Supporting Information


Chikkagundagal K. Mahesha, a Devesh S. Agarwal, a Pidiyara Karishma, a Datta Markad, b Sanjay K. Mandal, b and Rajeev Sakhua a*

a Department of Chemistry, Birla Institute of Technology & Science, Pilani, Rajasthan 333031, India
Fax: + 91-1596-244183; Email: sakhua.rajeev@gmail.com; Phone: + 91-1596-245711
b Department of Chemical Sciences, Indian Institute of Science Education and Research Mohali, Sector 81, S.A.S. Nagar, Manauli P.O., Punjab 140306, India

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Contents</th>
<th>Page No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1H NMR and 13C NMR of 3aa-3li, 4aa, 5aa, 6aa, 7aa</td>
<td>2-36</td>
</tr>
<tr>
<td>2.</td>
<td>HRMS Spectra of 3aa-3li, 4aa, 5aa, 6aa, 7aa</td>
<td>37-45</td>
</tr>
<tr>
<td>3.</td>
<td>COSY, HSQC and HMBC of 3aa</td>
<td>46-47</td>
</tr>
<tr>
<td>4.</td>
<td>1H NMR &amp; HRMS of crude complex (B1)</td>
<td>48</td>
</tr>
<tr>
<td>5.</td>
<td>HRMS Analysis of crude reaction mixture at different intervals of time</td>
<td>49</td>
</tr>
<tr>
<td>6.</td>
<td>X-Ray data of 3fc</td>
<td>50</td>
</tr>
</tbody>
</table>

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\( ^1 \)H NMR of 3aa

\[
\begin{array}{c}
\text{N} \\
\text{N} \\
\text{O} \\
\text{C} \\
\text{H}_3 \\
\text{O} \\
\text{C} \\
\text{H}_3 \\
\end{array}
\]

\( ^{13} \)C NMR of 3aa

\[
\begin{array}{c}
\text{N} \\
\text{N} \\
\text{O} \\
\text{C} \\
\text{H}_3 \\
\text{O} \\
\text{C} \\
\text{H}_3 \\
\end{array}
\]
$^1$H NMR of 3ac

$^{13}$C NMR of 3ac
$^1$H NMR of 3ba

$^{13}$C NMR of 3ba
$^1$H NMR of 3bb

$^{13}$C NMR of 3bb
$^1$H NMR of 3ca

$^{13}$C NMR of 3ca
\(^1\)H NMR of 3cb

\[ \text{NMR Spectrum Image} \]

\[ \text{Chemical Formula Image} \]

\(^{13}\)C NMR of 3cb

\[ \text{NMR Spectrum Image} \]

\[ \text{Chemical Formula Image} \]
$^1$H NMR of 3cc

$^{13}$C NMR of 3cc
$^1$H NMR of 3da

$^{13}$C NMR of 3da
$^1$H NMR of 3db

![1H NMR spectrum of 3db](image)

$^{13}$C NMR of 3db

![13C NMR spectrum of 3db](image)
$^1$H NMR of 3dc

$^{13}$C NMR of 3dc
$^{13}$C NMR of 3eb

$^{19}$F NMR of 3eb
$^1$H NMR of 3fa

$^{13}$C NMR of 3fa
$^{1}\text{H NMR of 3fb}$

$^{13}\text{C NMR of 3fb}$
$^1$H NMR of 3ga

$^{13}$C NMR of 3ga
$^1$H NMR of 3hb

$^{13}$C NMR of 3hb
\(^1\)H NMR of 3ib

\[^{13}\text{C}\] NMR of 3ib
1H NMR of 3ja

13C NMR of 3ja
$^1$H NMR of 3ad

$^{13}$C NMR of 3ad
$^1$H NMR of 3ae

$^{13}$C NMR of 3ae
\[ ^1H \text{ NMR of 3ai} \]

\[ ^{13}C \text{ NMR of 3ai} \]
$^1$H NMR of 3bi

$^{13}$C NMR of 3bi
\(^1\)H NMR of 3ci

\[^{13}\]C NMR of 3ci
$^1$H NMR of 3la

$^{13}$C NMR of 3la
\(^1\)H NMR of 4aa

\[^{13}\text{C}\] NMR of 4aa

[Chemical Structures and Spectra]
$^{1}H$ NMR of 5aa

$^{13}C$ NMR of 5aa
**$^1$H NMR of 6aa**

![H NMR spectrum of 6aa](image)

**$^{13}$C NMR of 6aa**

![C NMR spectrum of 6aa](image)
HRMS Spectra of 3aa

Cpd 1: 2.465 321.1238: ESI Scan (rt 2.316-2.681 min, 23 scans) Frag=135.0V KMH-95.d Subtract

Counts vs. Mass-to-Charge (m/z)

HRMS Spectra of 3ab

Cpd 1: 0.487 432.2382: ESI Scan (rt 0.453-0.586 min, 9 scans) Frag=135.0V KMH-47.d Subtract

Counts vs. Mass-to-Charge (m/z)

HRMS Spectra of 3ac

Cpd 1: 1.149 383.1485: ESI Scan (rt 1.082-1.232-1.298 min, 6 scans) Frag=135.0V KMH-62.d Subtract

Counts vs. Mass-to-Charge (m/z)

HRMS Spectra of 3ba

Cpd 1: 1.089 335.1394: ESI Scan (rt 1.006-1.023, 1.172-1.288 min, 10 scans) Frag=135.0V KMH-50.d Subtract

Counts vs. Mass-to-Charge (m/z)
HRMS Spectra of 3la

HRMS Spectra of 3li

HRMS Spectra of 4aa

HRMS Spectra of 5aa
HRMS Spectra of 6aa

Cpd 1: 0.178 512.5028: +ESI Scan (rt: 0.145, 0.245-0.311 min, 6 scans) Frag=80.0V KMH-159.d...
COSY spectra of 3aa

HSQC Spectra of 3aa
HMBC spectra of 3aa

Apr21-2018/15
KMH-95

{(4.44, 14.31)}
{(2.81, 113.64)}
{(2.81, 121.75)}
{(2.81, 125.45)}
{(2.81, 139.42)}
{(4.44, 166.48)}
{(2.81, 166.48)}
{(1.42, 61.63)}
{(4.44, 166.45)}
{(2.81, 166.45)}
{(2.81, 14.31)}
$^1$H NMR of Crude Complex (B1)

HRMS Spectra of Crude complex (B1)
HRMS Analysis of crude reaction mixture at different interval of time

After 2 h, of start of reaction $[1a + [\text{Ir(COD)Cl}]_2 + \text{AgSbF}_6]$ at room temperature

After 4 h, of start of reaction $[1a + [\text{Ir(COD)Cl}]_2 + \text{AgSbF}_6 + 2a]$ at room temperature

After 2 h, of start of reaction $[1a + [\text{Ir(COD)Cl}]_2 + \text{AgSbF}_6 + 2a]$ at room temperature
**X-Ray data of 3fc**

Crystals of 3fc were screened under a microscope for mounting in a nylon loop attached to a goniometer head. Initial crystal evaluation and data collection were performed on a Kappa APEX II diffractometer equipped with a CCD detector (with the crystal-to-detector distance fixed at 60 mm) and sealed-tube monochromated MoKα radiation using the program APEX2. By using the program SAINT\(^1\) for the integration of the data, reflection profiles were fitted, and values of F\(^2\) and σ(F\(^2\)) for each reflection were obtained. Data were also corrected for Lorentz and polarization effects. The subroutine XPREP\(^1\) was used for the processing of data that included determination of space group, application of an absorption correction (SADABS)\(^1\), merging of data, and generation of files necessary for solution and refinement. The crystal structure was solved and refined using SHELX 97.\(^2\) In each case, the space group was chosen based on systematic absences and confirmed by the successful refinement of the structure. Positions of most of the non-hydrogen atoms were obtained from a direct methods solution. Several full-matrix least-squares/difference Fourier cycles were performed, locating the remainder of the non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with individual isotropic displacement parameters. All figures were drawn using MERCURY V 3.0\(^3\) and Platon.\(^4\)

Crystal data for 3fc. C\(_{24}\)H\(_{17}\)ClN\(_2\)O\(_3\), \(M_r = 416.84\), \(T = 273(2)\) K, triclinic, space group \(P-1\) (No. 2), \(a = 8.5002(7)\) Å, \(b = 9.3931(8)\) Å, \(c = 13.1098(11)\) Å, \(α = 83.94(2)°\), \(β = 80.207(2)°\), \(γ = 80.262(2)°\), \(V = 1012.10(15)\) Å\(^3\), \(Z = 2\), \(D_c = 1.368\) g cm\(^{-3}\), \(μ = 0.218\) mm\(^{-1}\). \(R_{int} = 0.0283\), final \(R_I = 0.0400\), \(wR_2 = 0.1147\) for 2899 observed reflections [\(I > 2σ(I)\)] and \(R_1 = 0.0509\), \(wR_2 = 0.1316\) for all 3563 reflections; GOF = 1.085. CCDC No. 1841375.

Fully labelled ORTEP of 3fc is shown below:

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**References**