Supporting Informations

Palladium Catalyzed Regioselective Aroylation and Acetoxylation of 3,5-Diarylisoxazole via Ortho C–H Functionalizations

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Contents:

1. Preparation of starting material S2
2. Spectra of all compounds S3 – S58
Preparation of starting material:

3,5-Diarylisoazoles were prepared by the method of Click chemistry using phenyl acetylene derivatives with substituted aldoximes.

**Spectra**

Phenyl(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1a): $^1$H NMR (400 MHz, CDCl$_3$):
Phenyl(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1a): $^{13}$C NMR (100 MHz, CDCl$_3$):
Phenyl(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1a): Mass Spectra:

[Mass Spectral Figure]

Charge=175.0V

Molecular ion at m/z 326.1175

Mass to Charge (m/z): 305.1529
(5-Methyl-2-(5-phenylisoxazol-3-yl)phenyl)(phenyl)methanone (2a): $^1$H NMR (400 MHz, CDCl$_3$):
(5-Methyl-2-(5-phenylisoxazol-3-yl)phenyl)(phenyl)methanone (2a): $^{13}$C NMR (100 MHz, CDCl$_3$):
(5-Methyl-2-(5-phenyloxazol-3-yl)phenyl)(phenyl)methanone (2a): Mass Spectra:

Sample Name  | Position | Vial 1 | Instrument Name | Instrument 1 | User Name | IRM Calibration Status | Acquired Time
--- | --- | --- | --- | --- | --- | --- | ---
Inj Vol | InjPosition | ACQ Method | SampleType | Comment | | | 8/10/2012 11:57:10 AM

$^{+}$ESI Scan (12.2-27.6 sec, 17 scans) Frag=175.0V

340.1331

Counts (%) vs. Mass-to-Charge (m/z)
(5-Methoxy-2-(5-phenylisoxazol-3-yl)phenyl)(phenyl)methanone (3a): $^1$H NMR (400 MHz, CDCl$_3$):
(5-Methoxy-2-(5-phenylisoxazol-3-yl)phenyl)(phenyl)methanone (3a): $^{13}$C NMR (100 MHz, CDCl$_3$):
(5-Methoxy-2-(5-phenylisoxazol-3-yl)phenyl)(phenyl)methanone (3a): Mass Spectra:
(2-(5-Phenylisoxazol-3-yl)phenyl)(p-tolyl)methanone (1b): $^1$H NMR (400 MHz, CDCl$_3$):

![NMR Spectrum Image]
(2-(5-Phenylisoxazol-3-yl)phenyl)(p-tolyl)methanone (1b): $^{13}$C NMR (100 MHz, CDCl$_3$):
(2-(5-Phenylisoxazol-3-yl)phenyl)(p-tolyl)methanone (1b): Mass Spectra:
(4-tert-Butylphenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1c): $^1$H NMR (400 MHz, CDCl$_3$):
(4-tert-Butylphenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1c): \(^{13}\)C NMR (100 MHz, CDCl\(_3\)):
(4-tert-Butylphenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1c): Mass Spectra
(4-Methoxyphenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1d): $^1$H NMR (400 MHz, CDCl$_3$):

![NMR Spectrum]

**Experimental Details**
- **Spectral Data**: NMR
- **Sample**: N/A
- **Solvent**: CDCl$_3$
- **Temperature**: not used
- **Sample Type**: N/A
- **Acquisition Mode**: N/A
- **Signal**: N/A
- **Field**: N/A
- **Frequency**: 400 MHz
- **Processing**: N/A
- **Time**: Apr 5 2012

**Peak Assignment**
- **Ph**: N/A
- **OMe**: N/A
- **N**: N/A
- **O**: N/A
- **Ph**: N/A

**Notes**
- Electronic Supplementary Material (ESI) for RSC Advances
- This journal is © The Royal Society of Chemistry 2013
(4-Methoxyphenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1d): $^{13}$C NMR (100 MHz, CDCl$_3$):
(4-Methoxyphenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1d): Mass Spectra:
(3-Chlorophenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1e): $^1$H NMR (400 MHz, CDCl$_3$):
(3-Chlorophenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1e): $^{13}$C NMR (100 MHz, CDCl$_3$):
(3-Chlorophenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1e): Mass Spectra:

![Mass Spectra Graph]

- ESI Scan (4.4-16.0 sec, 13 scans) Frag=175.0V
- 360.0783

Electronic Supplementary Material (ESI) for RSC Advances
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[1, 1'-Biphenyl]-4-yl(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1f): $^1$H NMR (400 MHz, CDCl$_3$):
[1, 1'-Biphenyl]-4-yl(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1f): $^{13}$C NMR (100 MHz, CDCl$_3$):

![NMR spectrum image]
[1,1'-Biphenyl]-4-yl(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1f): Mass Spectra:
Methyl 4-(2-(5-phenylisoxazol-3-yl)benzoyl)benzoate (1g): \(^1\text{H} \text{NMR (400 MHz, CDCl}_3\)): 

![NMR Spectrum Image]
Methyl 4-(2-(5-phenylisoxazol-3-yl)benzoyl)benzoate (1g): $^{13}$C NMR (100 MHz, CDCl$_3$):
Methyl 4-(2-(5-phenylisoxazol-3-yl)benzoyl)benzoate (1g): Mass Spectra:

[Mass Spectra Diagram]

Sample Name: AB-313
Inj Vol: 10
Data Filename: AB-313.d

Instrument Name: Instrument 1
Sample Type: Sample
User Name: IRM Calibration Status: Success
Acquired Time: 9/6/2013 11:44:56 AM
(2,6-Dichlorophenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1h): $^1$H NMR (400 MHz, CDCl$_3$):
(2,6-Dichlorophenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1h): $^{13}$C NMR (100 MHz, CDCl$_3$):
(2,6-Dichlorophenyl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1h): Mass Spectra:

+ESI Scan (8.3-31.5 sec, 25 scans) Frag=175.0V

394.0393
(Naphthalen-2-yl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1i): $^1$H NMR (400 MHz, CDCl$_3$):
(Naphthalen-2-yl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1i): $^{13}$C NMR (100 MHz, CDCl$_3$):
(Naphthalen-2-yl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1i): Mass Spectra:
(2-(5-Phenylisoxazol-3-yl)phenyl)(thiophen-2-yl)methanone (1j): $^1$H NMR (400 MHz, CDCl₃):
(2-(5-Phenylisoxazol-3-yl)phenyl)(thiophen-2-yl)methanone (1j): $^{13}$C NMR (100 MHz, CDCl$_3$):
(2-(5-Phenylisoxazol-3-yl)phenyl)(thiophen-2-yl)methanone (1j): Mass Spectra:

![Mass Spectra Image]
(Furan-2-yl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1k): $^1$H NMR (400 MHz, CDCl$_3$):
(Furan-2-yl)(2-(5-phenylisoxazol-3-yl)phenyl)methanone (1k): $^{13}$C NMR (100 MHz, CDCl$_3$):
(Furan-2-yl)(2-(5-phenyloxazol-3-yl)phenyl)methanone (1k): Mass Spectra:

![Mass Spectra Image]

- ESI Scan (4.3-23.6 sec, 21 scans) Frag=175.0V
- 316.0970
- 217.1055

Counts (%) vs. Mass-to-Charge (m/z)
2,2,6,6-Tetramethylpiperidin-1-yl benzoate (A): $^1$H NMR (600 MHz, CDCl$_3$)
2,2,6,6-Tetramethylpiperidin-1-yl benzoate (A): $^{13}$C NMR (150 MHz, CDCl$_3$)

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**SFO1** 150.9279574 MHz

**NDC1** 13C

**P1** 10.50 ussec

**PLM1** 95.00000000 W

**SFO2** 600.1724007 MHz

**NDC2** 1H

**CXP2** 20.000000000 W

**P1W2** 61.7140000 W

**P1W3** 0.3023399999 W

**#2 - Processing parameters**

| ST | 16384 |
| GF | 150.9128415 MHz |
| NDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| FC | 140 |
2-(5-Phenylisoxazol-3-yl)phenyl acetate (1a'): $^1$H NMR (400 MHz, CDCl$_3$):

![NMR spectrum image]
2-(5-Phenylisoxazol-3-yl)phenyl acetate (1a'): $^{13}$C NMR (100 MHz, CDCl$_3$):
2-(5-Phenylisoxazol-3-yl)phenyl acetate (1a'): Mass Spectra:
5-Methyl-2-(5-phenylisoxazol-3-yl)phenyl acetate (2a'): $^1$H NMR (400 MHz, CDCl$_3$):
5-Methyl-2-(5-phenylisoxazol-3-yl)phenyl acetate (2a'): $^{13}$C NMR (100 MHz, CDCl$_3$):
5-Methyl-2-(5-phenylisoxazol-3-yl)phenyl acetate (2a'): Mass Spectra:
5-Methoxy-2-(5-phenylisoxazol-3-yl)phenyl acetate (3a'): $^1$H NMR (400 MHz, CDCl$_3$):
5-Methoxy-2-[(5-phenylisoxazol-3-yl)phenyl acetate (3a'): $^{13}$C NMR (100 MHz, CDCl$_3$):
5-Methoxy-2-(5-phenylisoxazol-3-yl)phenyl acetate (3a'): Mass Spectra:
3-Nitro-2-(5-phenylisoxazol-3-yl)phenyl acetate (4a'): $^1$H NMR (400 MHz, CDCl$_3$):
3-Nitro-2-(5-phenylisoxazol-3-yl)phenyl acetate (4a"): $^{13}$C NMR (100 MHz, CDCl$_3$):
3-Nitro-2-(5-phenylisoxazol-3-yl)phenyl acetate (4a'): Mass Spectra:
2-((5-Phenylisoxazol-3-yl)methyl)phenyl acetate (5a'): $^1$H NMR (400 MHz, CDCl$_3$):
2-((5-Phenylisoxazol-3-yl)methyl)phenyl acetate (5a'): $^{13}$C NMR (100 MHz, CDCl$_3$):
2-((5-Phenylisoxazol-3-yl)methyl)phenyl acetate (5a'): Mass Spectra: