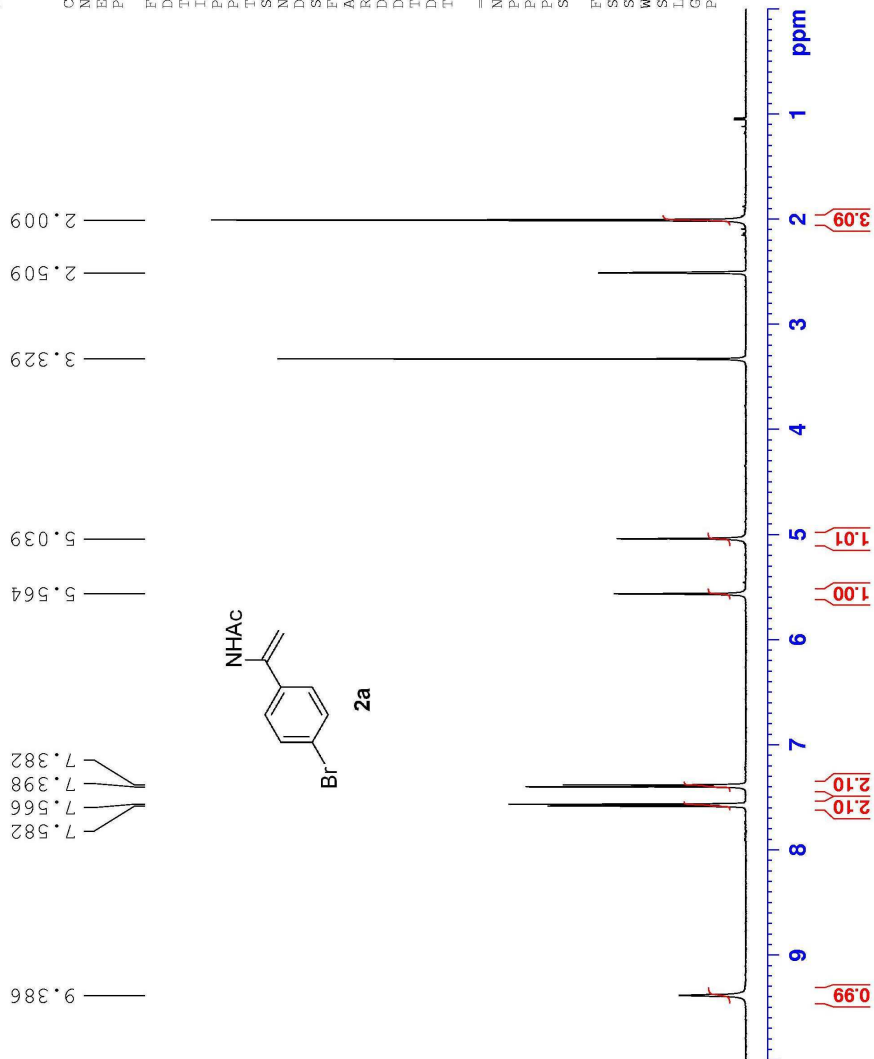
### References for Supporting Information

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101535-30



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101535-30



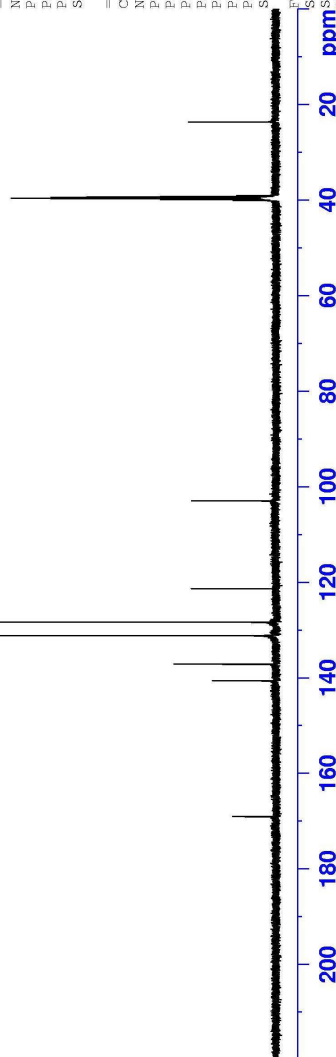
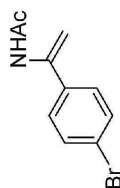
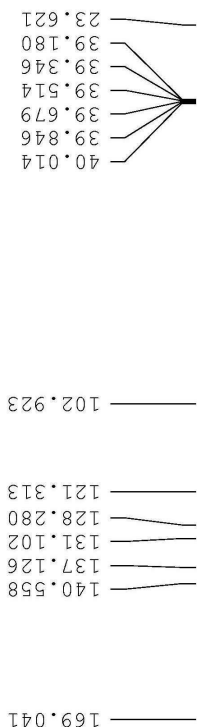
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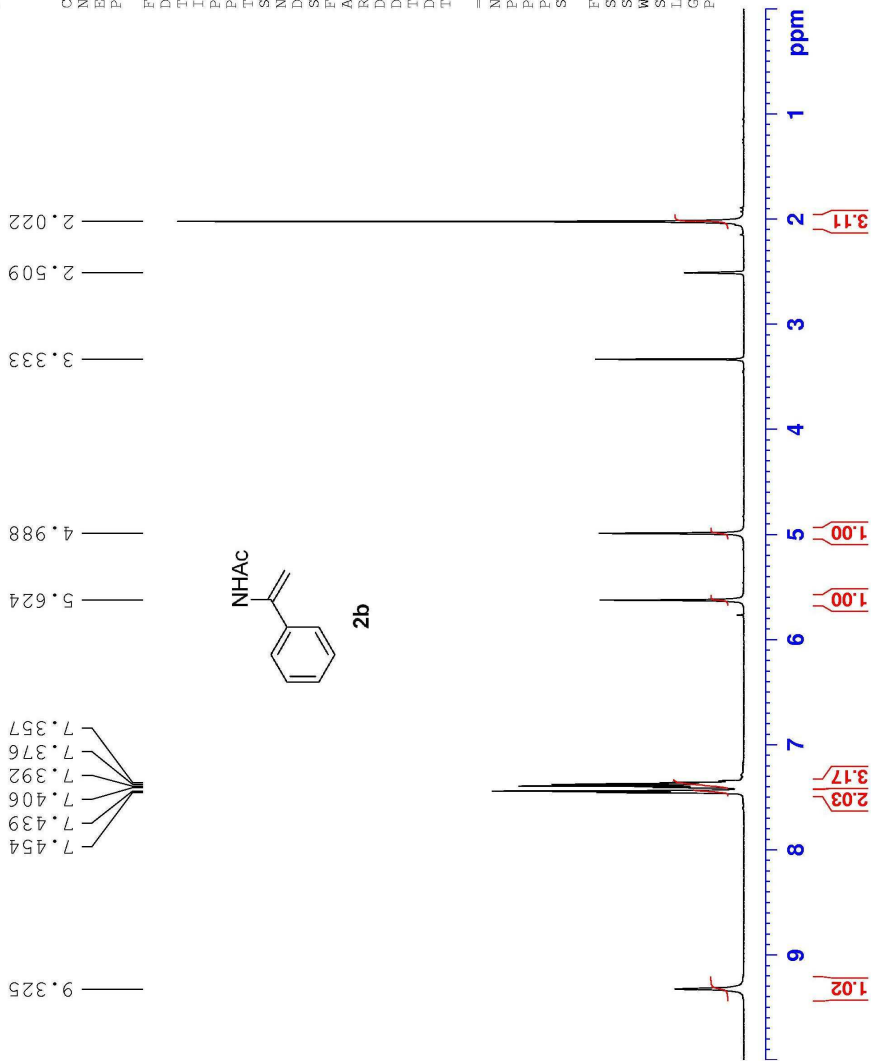
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9439-97-A



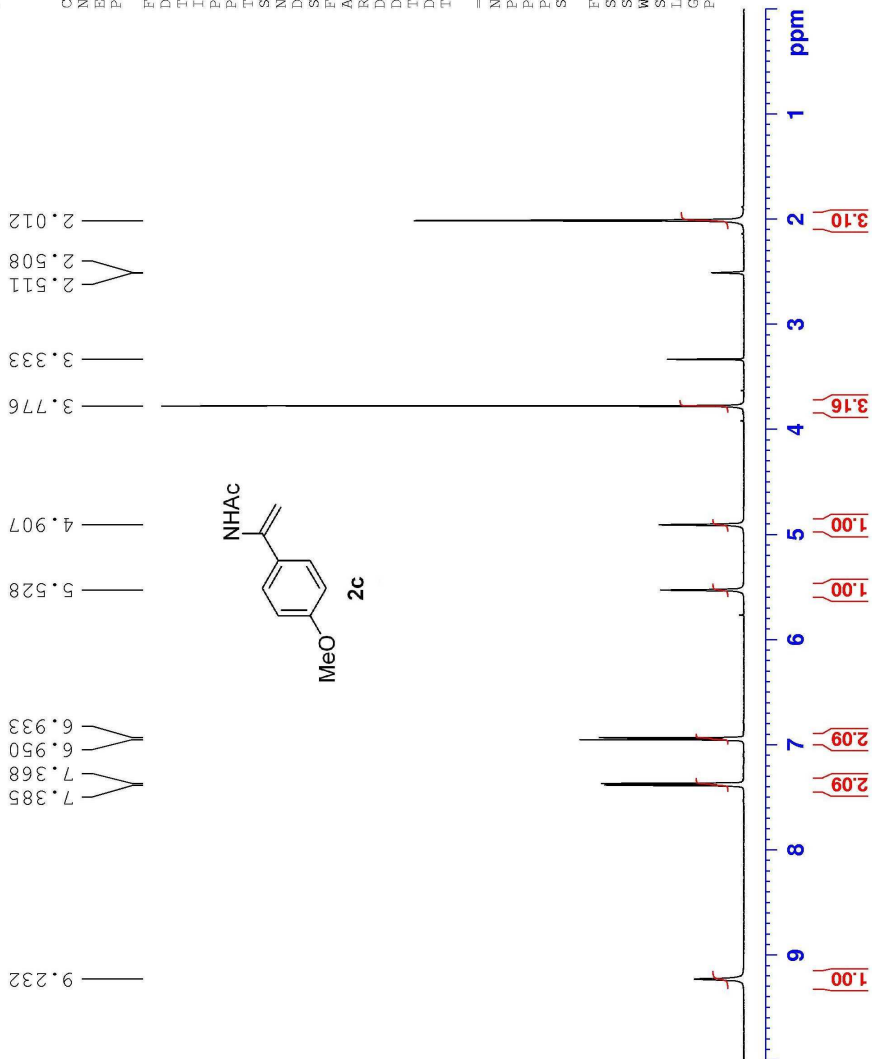
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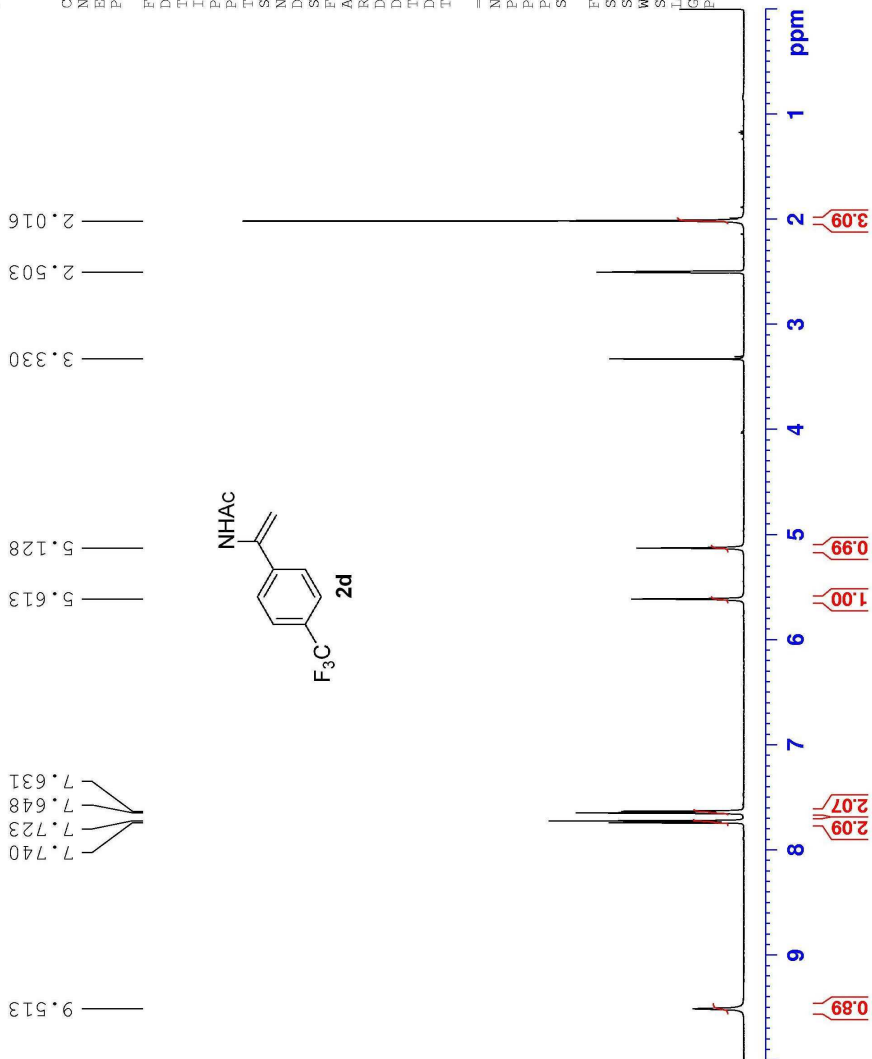
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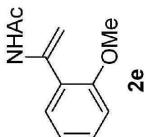
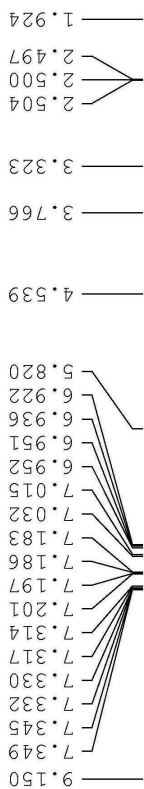




9439-12 in DMSO



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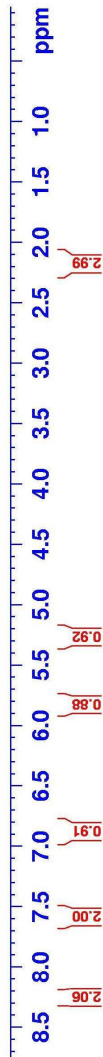
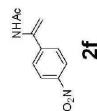
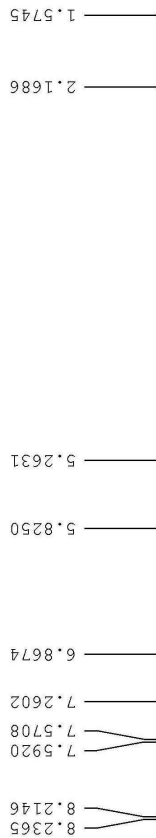


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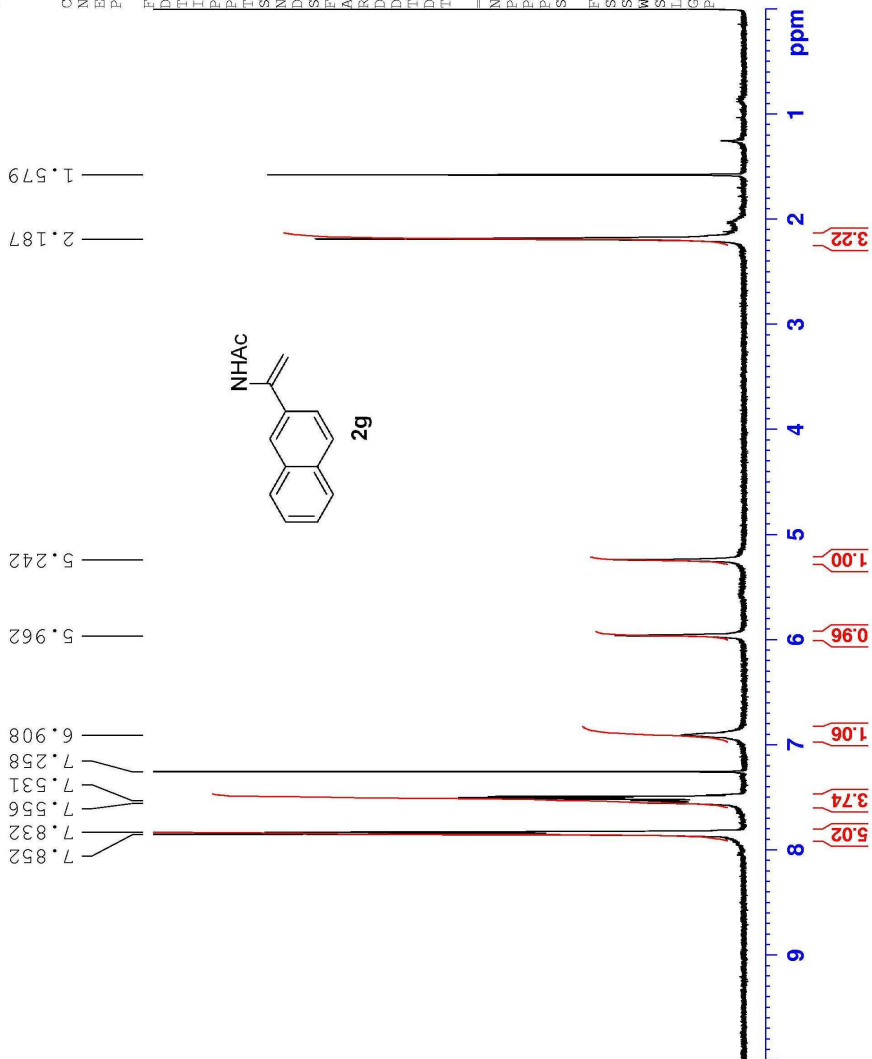
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9439-24



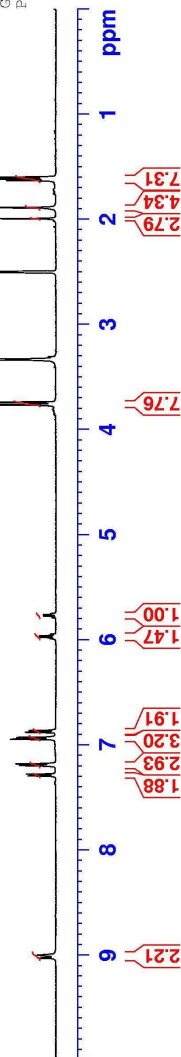
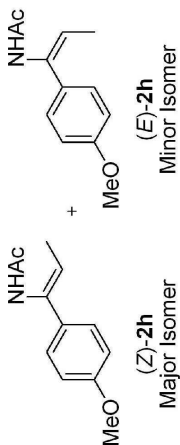
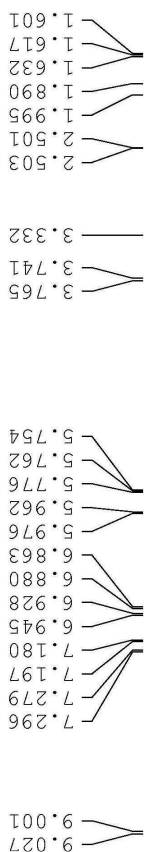
9439-102-C



Current Data Parameters  
NAME 9439-102-C  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20091007  
Time 5.42  
INSTRUM spect  
PROBHD 5 mm PABBI 1H-  
PULPROG zg10  
TD 32768  
SOLVENT DMSO  
NS 16  
DS 0  
SWH 7500.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 575  
DW 66.667 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 1

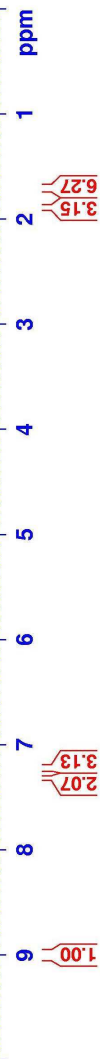
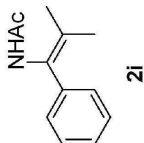
CHANNEL f1  
NUC1 1H  
P1 7.78 usec  
PL1 3.00 dB  
PL1W 14.98121262 W  
SF01 500.1325007 MHz  
F2 - Processing parameters  
SI 16384  
SF 500.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



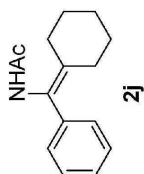
9439-108-C



Current Data Parameters  
 NAME 9439-108-C  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20091007  
 Time 5.45  
 INSTRUM spect  
 PROBHD 5 mm PABBI 1H-  
 PULPROG zg10  
 TD 32768  
 SOLVENT DMSO  
 NS 16  
 DS 0  
 SWH 7500.000 Hz  
 FIDRES 0.228882 Hz  
 AQ 2.1845834 sec  
 RG 575  
 DW 66.667 usec  
 DE 6.50 usec  
 TE 298.0 K  
 D1 1.0000000 sec  
 TD0 1  
 CHANNEL f1  
 NUC1 1H  
 P1 7.78 usec  
 PL1 3.00 dB  
 PL1W 14.98121262 W  
 SF01 500.1325007 MHz  
 F2 - Processing parameters  
 SI 16384  
 SF 500.1300037 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



9439-108-D

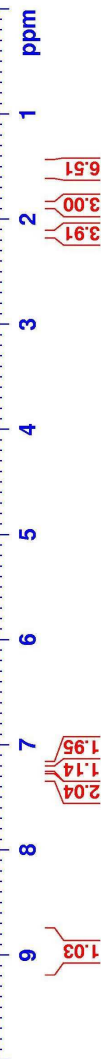


Current Data Parameters  
NAME 9439-108-d  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090821  
Time 9.37  
INSTRUM spect  
PROBHD 5 mm PABBI 1H-  
PULPROG zg10  
TD 32768  
SOLVENT DMSO  
NS 16  
DS 0  
SWH 7500.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 406  
DW 66.667 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 1

CHANNEL f1  
NUC1 1H  
P1 7.78 usec  
PL1 3.00 dB  
PL1W 14.98121262 W  
SF01 500.1325007 MHz

F2 - Processing parameters  
SI 16384  
SF 500.1300044 MHz  
WDW EM  
SSB 0  
LB 0  
GB 0  
PC 1.00



9439-21 in DMSO



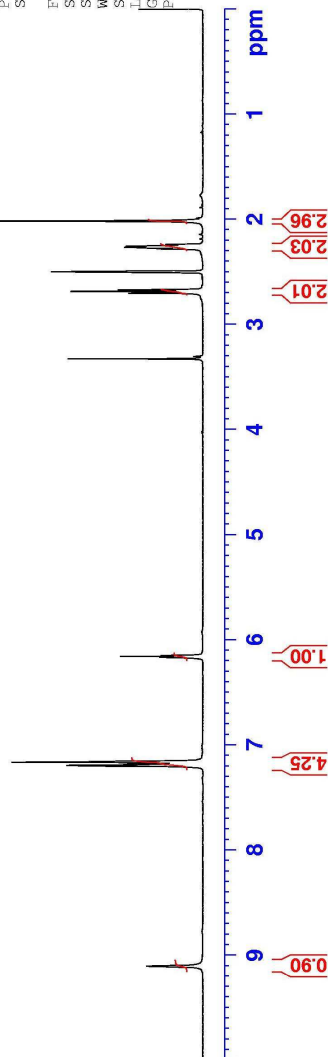
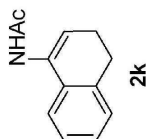
Current Data Parameters  
NAME 9439-21-3  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20090625  
Time 9.49  
INSTRUM spect  
PROBHD 5 mm PABBI 1H-  
PULPROG zg10  
TD 32768  
SOLVENT DMSO  
NS 16  
DS 0  
SWH 7500.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 645  
DW 66.667 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 1

CHANNEL f1  
NUC1 1H  
P1 8.00 usec  
PL1 3.00 dB  
PL1W 14.98121262 W  
SF01 500.1325007 MHz

F2 - Processing parameters  
SI 16384  
SF 500.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

9.107  
7.195  
7.186  
7.165  
7.158  
6.168  
6.159  
6.150  
3.329  
2.704  
2.688  
2.672  
2.500  
2.282  
2.267  
2.257  
2.241  
2.018

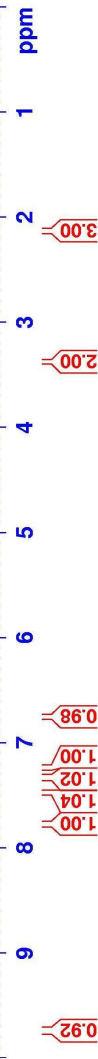
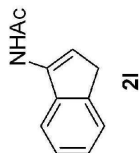


9439-95



Current Data Parameters  
 NAME 9439-95  
 EXPNO 2  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20091020  
 Time 11.38  
 INSTRUM spect  
 PROBHD 5 mm PABBI 1H-  
 PULPROG zg10  
 TD 32768  
 SOLVENT DMSO  
 NS 16  
 DS 0  
 SWH 7500.000 Hz  
 FIDRES 0.228882 Hz  
 AQ 2.1845834 sec  
 RG 1440  
 DW 66.667 usec  
 DE 6.50 usec  
 TE 298.0 K  
 D1 1.0000000 sec  
 TD0 1  
 CHANNEL f1  
 NUC1 1H  
 P1 7.78 usec  
 PL1 3.00 dB  
 PL1W 14.98121262 W  
 SF01 500.1325007 MHz  
 F2 - Processing parameters  
 SI 16384  
 SF 500.1300000 MHz  
 EQ 1H  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00

9.738  
 7.782  
 7.766  
 7.482  
 7.467  
 7.342  
 7.327  
 7.313  
 7.247  
 7.233  
 7.218  
 6.755  
 6.750  
 6.746  
 3.376  
 3.372  
 3.328  
 2.516  
 2.512  
 2.509  
 2.505  
 2.131





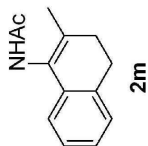
9439-110



Current Data Parameters  
NAME 9439-110  
EXPNO 5  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20091020  
Time 16.24  
INSTRUM spect  
PROBHD 5 mm PABBI 1H-  
PULPROG zg10  
TD 32768  
SOLVENT DMSO  
NS 16  
DS 0  
SWH 7500.000 Hz  
FIDRES 0.228882 Hz  
AQ 2.1845834 sec  
RG 1290  
DW 66.667 usec  
DE 6.50 usec  
TE 298.0 K  
D1 1.0000000 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 1H  
P1 7.78 usec  
PL1 3.00 dB  
PL1W 14.98121262 W  
SF01 500.1325007 MHz  
F2 - Processing parameters  
SI 16384  
SF 500.1300000 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

3.328  
2.747  
2.731  
2.715  
2.515  
2.512  
2.509  
2.505  
2.309  
2.293  
2.277  
2.026  
1.768

7.152  
7.138  
7.123  
7.110  
7.099  
7.097  
7.085  
7.045  
7.030

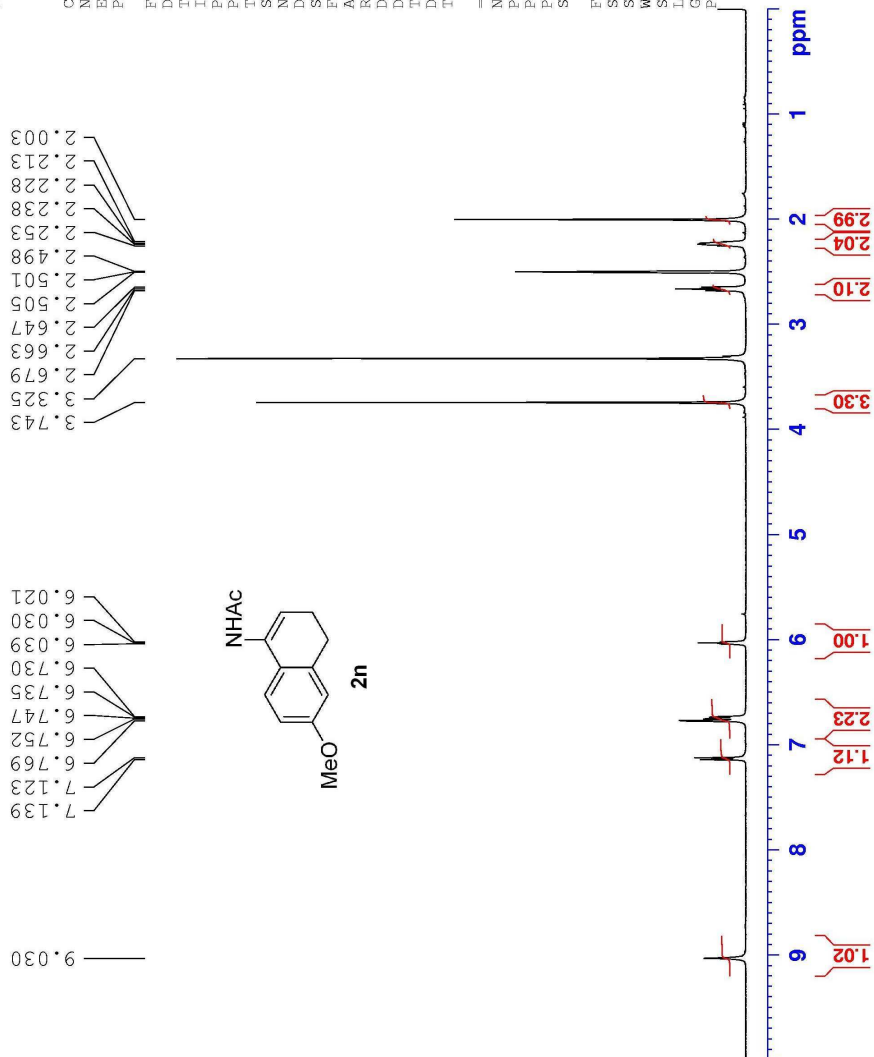


8.934

0.89 3.04 0.93 1.89 2.00 2.79 2.81

ppm

|                             |                 |
|-----------------------------|-----------------|
| Current Data Parameters     |                 |
| NAME                        | 9439-93-a       |
| EXPNO                       | 1               |
| PROCNO                      | 1               |
| F2 - Acquisition Parameters |                 |
| Date_                       | 20090821        |
| Time                        | 9.41            |
| INSTRUM                     | spect           |
| PROBHD                      | 5 mm PABBI 1H-  |
| PULPROG                     | zg10            |
| TD                          | 32768           |
| SOLVENT                     | DMSO            |
| NS                          | 16              |
| DS                          | 0               |
| SWH                         | 7500.0 MHz      |
| FIDRES                      | 0.228882 Hz     |
| AQ                          | 2.1845834 sec   |
| RG                          | 912             |
| DW                          | 66.667 usec     |
| DE                          | 6.50 usec       |
| TE                          | 298.0 K         |
| D1                          | 1.0000000 sec   |
| TD0                         | 1               |
| ===== CHANNEL f1 =====      |                 |
| NUC1                        | 1H              |
| P1                          | 7.78 usec       |
| PL1                         | 3.00 dB         |
| PL1W                        | 14.98121262 W   |
| SFO1                        | 500.1325007 MHz |
| F2 - Processing parameters  |                 |
| SI                          | 16394           |
| SF                          | 500.1300042 MHz |
| WDW                         | EM              |
| SSB                         | 0               |
| LB                          | 0.30 Hz         |
| GB                          | 0               |
| PC                          | 1.00            |



9439-93-B



Current Data Parameters  
 NAME 9439-93-b  
 EXPNO 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20090821  
 Time 9.45  
 INSTRUM spect  
 PROBHD 5 mm PABBI 1H-  
 PULPROG zg10  
 TD 32768  
 SOLVENT DMSO  
 NS 16  
 DS 0  
 SWH 7500.000 Hz  
 FIDRES 0.228882 Hz  
 AQ 2.1845834 sec  
 RG 575  
 DW 66.667 usec  
 DE 6.50 usec  
 TE 298.0 K  
 D1 1.0000000 sec  
 TD0 1

CHANNEL f1  
 NUC1 1H  
 P1 7.78 usec  
 PL1 3.00 dB  
 PL1W 14.98121262 W  
 SF01 500.1325007 MHz  
 F2 - Processing parameters  
 SI 16384  
 SF 500.1300048 MHz  
 WDW EM  
 SSB 0  
 LB 0  
 GB 0  
 PC 1.00

9.009  
 6.894  
 6.876  
 6.153  
 6.144  
 6.135  
 3.325  
 2.591  
 2.575  
 2.560  
 2.503  
 2.500  
 2.496  
 2.230  
 2.203  
 2.011

