Design, Synthesis and Biological Evaluation of Novel 1,3-Diarylpyrazoles as Cyclooxygenase Inhibitors, Antiplatelet, and Anticancer Agents

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General procedure for the preparation of 3/4-[(1E)-1-(2-phenylhydraziniliden)ethyl] pyridine derivatives (2a, 7)

A solution of acetylpyridine derivative (0.052 mol), phenyl hydrazine (6.27 g, 0.058 mol) and acetic acid (2 ml, 0.035 mol) in ethanol was stirred for 2 h at reflux, and then evaporated. The precipitated was filtered off and dried.

**N-Phenyl-\(N'-(1\text{-pyridine-3-il-ethyliden})\)hydrazine 2a**

Yield %90, mp 138.8 – 140 °C [1]. IR (FTIR/FTNIR-ATR): 3169 cm\(^{-1}\) (N-H), 2993 cm\(^{-1}\) (aliphatic C-H). \(^1\)H-NMR (CDCl\(_3\)) \(\delta\): 8.98 (1H, d, \(J=2.4 \) Hz), 8.53 (1H, dd, \(J=1.6 \) Hz, 4.8 Hz), 8.10 (1H, dt, \(J=2 \) Hz, 8 Hz), 7.47 (1H, s), 7.31-7.26 (3H, m), 7.18 (2H, d, \(J=7.2 \) Hz), 6.91 (1H, t, \(J=7.2 \) Hz), 2.26 (3H, s). HRMS C\(_{13}\)H\(_{14}\)N\(_3\) [M+H]\(^+\) Calc. 212.1188, Found m/z 212.1187. Anal. Calc. (%) for C\(_{13}\)H\(_{13}\)N\(_3\) Calc. % C: 73.91 H: 6.20 N: 19.89, Found % C: 74.27 H: 6.08 N: 19.60.

**4-(1-(2-phenylhydrazono)ethyl)pyridine 7**

Yield 85%, mp 147°C (Lit. Mp. [1, 2]: 148-149 °C). IR (FTIR/FTNIR-ATR): 3227 cm\(^{-1}\) (N-H), 1941 cm\(^{-1}\) (C=N). \(^1\)H-NMR (CDCl\(_3\)) \(\delta\): 8.58-8.60 (2H, d, \(J=6.4 \) Hz), 7.65 (2H, d, \(J=6 \) Hz), 7.59 (1H, s), 7.29-7.33 (2H, t, \(J=7.6 \) Hz), 7.19-7.22 (2H, d, \(J=7.6 \) Hz), 6.92-6.95 (1H, m, \(J=6.8 \) Hz, \(J=7.6 \) Hz), 2.22 (3H, s). HRMS C\(_{13}\)H\(_{13}\)N\(_3\) [M+H]\(^+\) Calc. 212.1188, Found m/z 212.1180. Anal. Calc. (%) for C\(_{13}\)H\(_{13}\)N\(_3\). C: 73.91 H: 6.20 N: 19.89, Found C: 74.21 H: 6.22 N: 19.38.

General procedure for the preparation of 1-phenyl-3-(pyridine-3/4-yl)-1H-pyrazole-4-carbaldehyde derivatives (3a,8)

In a dry flask, phosphoroxy chloride (POCl\(_3\)) (0.124 mol) was added drop wise to an ice-cold stirred solution of hydrazon derivative (0.041 mol) in 80 ml DMF. The reaction mixture was allowed to attain room temperature, and then heated at 50 °C for 4 h. The resulting mixture was poured onto crushed ice, neutralized with dilute NaOH and left overnight. The yellow precipitate obtained was purified by crystallization in toluene.

**1-Phenyl-3-(pyridine-3-yl)-1H-pyrazole-4-carbaldehyde 3a**

Yield %85, mp 158.8–160 °C [1]. IR (FTIR/FTNIR-ATR): 1673 cm\(^{-1}\) (C=O). \(^1\)H-NMR (CDCl\(_3\)) \(\delta\): 10.06 (1H, s), 9.12 (1H, d, \(J=2.4 \) Hz), 8.70 (1H, dd, \(J_a=1.6 \) Hz, \(J_b=4.8 \) Hz), 8.58 (1H, s), 8.47 (1H, dt, \(J_a=2 \) Hz, \(J_b=8 \) Hz), 7.81 (2H, d, \(J=7.6 \) Hz), 7.56-7.52 (2H, m), 7.46-7.41 (2H, m). HRMS C\(_{15}\)H\(_{12}\)N\(_3\)O [M+H]\(^+\) Calc. 250.0980, Found m/z 250.0979.

3
Anal. Calc. (%) for C_{15}H_{12}N_{3}O Calc. % C: 72.28 H: 4.45 N: 16.86, Found % C: 72.06 H: 4.46 N: 16.77

1-Phenyl-3-(pyridine-4-yl)-1H-pyrazole-4-carbaldehyde 8

Yield 13.8 (92%), mp 147 – 149 °C [1]. IR (FTIR/FTNIR-ATR): 1669 cm\(^{-1}\) (C=O). \(^{1}\)H-NMR (DMSO-\(d_6\)) \(\delta\): 10.04 (1H, s), 9.43 (1H, s), 8.73 (2H, d, \(J=1.6\) Hz), 8.02-7.97 (4H, m), 7.62-7.58 (2H, m), 7.48-7.44 (1H, m). HRMS C_{15}H_{12}N_{3}O [M+H]\(^+\) Calc. 250.0980, Found m/z 250.0980. Anal. Calc. (%) for C_{15}H_{11}N_{3}O C: 72.20 H: 4.42 N: 17.07

General procedure for the preparation of (2E)-3-[1-Substituted phenyl-3-(pyridine-3/4-yl)-1H-pyrazole-4-yl]prop-2-enoic acid derivatives (4a, 9)

To a solution of 1-phenyl-3-(pyridin-3/4-yl)-1H-pyrazole-4-carbaldehyde (8.72 mmol) in pyridine (20 ml), malonic acid (0.035 mol), and piperidine (0.0131 mol) were added, and the reaction mixture was refluxed for 4 h. On cooling, the reaction mixture was poured onto a solution (100 ml) of crushed ice and concentrated HCl (50% by volume) mixture, then, pH was adjusted to 5. The resulting precipitated was filtered off, washed with acidified water and dried.

(2E)-3-[1-Phenyl-3-(pyridine-3-yl)-1H-pyrazole-4-yl]acrylic acid 4a

Yield %91, mp 240–242 °C [1]. IR (FTIR/FTNIR-ATR): 1668 cm\(^{-1}\) (C=O). \(^{1}\)H-NMR (DMSO-\(d_6\)) \(\delta\): 12.38 (1H, s), 9.27 (1H, s), 8.84 (1H, d, \(J=1.6\) Hz), 8.69 (1H, dd, \(J=1.6\) Hz, \(J=5.2\) Hz), 8.05 (1H, dt, \(J=2\) Hz, 7.6 Hz), 7.94 (2H, d, \(J=7.6\) Hz), 7.61-7.55 (2H, m), 7.48 (1H, d, \(J=15.6\) Hz), 7.41 (2H, m), 6.48-6.44 (1H, d, \(J=16\) Hz). HRMS C_{17}H_{14}N_{3}O_{2} [M+H]\(^+\) Calc. 292.1086, Found m/z 292.1095.

(2E)-3-[1-Phenyl-3-(pyridine-4-yl)-1H-pyrazol-4-yl]acrylic acid 9

Yield 78%, mp 287 – 288 °C [1]. IR (FTIR/FTNIR-ATR): 1681cm\(^{-1}\) (C=O), 2440 cm\(^{-1}\) (C=C). \(^{1}\)H-NMR (DMSO-\(d_6\)) \(\delta\): 9.27 (1H, s), 8.75-8.73 (2H, d, \(J=6\) Hz), 7.96-7.93 (2H, d, \(J=8.4\) Hz), 7.67-7.65 (2H, d, \(J=6.4\) Hz), 7.60-7.53 (3H, m), 7.43-7.40 (1H, t, \(J=7.6\) Hz), 6.51-6.47 (1H, d, \(J=16\) Hz). HRMS C_{17}H_{14}N_{3}O [M+H]\(^+\) Calc. 292.1086, Found m/z 292.1089. Anal. Calc. (%) for C_{17}H_{13}N_{3}O C: 70.09 H: 4.50 N: 14.42, Found C: 69.92 H: 4.65 N: 14.34
Figure S1. Spectral data of Compound 2a

Figure S2. Spectral data of Compound 2b
Figure S3. Spectral data of Compound 7

Figure S4. Spectral data of Compound 3a
Figure S5. Spectral data of Compound 3b
**Elemental Composition Report**

**Single Mass Analysis**

- **Tolerance = 5.0 PPM** / **DBE: min = -1.5, max = 50.0**
- **Element prediction: Off**
- **Number of isotope peaks used for i-FIT = 3**

Monoisotopic Mass, Even Electron Ions

125 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:
- C: 1-40
- H: 1-40
- N: 1-8
- O: 1-5
- S: 1-1

**Ni-272-2.71 (2.763) Cm (69.71)**

**i: TOF MS ESI**

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<th>PPM</th>
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<th>i-FIT</th>
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**Minimum:** 5.0  5.0  -1.5  50.0

**Maximum:**

**Figure S6. Spectral data of Compound 8**

**Hi-Eq. 13 May 2011**

Archive directory: /export/home/mwet/sequoia/data
Sample Directory: Hi-Eq.13May011
File: PROTON

Dipole frequency: 24.59

Solvent: DMSO

Average temperature: 273K

**Hioe11**

Relax delay 1.000 sec
relax 4.500 degrees
Aqua time 1.000 sec
width 640.0 Hz
10 repetitions

COHERENCE 5K, 400.253907 MHz

DATA PROCESSING

FT size 32768

Total time 6 min, 59 sec
Figure S7. Spectral data of Compound 4a

Figure S8. Spectral data of Compound 4b
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
158 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:

Ni-275 57 (2.233) Cm (57:60)
1: TOF MS ES+

Minimum: 5.0  5.0  50.0
Maximum: 100  100  100
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
370.0852  370.0862  -1.0  -2.7  12.5  257.3  0.0  C18 H16 N3 O4 S
Figure S9. Spectral data of Compound 9

Figure S10. Spectral data of Compound 5a
**Elemental Composition Report**

**Single Mass Analysis**
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
100 formula(s) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
Ni_288.62 (2.012) Cm (52.55)
1: TOF MS ES+

Overview of Masses:

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<th>FPP</th>
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<td>0.6</td>
<td>C23 H23 N6 O</td>
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Figure S11. Spectral data of Compound 5b
**Elemental Composition Report**

**Single Mass Analysis**
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
26 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:

N=274 50 (1.949) Cm (49.52)
1: TOF MS ES+

**Figure S12. Spectral data of Compound 5c**
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM  /  DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
75 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:

Ni-278X 52 (2.012) Cm (52.5%) 1: TOF MS ES+

Minimum:
Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm) Formula
399.1918  399.1920  -0.2  -0.5  10.5  180.2  1.0  C22 H27 N2 O5
399.1933  -1.5  -3.8  15.5  179.6  0.4  C23 H23 N6 O
Figure S13. Spectral data of Compound 5d
Figure S14. Spectral data of Compound 5e
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
188 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
Ni283.49 (1.916) Cm (48:50)
t: TOF MS ES+

Minimum: 5.0  5.0  -1.5
Maximum: 50.0
Mass  Calcd. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
382.1678  382.1668  1.0  2.6  16.5  436.6  0.0  C23 H20 N5 O
Figure S15. Spectral data of Compound 5f
**Figure S16. Spectral data of Compound 5g**

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**Elemental Composition Report**

**Single Mass Analysis**
- Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
- Element prediction: Off
- Number of isotope peaks used for i-FIT = 3

**Monoisotopic Mass, Even Electron Ions**
- 21 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
- Elements Used:
  - C: 1-30
  - H: 1-30
  - N: 1-8
  - O: 1-1
  - N267 51 (1.981) Cm (51:54)
  - 1: TOF MS ES+

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<td>382.1676</td>
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**Additional Spectral Information**

**File:** 399_2014032001

**Archive directory:** /home/analyst/massspec/data

**Sample directory:** mi-264_2014032001

**Pulse Sequence:** Signal

**Solvent:** Chloroform

**Temp.:** 23.5 °C / 298.1 K

**Number of scans:** 32

**Scan delay:** 3.00 s

**Pulse 60.0° deg.**

**Acq. time:** 3.91 s

**Width 1420.0 ms**

**6 experiments**

**OBSERVED:** 13.456789 MHz

**DATA PROCESSED**

**FT field 32768**

**Total time:** 0 min, 15 sec

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**Diagram:**

[Graph showing spectral data]
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
101 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Elements Used:
N: 298.54 (2.104) Cm (54.56)
1: TOF MS ES+

Maximum:

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Figure S17. Spectral data of Compound 5h
Figure S18. Spectral data of Compound 5i
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
27 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:

1: TOF MS ES+

Minimum: 5.0 5.0 -1.5
Maximum: 100.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
396.1806  396.1824  -1.8  -4.5  16.5  115.8  0.0  C24 H22 N5 O
Figure S19. Spectral data of Compound 5j
### Figure S20. Spectral data of Compound 5k

**MS-312_1573c0811**

- **Archive directory:** /export/home/serai/research/data
- **Sample directory:** MS-312_1573c0811
- **File:** PROTON
- **Pulse Sequence:** a2p2l
- **Solvent:** DMSO
- **Temperature:** Room temp
- **Mass range:** 50-2000 Da

- **Parameters:**
  - Delay 1.000 sec
  - Pulse 60.0 degrees
  - Accum. time 1.861 sec
  - Width 0.002 Da
  - 18 acquisitions

- **Headers:**
  - **Column:** 1.490 AM
  - **Dimensions:** 37.5 MHz

- **Total time:** 6 min, 31 sec
**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

160 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Elements Used:

Ni-312.47 (1.821) Cm (47.49)

1: TOF MS ES+

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Figure S21. Spectral data of Compound 5I
Figure S22. Spectral data of Compound 5m
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron ions
395 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
N=292.36 (1.382)
1: TOF MS ES+

Minimum: 5.0  5.0  -1.5
Maximum: 50.0

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>460.1447</td>
<td>460.1443</td>
<td>0.4</td>
<td>0.9</td>
<td>16.5</td>
<td>235.6</td>
<td>0.0</td>
<td>C24 H22 N5 O3 S</td>
</tr>
<tr>
<td>460.1437</td>
<td>1.0</td>
<td>2.2</td>
<td>7.5</td>
<td>241.0</td>
<td>5.3</td>
<td>C16 H26 N7 O5 S2</td>
<td></td>
</tr>
</tbody>
</table>
Figure S23. Spectral data of Compound 5n.
**Figure S24.** Spectral data of Compound 5o
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
395 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
M+293.2 42 (1.635) Cm (42.44)
1 TOF MS ES+

Minimum: 5.0 5.0 -1.5
Maximum: 50.0

Mass Calc. Mass mDa PPM DBE i-FIT i-FIT (Norm) Formula
460.1440 460.1443 -0.3 -0.7 16.5 118.9 0.0 C24 H22 N5 O3 S
460.1437 0.3 0.7 7.5 129.1 10.3 C16 H26 N7 O5 S2
Figure S25. Spectral data of Compound 5p
Figure S26. Spectral data of Compound 5r
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
200 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
Ni-314 45 (1.758) Cm (45.48)
1: TOF MS ES+

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<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>474.1578</td>
<td>474.1561</td>
<td>1.7</td>
<td>3.6</td>
<td>15.5</td>
<td>208.7</td>
<td>3.4</td>
<td>C₂₈ H₂₈ N O₂ S₂</td>
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<tr>
<td>474.1600</td>
<td>-2.2</td>
<td>-4.6</td>
<td>16.5</td>
<td>205.3</td>
<td>0.0</td>
<td>C₂₅ H₂₄ N₅ O₃ S</td>
<td></td>
</tr>
</tbody>
</table>
Figure S27. Spectral data of Compound 5s
Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
134 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
NI-310.44 (1.692)
1: TOF MS ES+

3.24e+004

Minimum: 5.0  5.0  -1.5
Maximum: 50.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
474.1590  474.1600  -1.0  -2.1  16.5  111.5  0.0  C25 H24 N5 O3 S

Figure S28. Spectral data of Compound 10a
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
75 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
N1-284 42 (1.636) Cm (41.64)
1: TOF MS ES+

<table>
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<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>399.1945</td>
<td>399.1933</td>
<td>1.2</td>
<td>3.0</td>
<td>15.5</td>
<td>497.9</td>
<td>0.0</td>
<td>C23 H23 N6 O</td>
</tr>
</tbody>
</table>
Figure S29. Spectral data of Compound 10b
**Elemental Composition Report**

**Single Mass Analysis**  
Tolerance = 5.0 PPM  /  DBE: min = -1.5, max = 50.0  
Element prediction: Off  
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions  
160 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)  
Elements Used:  

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>413.2079</td>
<td>413.2090</td>
<td>-1.1</td>
<td>-2.7</td>
<td>15.5</td>
<td>203.0</td>
<td>0.0</td>
<td>C24 N25 N6 O</td>
</tr>
</tbody>
</table>

Minimum:  
Maximum:  
5.0  5.0  50.0
Figure S30. Spectral data of Compound 10c
Figure S31. Spectral data of Compound 10d

EI-248_13AMa0011

Sample directory: /report/home/wendell/research/Data
Sample directory: EI-248_13AMa0011
File: PREP

Path Sequence: <unknown>
Solvent: H2SO
Ambient temperature: 298K
NMRs: 6H2O - "accurate400"

Delay: 1.008 sec
Pulse: 90.4 degrees
Avg. time 1.942 sec
Width 48.8 Hz
1F acquisitions
OSMSIR EI, 400.135613 MHz
DATA PROCESSING
FF line 33576
Total time 0 min, 0 sec
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM  /  DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
77 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:

191.5831  192.0867  382.1650
383.1700  384.1741

Minimum:  5.0  5.0  -1.5
Maximum:  50.0

Mass  Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm)  Formula
382.1650  382.1654  -0.4  -1.0  11.5  387.1  2.0  C22 H24 N5 O5
382.1668  -1.6  -4.7  16.5  385.2  0.1  C23 H26 N5 O
Figure S32. Spectral data of Compound 10e
Figure S33. Spectral data of Compound 10f

File: 10f_140200211
Archive directory: /export/home/report/sequoia/data
Sample directory: M1-102_140200211
File: PROTON
Pulse sequence: shgpol
Solvent: DMSO
Reagent temperature: 298K (25.0°C)
Make-up solvent: DMSO
Molar delay 1.000 sec
Power 62.5 degrees
Avg. time 1.992 sec
Width 600.0 Hz
n repetitions
Centered 64, 400.1759413 MHz
DATA PROCESSED
PT size 27760
Total time 0 min. 25 sec
Elemental Composition Report

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
77 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)
Elements Used:
Ni-282 43 (1.000) Cm (43.45)
1: TOF MS ES+

Minimum: 5.0 5.0 5.0 50.0
Maximum: 382.1663 382.1668 -0.5 -1.3 16.5 456.6 0.7
Calc. Mass  mDa  PPM  DBE  i-FIT  i-FIT (Norm) Formula
382.1663 382.1668 0.9 2.4 11.5 456.6 0.7
382.1654

Page 1 of 1

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Figure S34. Spectral data of Compound 10g
Figure S35. Spectral data of Compound 10h
**Elemental Composition Report**

**Single Mass Analysis**

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

45 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:


Ni-300 44 (1.693) Cm (44:46)

1: TOF MS ES+

---

<table>
<thead>
<tr>
<th>Mass</th>
<th>Calc. Mass</th>
<th>mDa</th>
<th>PPM</th>
<th>DBE</th>
<th>i-FIT</th>
<th>i-FIT (Norm)</th>
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<tbody>
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<td>396.1821</td>
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<td>-0.3</td>
<td>-0.8</td>
<td>16.5</td>
<td>414.1</td>
<td>0.0</td>
<td>C24 H22 N5 O</td>
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</tbody>
</table>
Figure S36. Spectral data of Compound 10i
References
