Supporting information

Iron Catalyzed Chlorination of Benzylic Aldehydes and Ketones

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Table of Contents
Catalyst preparation and characterization 2
References 2
Copies of NMR-specta and GC-MS chromatograms 3-60

**Benzylic chloride (1)**
(1-Chloroethyl)benzene (2) 5-6
1-(Chloromethyl)-4-methylbenzene (3) 7-8
1-(Chloromethyl)-4-(1,1-dimethylethyl)benzene (4) 9-10
4-(Chloromethyl)-1,1'-biphenyl (5) 11-12
1-(1-Chloroethyl)-4-(trifluoromethyl)benzene (6) 13-14
1-(1-Chloroethyl)-4-chlorobenzene (7) 15-16
1-(1-Chloroethyl)-4-bromobenzene (8) 17-18
1-(1-Chloroethyl)-4-fluorobenzene (9) 19-20
4-(1-Chloroethyl)benzoic acid ethyl ester (10) 21-22
(1-Chloropropyl)benzene (11) 23-25
(1-Chloropentyl)benzene (12) 26-28
1-(1,4-Dichlorobutyl)-4-fluorobenzene (13) 29-32
(1-Chloro-2,2-dimethylpropyl)-benzene (14) 33-34
1-(Bromomethyl)-4-methylbenzene (15) 35-36
1-(Iodomethyl)-4-methylbenzene (16) 37-38
1-Chloro-1-deuterio-1-phenylethane (17) 39-40
1-(1-Chloroethyl)-3-methyl benzene (18) 41-42
3-(1-Chloroethyl)-benzonitrile (19) 43-44
1-(1-Chloromethyl)-2-methylbenzene (20) 45-46
1-Chloro-2-(1-chloroethyl)-benzene (21) 47-48
9-Chloro-fluorene (22) 49-50
2-(1-Chloroethyl)naphthalene (23) 51-52
[Chloro(cyclohexyl)methyl]benzene (24) 53-55
1-(Chloroethyl)-4-chlorobenzene (7) 56-57
(1-Chloro-3-buten-1-yl)benzene (26) 58-60
Catalyst Preparation and Characterization

General considerations. The catalysts employed for substrate scope screening were prepared from iron(II) acetate (Sigma Aldrich, 99.99%) by atmospheric oxidation at room temperature or at 110 °C for 24 and 72 hours in a conventional drying oven yielding tile red powder with iron contents of 31.25%, 34.65% and 37.39% respectively. Details on preparation, yields and characterization of the catalysts by IR, elemental analysis, Mössbauer spectroscopy and ICP are given in earlier work.¹

Verification of consistent reactivity: Chlorination of acetophenone (2 mmol) using 4 mol-% of Fe(III) catalysts in ethyl acetate (1 mL) with 1.5 eq of Me₃SiCl and 1.2 eq of Et₃SiH.

References:

(1) R. Savela, R. Leino, Synthesis 2015, in press.
Copies of Spectra:
Data File: C:\CHEM32\DATA\RMS\CL 2013-06-04 13-14-27\030613000002.D
Sample Name: Purity E-Cl DCM

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Last changed: 2/27/2013 6:06:31 PM by CL
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Method Info: AASC-default

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Use Multiplier & Dilution Factor with ISTDs

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Injection Volume : 1 µL
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Last changed : 7/3/2013 12:43:14 PM by RMS
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Use Multiplier & Dilution Factor with ISTDs

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Dilution: 1.0000

Use Multiplier & Dilution Factor with INTRA

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Injection Date: 16-Oct-13, 10:12:55
Injection Volume: 1 µl

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(modified after loading)

Method Info: AAS-C-default

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Area Percent Report

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Dilution: 1.0000
Use Multiplier & Dilution Factor with ISIDa

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Use Multiplier & Dilution Factor with ISTDs

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Use Multiplier & Dilution Factor with ISIDs

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Injection : 1
Injection Volume : 1 μl

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Analysis Method : C:\CHEM32\1\METHODS\AA-DEFAULT.M
Last changed : 10/3/2013 1:08:17 PM by RMS
Method Info : AgSC-default

Areas Percent Report

Sorted By : Signal
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Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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Inj.: 1
Ind Volume: 1 µl
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Area Percent Report

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Dilution: 1.0000
Use Multiplier & Dilution Factor with ISIDs

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**Area Percent Report**

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**Use Multiplier + Dilution Factor with ISSTDs**

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Inj Volume: 1 μl
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Last changed: 3/27/2013 6:06:31 PM by OL
Analysis Method: C:\CHEN32\1\METHODS\AA-DEFAULT.M
Last changed: 10/22/2013 1:03:52 PM by RMS
(modified after loading)
Method Info: AARc-default

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Dilution: 1.0000
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**Area Percent Report**

Sorted by : Signal  
Multiplier: 1.0000  
Dilution: 1.0000  

Use Multiplier & Dilution Factor with ISIDs

| Peak RetTime Type Width Area [min] [min] [pA]* [pA] %  |
|-------------------|-------------------|-------------------|-------------------|-------------------|
| 1  6.259 SB 0.0289 16.03922 8.56970 1.56219 |
| 2  8.784 SB 0.0232 1007.83917 881.20764 84.15187 |
| 3  11.229 SB 0.0227 2.83626 1.97612 0.27623 |
Data File C:\CHEMS\DATA\RMS\DM 2013-10-17 11-20-26\001F0101.D
Sample Name: 3-CN-A-Cl pu

Acq. Operator : RMS
Acq. Method : C:\CHEMS\DATA\RMS\DM 2013-10-17 11-20-26\AA-DEFAULT.M
Acq. Instrument : instrument 1
Injection Date : 17-Oct-13, 11:21:55
Injection : 1
Inj Volume : 1 ll

Analysis Method : C:\CHEMS\METHODS\AA-DEFAULT.M
Last changed : 10/17/2013 11:33:52 PM by RMS
(modified after loading)
Method Info : AAS-C-default

Area Percent Report

Sorted by : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: FIDL A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime Type</th>
<th>Width</th>
<th>Area [mV]</th>
<th>Height Area</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.818 gs</td>
<td>0.0203</td>
<td>21.36726</td>
<td>21.36726</td>
<td>100.00</td>
</tr>
<tr>
<td>2</td>
<td>11.391 mv</td>
<td>0.0393</td>
<td>2880.62720</td>
<td>2880.62720</td>
<td>97.3113</td>
</tr>
<tr>
<td>3</td>
<td>11.443 vs</td>
<td>0.0233</td>
<td>58.16154</td>
<td>58.16154</td>
<td>41.6221</td>
</tr>
</tbody>
</table>

44
Data File: C:\CHEM32\DATA\RMS\DM 2013-10-10 13-03-32\001F0101.D
Sample Name: O-M-B-Ch purity

Acq. Operator: RMS
Seq. Line: 1
Acq. Instrument: Instrument 1
Location: Vial 1
Injection Date: 10-Oct-13, 11:07:00
Inj: 1
Inj Volume: 1 µl

Acq. Method: C:\CHEM32\DATA\RMS\DM 2013-10-10 13-03-32\AA-DEFAULT.M
Last changed: 2/27/2013 6:06:31 PM by OL
Analysis Method: C:\CHEM32\METHODS\AA-DEFAULT.M
Last changed: 10/10/2013 2:24:18 PM by RMS
(modified after loading)
Method Info: AA3C-default

Area Percent Report

Sorted by: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: FID A,

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>[min]</td>
<td>[min] [pA-s] [pA]</td>
<td>%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>8.011</td>
<td>0.0228</td>
<td>664.2110</td>
<td>462.24857</td>
<td>99.86766</td>
</tr>
<tr>
<td>2</td>
<td>11.230</td>
<td>0.0229</td>
<td>2.88410</td>
<td>1.99194</td>
<td>0.43234</td>
</tr>
</tbody>
</table>

46
Data File C:\CHEM32\1\DATA\RMS\DM 2013-09-05 12-10-52\001F0101.D
Sample Name: 2'-Cl-A-Cl purity

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISIDs

Signal 1: FID: A,

<table>
<thead>
<tr>
<th>Peak</th>
<th>Ret Time</th>
<th>Width</th>
<th>Area [min]</th>
<th>Height [pA]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.414</td>
<td>0.0223</td>
<td>268.82468</td>
<td>189.97830</td>
<td>99.03471</td>
</tr>
<tr>
<td>2</td>
<td>11.120</td>
<td>0.0223</td>
<td>2.62012</td>
<td>1.87518</td>
<td>0.96125</td>
</tr>
</tbody>
</table>
Areas Percent Report

- Sorted By: Signal
- Multiplier: 1.0000
- Dilution: 1.0000
- Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID A,
Data File C:\CHM32\DATA\RM\DM 2013-09-17 12-34-37\001F0101.d
Sample Name: np purity

---

Acq. Operator : RMS
Acq. Instrument : Instrument 1
Location : Vial 1
Injection Date : 17-Sep-11, 12:36:03
Injection : 1
Injection Volume : 1 μl
Acq. Method : C:\CHM32\DATA\RM\DM 2013-09-17 12-34-37\AA-DEFAULT.M
Last changed : 3/27/2013 4:06:31 PM by CL
Analysis Method : C:\CHM32\METHODS\AA-DEFAULT.M
Last changed : 9/17/2013 1:08:44 PM by RMS
/methods/modified after loading/
Method Info : AAS-Default

---

**Area Percent Report**

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISST

Signal 1: FID

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.240</td>
<td>SB</td>
<td>0.0229</td>
<td>2.29018</td>
<td>1.58155</td>
<td>1.42574</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>12.293</td>
<td>SB</td>
<td>0.0380</td>
<td>6.97053</td>
<td>2.80551</td>
<td>4.34960</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>14.270</td>
<td>SB</td>
<td>0.0235</td>
<td>151.03313</td>
<td>100.82555</td>
<td>94.22265</td>
<td></td>
</tr>
</tbody>
</table>

---

52
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000

Use Multiplier & Dilution Factor with ISIDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.413 SB</td>
<td>0.0290</td>
<td>24.441999</td>
<td>12.491589</td>
<td>1.00024</td>
</tr>
<tr>
<td>2</td>
<td>9.414 SB</td>
<td>0.0226</td>
<td>12.699355</td>
<td>8.948727</td>
<td>0.81549</td>
</tr>
<tr>
<td>3</td>
<td>9.795 MV</td>
<td>0.0249</td>
<td>8.15893</td>
<td>5.33662</td>
<td>0.33228</td>
</tr>
<tr>
<td>4</td>
<td>9.896 VB</td>
<td>0.0292</td>
<td>2418.032711</td>
<td>1214.165839</td>
<td>98.15199</td>
</tr>
</tbody>
</table>

Acq. Operator: RMS
Acq. Instrument: Instrument 1
Location: Vial 1
Injection Date: 13-Nov-13, 12:11:45
Inj: 1
Inj Volume: 1 µL
Acq. Method: C:|CHEM32|\DATA\RMS\DM 2013-11-13 12-10-16\001F0101.D
Last changed: 2/27/2013 4:06:31 PM by OL
Analysis Method: C:\CHEM32\\METHODS\AA-DEFAULT.M
Last changed: 11/13/2013 1:10:46 PM by RMS (modified after loading)
Method Info: AAGC-default
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISSTDs

Signal 1: FID1 A,

<table>
<thead>
<tr>
<th>#</th>
<th>Ret Time</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.593</td>
<td>3B</td>
<td>0.0245</td>
<td>10.2339</td>
<td>6.46494</td>
<td>0.63017</td>
</tr>
<tr>
<td>2</td>
<td>9.614</td>
<td>3B</td>
<td>0.0288</td>
<td>15.44029</td>
<td>955.61133</td>
<td>98.18433</td>
</tr>
<tr>
<td>3</td>
<td>9.919</td>
<td>3B</td>
<td>0.0219</td>
<td>3.98725</td>
<td>2.75519</td>
<td>0.24852</td>
</tr>
<tr>
<td>4</td>
<td>10.032</td>
<td>3B</td>
<td>0.0215</td>
<td>2.00694</td>
<td>1.41486</td>
<td>0.12358</td>
</tr>
<tr>
<td>5</td>
<td>20.264</td>
<td>3B</td>
<td>0.0277</td>
<td>9.75792</td>
<td>6.16153</td>
<td>0.22140</td>
</tr>
<tr>
<td>6</td>
<td>20.197</td>
<td>3B</td>
<td>0.0261</td>
<td>9.98768</td>
<td>6.78463</td>
<td>0.61500</td>
</tr>
</tbody>
</table>