Supporting information for:

Visible Light Photoredox-Catalyzed α-Alkylation of Cyclic Tertiary Arylamines

Ji-Tao Xu†, Guo-Qiang Xu†, Zhu-Yin Wang† and Peng-Fei Xu*†

†State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, P. R. China

*E-mail: xupf@lzu.edu.cn

Contents

1. Screening of Reaction Conditions .................................................................2
2. The Safety Issues for Handling of Azido Compounds ..................................4
3. Reaction Device Diagrams ...........................................................................4
4. Mechanistic Studies ......................................................................................6
5. References ....................................................................................................11
6. X-Ray Crystallographic Data of Product .....................................................12
7. NMR Spectra of New Compounds ..............................................................13
### 1. Screening of Reaction Conditions

Table S1. Optimization of the Reaction Conditions

<table>
<thead>
<tr>
<th>entry</th>
<th>photocatalyst(mol %)</th>
<th>solvent</th>
<th>base (equiv)</th>
<th>time (h)</th>
<th>yield (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>22</td>
</tr>
<tr>
<td>2</td>
<td>Ru(bpy)$_3$Cl$_2$6H$_2$O (1)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>19</td>
</tr>
<tr>
<td>3</td>
<td>Ru(phen)$_3$(PF$_6$)$_2$(1)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>4</td>
<td>Ir(dFCF$_3$ppy)$_2$ (dibpy)PF$_6$(1)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>22</td>
</tr>
<tr>
<td>5</td>
<td>fac-Ir(ppy)$_3$(1)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>6</td>
<td>Ir(ppy)$_2$ (dibbpy)PF$_6$(1)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>7</td>
<td>Aer-Mes'ClO$_4$(4)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>N.R.</td>
</tr>
<tr>
<td>8</td>
<td>4ctIPN(2)</td>
<td>DCM</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>15</td>
</tr>
<tr>
<td>9</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DCE</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>21</td>
</tr>
<tr>
<td>10</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>toluene</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>N.R.</td>
</tr>
<tr>
<td>11</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>MeCN</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>16</td>
</tr>
<tr>
<td>12</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>THF</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>N.R.</td>
</tr>
<tr>
<td>13</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>32</td>
</tr>
<tr>
<td>14</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMA</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>25</td>
</tr>
<tr>
<td>15</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMSO</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>30</td>
</tr>
<tr>
<td>16</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>EtOH</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>28</td>
</tr>
<tr>
<td>17</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>EtOAc</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>18</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>Dioxan</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>19</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>acetone</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>27</td>
</tr>
<tr>
<td>20</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>MeOH</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>19</td>
</tr>
<tr>
<td>20</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>HFIP</td>
<td>KOAc(1.0)</td>
<td>24</td>
<td>trace</td>
</tr>
<tr>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>22</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>NaOAc(1.0)</td>
<td>24</td>
<td>31</td>
</tr>
<tr>
<td>23</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>CsOAc(1.0)</td>
<td>24</td>
<td>23</td>
</tr>
<tr>
<td>24</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>KHCO$_3$(1.0)</td>
<td>24</td>
<td>28</td>
</tr>
<tr>
<td>25</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>K$_2$CO$_3$(1.0)</td>
<td>24</td>
<td>26</td>
</tr>
<tr>
<td>26</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>NH$_4$OAc(1.0)</td>
<td>24</td>
<td>21</td>
</tr>
<tr>
<td>27</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>PhCO$_2$K(1.0)</td>
<td>24</td>
<td>35</td>
</tr>
<tr>
<td>28</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>EtCO$_2$K(1.0)</td>
<td>24</td>
<td>28</td>
</tr>
<tr>
<td>29</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>Et$_3$N (1.0)</td>
<td>24</td>
<td>32</td>
</tr>
<tr>
<td>30</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>Pyridine(1.0)</td>
<td>24</td>
<td>31</td>
</tr>
<tr>
<td>31</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>DBU(1.0)</td>
<td>24</td>
<td>33</td>
</tr>
<tr>
<td>32</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>K$_2$HPO$_4$ (1.0)</td>
<td>24</td>
<td>31</td>
</tr>
<tr>
<td>33</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>DIPEA (1.0)</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>34</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>DABCO (1.0)</td>
<td>24</td>
<td>18</td>
</tr>
<tr>
<td>35</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>2,6-lutidine (1.0)</td>
<td>24</td>
<td>26</td>
</tr>
<tr>
<td>36</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>PhCO$_2$Na (1.0)</td>
<td>24</td>
<td>33</td>
</tr>
<tr>
<td>37</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>i-PrCO$_2$K (1.0)</td>
<td>24</td>
<td>40</td>
</tr>
<tr>
<td>38</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>t-BuCO$_2$K (1.0)</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>39</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (1)</td>
<td>DMF(2 mL)</td>
<td>p-CH$_3$COO-PhCOOK (1.0)</td>
<td>24</td>
<td>23</td>
</tr>
<tr>
<td>40$^b$</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$ (3)</td>
<td>DMF(2 mL)</td>
<td>i-PrCO$_2$K (3.0)</td>
<td>48</td>
<td>50</td>
</tr>
<tr>
<td>41$^c$</td>
<td>Ru(bpy)$_3$(PF$_6$)$_2$</td>
<td>DMF(2 mL)</td>
<td>i-PrCO$_2$K</td>
<td>12</td>
<td>60</td>
</tr>
</tbody>
</table>
Unless otherwise noted, reaction conditions are as follows: 1a (0.2 mmol), 2a (0.2 mmol), photocatalyst (1 mol %), base (1.0 equiv, 0.2 mmol), solvent (2 mL), blue LEDs, 25 °C and under a N₂ atmosphere, isolated yield. ¹ 1a (0.6 mmol), 2a (0.2 mmol), Ru(bpy)_3(PF_6)_2 (3 mol %). ² 1a (0.6 mmol), 2a (0.2 mmol). ³ 1a (0.6 mmol), 2a (0.2 mmol), H₂O (1.0 equiv, 0.2 mmol). ⁴ 1a (0.6 mmol), 2a (0.2 mmol), 0 °C, 420–425 nm. ⁵ 1a (0.6 mmol), 2a (0.2 mmol), –10 °C, 450–455 nm, H₂O (1.0 equiv, 0.2 mmol).

2. The Safety Issues for Handling of Azido Compounds.

Sodium azide (NaN₃) is toxic (LD₅₀ oral = 27 mg/kg for rats) and can be absorbed through skin.

Appropriate gloves are necessary when using it. It is decomposed explosively upon heating to above 275 °C. Sodium azide are relatively safe especially in aqueous solution, unless acidified to form HN₃, which is volatile and highly toxic.

Organic azides are potentially explosive substances that decomposed with the slight input of energy from external sources (heat, light, pressure, etc). When the designed organic azides used for the project, we keep in mind the following equation. It is noted that this equation takes into account all nitrogen atoms in the organic azide, not just those in the azido group.

\[
\frac{N_C + N_O}{N_N} \geq 3 \text{ (N: number of the atom)}
\]

All organic azides are enough stable to be stored under -20 °C at least for 6 months.

3. Reaction Device Diagrams

The reaction was irradiated under a 450–455 nm blue LEDs in Parallel Light Reactor (WP-TEC-1020) designed by WATTCAS, and the material of the reaction tube is quartz. The distance from the light source to the bottom of the reactor tube is about 1 cm (including quartz reactor tube thickness). The power of the light source is 10 watts, and the diameter of hole was 15 mm with intensity of 1916.1 mW·cm⁻². The light source is not filtered.
3.1 General catalysis procedure for the synthesis of product 3

3.2 Procedure for scale-up preparation of 3aa
4. Mechanistic Studies

4.1 Trapping Experiments with TEMPO

As shown in eq 1, in standard conditions, the addition of 2,2,6,6-tetramethyl-1-piperidinylxoxy (TEMPO) into the reaction mixture, the desired product 3aa decreased to 46% and 32% yield sharply. Besides, BHT-trapped the postulated iminyl radical which was generated by α-amino radical reacted with vinyl azides could be successfully detected by HRMS, suggesting the involvement of a radical mechanism which indicated that the reaction might involve a radical process.

![Figure S1. 1-Phenylpyrrolidine 1a and vinyl azide 2a under standard conditions with 2,6-Di-\textit{t}-butyl-4-methylphenol (BHT) (3.0 equiv)](image)

4.2 H$_2^{18}$O isotope labeling experiment:

**General procedure:** A dried quartz tube was charged with the catalyst Ru(bpy)$_3$(PF$_6$)$_2$ (3 mol %, 5.2 mg), the base i-PrCO$_2$K (2.0 equiv, 0.4 mmol, 50.4 mg) and then DMF (2.0 mL) was added via a syringe. Next, 1-Phenylpyrrolidine 1a (3.0 equiv, 0.6 mmol, 88.3 mg), vinyl azide 2a (0.2 mmol, 1.0 equiv, 29.3 mg) and H$_2^{18}$O (0.2 mmol, 3.6 mg, 1.0 equiv) were added via a syringe. The reaction mixture was
degassed by three cycles of freeze-pump-thaw. After the mixture was thoroughly degassed, the reaction was then irradiated under a 450–455 nm blue LEDs in Parallel Light Reactor (WP-TEC-1020). After 12 h, completion of the reaction, water was added and the organic product was extracted with ethyl acetate, the crude mixture was detected by HRMS. HRMS (ESI): [M+H]+ calcd for [C18H20N18O]: 268.1582, found: 268.1578.

4.3 Stern-Volmer Quenching Experiments

Stern-Volmer fluorescence quenching experiments were run with freshly prepared solutions of 10⁻⁵ M Ru(bpy)₃(PF₆)₂ in DMF at room temperature. The solutions were irradiated at 452 nm and fluorescence was measured from 500 nm to 800 nm (emission maximum is at 601 nm). The concentration of Ru(bpy)₃(PF₆)₂ stock solution was 10⁻⁵ M in DMF. The concentrations of the quencher stock solution were 3 mM, 5 mM, 7 mM, 10 mM, 15 mM and 25 mM in DMF, respectively. After being stirred with a thin glass rod, the emission spectrum was collected. Linear regression of I/I₀ against concentration is done in Origin 9.0.

![Fluorescence quenching data with Ru(bpy)₃(PF₆)₂ and variable N-Ph pyrrolidine 1a](image)

**Figure S2.** Fluorescence quenching data with Ru(bpy)₃(PF₆)₂ and variable N-Ph pyrrolidine 1a
Figure S3. Stern-Volmer plot of Ru(bpy)$_3$(PF$_6$)$_2$ and variable N-Ph pyrrolidine (the intensity data was collected at 601 nm).

Figure S4. Fluorescence quenching data with Ru(bpy)$_3$(PF$_6$)$_2$ and variable vinyl azides.
Figure S5. Stern-Volmer plot of Ru(bpy)$_3$(PF$_6$)$_2$ and variable vinyl azides (the intensity data was collected at 601 nm).

4.4 Measurement of quantum yield

According to the procedure of Yoon,$^2$ the photon flux of the LED was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (2.21 g) in 30 mL of a 0.05 M H$_2$SO$_4$ solution. A buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (50 mg) and sodium acetate (11.25 g) in 50 mL of a 0.5 M solution H$_2$SO$_4$. Both solutions were stored in the dark. To determine the photon flux of the LEDs, the ferrioxalate solution (2.0 mL) was placed in a cuvette and irradiated for 90 s at $\lambda_{\text{max}} = 420$ nm. After irradiation, the phenanthroline solution (0.35 mL) was added to the cuvette and the mixture was allowed to stir in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A nonirradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated using eq. 1.

$$\text{mol Fe}^{2+} = \frac{V \cdot \Delta A}{l \cdot \varepsilon}$$  \hspace{1cm} (1)

where $V$ is the total volume (0.00235 L) of the solution after addition of phenanthroline, $\Delta A$ is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, $l$ is the path length (1.00 cm), and $\varepsilon$ is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 L mol$^{-1}$ cm$^{-1}$).
\[
\text{photo flux} = \frac{\text{mol Fe}^{2+}}{\Phi \cdot t \cdot f} 
\]  

(2)

where \(\Phi\) is the quantum yield for the ferrioxalate actinometer (1.12 at \(\lambda_{\text{ex}} = 420\) nm), \(t\) is the irradiation time (90 s), and \(f\) is the fraction of light absorbed at \(\lambda_{\text{ex}} = 420\) nm by the ferrioxalate actinometer. This value is calculated using eq. 3 where \(A\) (420 nm) is the absorbance of the ferrioxalate solution at 420 nm.

\[
f = 1 - 10^{-A(420\text{ nm})}
\]

(3)

\[
\text{mol Fe}^{2+} = \frac{V \cdot \Delta A}{1 \cdot \varepsilon} = \frac{0.00235 \text{ L} \cdot 0.763}{1 \text{ cm} \cdot 11100 \text{ L mol}^{-1} \text{ cm}^{-1}} = 1.62 \times 10^{-7} \text{ mol}
\]

\[
\text{photo flux} = \frac{\text{mol Fe}^{2+}}{\Phi \cdot t \cdot f} = \frac{1.62 \times 10^{-7}}{1.12 \cdot 90 \cdot 1} = 1.61 \times 10^{-9} \text{ einstein} \cdot \text{s}^{-1}
\]

Determination of the reaction quantum yield at 420 nm and quantum yield measurement was performed in an oven-dried 20 mL quartz vial with a magnetic stirring bar. The catalyst Ru(bpy)$_3$(PF$_6$)$_2$ (3 mol %, 5.2 mg), the Base i-PrCO$_2$K (2.0 equiv, 0.4 mmol, 50.4 mg) and then DMF (2.0 mL) was added via a syringe. Next, 1-Phenylpyrrolidine 1a (3.0 equiv, 0.6 mmol, 88.3 mg), vinyl azide 2a (0.2 mmol, 1.0 equiv, 29.3 mg) and H$_2$O (0.2 mmol, 3.6 mg, 1.0 equiv) were added via a syringe. The reaction mixture was degassed by three cycles of freeze-pump-thaw. After the mixture was thoroughly degassed, the reaction was then irradiated under a 420–425 nm blue LEDs in Parallel Light Reactor (WP-TEC-1020) for 18000 s (5.0 h). Then water was added and the organic product was extracted with ethyl acetate. The crude mixture was purified by flash chromatography (silica gel, mixtures of petroleum/ethyl acetate=50:1–20:1), and the crude yield of the product 3aa was determined by $^1$H NMR based on a 1,3,5-trimethoxybenzene (0.1 mmol, 16.8 mg) standard and the final yield was 70% (1.4 x 10$^{-4}$ mol). The reaction quantum yield (\(\Phi\)) was determined using eq. 4, where the photon flux is 1.61 \(\cdot\) 10$^{-9}$ E s$^{-1}$ (determined by actinometry as described above), \(t\) is the reaction time (18000 s) and \(f\) is the fraction of incident light absorbed by the reaction mixture, determined using eq. 3 (\(A >3\) indicating that the fraction of light absorbed is >0.999).

\[
\Phi = \frac{\text{Mole number for product}}{\text{Mole number for absorption of photons}}
\]
\[ \phi = \frac{\text{Mol product}}{\text{flux} \times \text{t} \times f} = \frac{\text{Mol product}}{\text{flux} \times \text{t} \times f} = 4.83 \quad (\text{eq.4}) \]

We calculated the quantum yield of the model reaction of 1a with 2a to be 4.83. This result shows that the reaction contains radical chain propagation process.

5. Reference:

6. X-Ray Crystallographic Data of Compound 3at. (CCDC: 1909583)

Displacement ellipsoids are drawn at 30% probability level.

Single crystal of products 3at was obtained through slow evaporation of a solution in petroleum ether-dichloromethane at RT.

A suitable crystal was selected and measured on a Bruker APEX-II CCD, Dual, Cu at zero, Eos diffractometer. The crystal was kept at 153(2) K during data collection. Using Bruker SHELXTL, the structure was solved with the SHELXS-97 structure solution program using Direct Methods and refined with the SHELXL-2014, refinement package using Least Squares minimisation.

<table>
<thead>
<tr>
<th>Bond precision:</th>
<th>C-C = 0.0023 Å</th>
<th>Wavelength=1.54178</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell: a=9.0638(3) b=5.6389(2) c=28.3440(8)</td>
<td>alpha=90 beta=91.615(1) gamma=90</td>
<td></td>
</tr>
<tr>
<td>Temperature: 153 K</td>
<td>Calculated</td>
<td>Reported</td>
</tr>
<tr>
<td>Volume</td>
<td>1448.08(8)</td>
<td>1448.08(8)</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/n</td>
<td>P 21/n</td>
</tr>
<tr>
<td>Hall group</td>
<td>-P 2yn</td>
<td>-P 2yn</td>
</tr>
<tr>
<td>Moiety formula</td>
<td>C18 H18 F N O</td>
<td>?</td>
</tr>
<tr>
<td>Sum formula</td>
<td>C18 H18 F N O</td>
<td>C18 H18 F N O</td>
</tr>
<tr>
<td>Mr</td>
<td>283.33</td>
<td>283.33</td>
</tr>
<tr>
<td>Dx,g cm-3</td>
<td>1.300</td>
<td>1.300</td>
</tr>
<tr>
<td>Z</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Mu (mm-1)</td>
<td>0.720</td>
<td>0.720</td>
</tr>
<tr>
<td>F000</td>
<td>600.0</td>
<td>600.0</td>
</tr>
<tr>
<td>F000'</td>
<td>601.83</td>
<td></td>
</tr>
<tr>
<td>h,k,lmax</td>
<td>10,6,33</td>
<td>10,6,33</td>
</tr>
<tr>
<td>Nref</td>
<td>2564</td>
<td>2557</td>
</tr>
<tr>
<td>Tmin,Tmax</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tmin'</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Correction method= Not given</td>
<td>Theta(max)= 66.612</td>
<td></td>
</tr>
<tr>
<td>Data completeness= 0.997</td>
<td>wR2(reflections)= 0.1263( 2557)</td>
<td></td>
</tr>
<tr>
<td>R(reflections)= 0.0441( 2311)</td>
<td>Npar= 190</td>
<td></td>
</tr>
<tr>
<td>S = 1.046</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
7. NMR Spectra of New Compounds

$^1$H NMR of 3aa in CDCl$_3$

$^{13}$C NMR of 3aa in CDCl$_3$
$^1$H NMR of 3ab in CDCl$_3$

$^{13}$C NMR of 3ab in CDCl$_3$
$^1$H NMR of 3ac in CDCl$_3$

$^{13}$C NMR of 3ac in CDCl$_3$
$^1$H NMR of 3ad in CDCl$_3$

$^{13}$C NMR of 3ad in CDCl$_3$
$^1$H NMR of 3ae in CDCl$_3$

$^{13}$C NMR of 3ae in CDCl$_3$
$^1$H NMR of 3af in CDCl$_3$

$^{13}$C NMR of 3af in CDCl$_3$
$^1$H NMR of 3ag in CDCl$_3$

$^{13}$C NMR of 3ag in CDCl$_3$
$^1$H NMR of 3ah in CDCl$_3$

$^{13}$C NMR of 3ah in CDCl$_3$
$^1$H NMR of 3ai in CDCl$_3$

$^{13}$C NMR of 3ai in CDCl$_3$
$^1$H NMR of 3aj in CDCl$_3$

$^{13}$C NMR of 3aj in CDCl$_3$
$^1$H NMR of 3ak in CDCl$_3$

$^{13}$C NMR of 3ak in CDCl$_3$
$^1$H NMR of 3a$^1$ in CDCl$_3$

$^{13}$C NMR of 3a$^1$ in CDCl$_3$
$^1$H NMR of 3am in CDCl$_3$

$^{13}$C NMR of 3am in CDCl$_3$
$^1$H NMR of 3an in CDCl$_3$

$^{13}$C NMR of 3an in CDCl$_3$
$^1$H NMR of 3ao in CDCl$_3$

$^{13}$C NMR of 3ao in CDCl$_3$
$^1\text{H NMR of 3a}$ in CDCl$_3$

$^{13}\text{C NMR of 3a}$ in CDCl$_3$
$^1$H NMR of 3aq (mixture of diastereoisomers) in CDCl₃

$^{13}$C NMR of 3aq (mixture of diastereoisomers) in CDCl₃
$^1$H NMR of 3ar in CDCl$_3$

$^{13}$C NMR of 3ar in CDCl$_3$
$^1$H NMR of 3as in CDCl$_3$

$^{13}$C NMR of 3as in CDCl$_3$
$^{1}$H NMR of 3at in CDCl$_3$

$^{13}$C NMR of 3at in CDCl$_3$
$^1$H NMR of 3au in CDCl$_3$

$^{13}$C NMR of 3au in CDCl$_3$
$^1$H NMR of 3av in CDCl$_3$

$^{13}$C NMR of 3av in CDCl$_3$
$^{1}$H NMR of 3aw in CDCl$_3$

$^{13}$C NMR of 3aw in CDCl$_3$
$^1$H NMR of 3ax in CDCl$_3$

$^{13}$C NMR of 3ax in CDCl$_3$
$^{1}$H NMR of 3ay in CDCl$_3$

$^{13}$C NMR of 3ay in CDCl$_3$
$^1$H NMR of 3az in CDCl$_3$

$^{13}$C NMR of 3az in CDCl$_3$
$^1$H NMR of 3ba in CDCl$_3$

$^{13}$C NMR of 3ba in CDCl$_3$
$^{1}H$ NMR of 3bb in CDCl$_3$

$^{13}C$ NMR of 3bb in CDCl$_3$
$^1$H NMR of 3bc in CDCl$_3$

$^{13}$C NMR of 3bc in CDCl$_3$
$^1$H NMR of 3bd in CDCl$_3$

$^{13}$C NMR of 3bd in CDCl$_3$
$^1$H NMR of 3be in CDCl$_3$

$^{13}$C NMR of 3be in CDCl$_3$
$^1$H NMR of 3bf in CDCl$_3$

$^{13}$C NMR of 3bf in CDCl$_3$
$^1$H NMR of 3bg in CDCl$_3$

$^{13}$C NMR of 3bg in CDCl$_3$
$^1$H NMR of 4 in CDCl$_3$

$^{13}$C NMR of 4 in CDCl$_3$
$^1$H NMR of 5 in CDCl$_3$

$^{13}$C NMR of 5 in CDCl$_3$
$^1$H NMR of 6 in CDCl$_3$

[Image of $^1$H NMR spectrum]

$^{13}$C NMR of 6 in CDCl$_3$

[Image of $^{13}$C NMR spectrum]
$^1$H NMR of 7 in CDCl$_3$

$^{13}$C NMR of 7 in CDCl$_3$
$^1$H NMR of 8 in CDCl$_3$

$^{13}$C NMR of 8 in CDCl$_3$