# Supplementary Information

## Facile cyclization in the synthesis of highly fused diaza cyclooctanoid compounds using retrievable nano magnetite-supported sulfonic acid catalyst

Sudipta Pathak, a Kamalesh Debnath, a Md. Masud Rahaman Mollick b and Animesh Pramanik* a

aDepartment of Chemistry and bDepartment of Polymer Science & Technology, University of Calcutta, 92, A. P. C. Road, Kolkata-700 009, India; Fax: +91-33-2351-9755; Tel: +91-33-2484-1647.

E-mail: animesh_in2001@yahoo.co.in

## Content

<table>
<thead>
<tr>
<th>Content</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Preparation of Fe₃O₄ nano particle</td>
<td>S1</td>
</tr>
<tr>
<td>2. Preparation of Fe₃O₄-OSO₃H catalyst</td>
<td>S2</td>
</tr>
<tr>
<td>3. IR spectrum of Fe₃O₄ and Fe₃O₄-OSO₃H</td>
<td>S2</td>
</tr>
<tr>
<td>4. ¹H NMR and ¹³C NMR spectra</td>
<td>S3-S23</td>
</tr>
</tbody>
</table>

### Preparation of Fe₃O₄ nano particle:

FeCl₃·6H₂O (8.1 g) and urea (5.4 g) were stirred in 300 mL double distilled water at 85 to 90 °C for 2 h. The resultant brown colored reaction mixture was cooled to room temperature and FeSO₄·7H₂O (4.2 g) was added into it. The pH of the resultant solution was maintained at 10 by addition of the 0.1 M NaOH. The molar ratio of Fe(III) to Fe(II) in the above system was nearly 2.00. The obtained hydroxides were kept under ultrasonication in the sealed flask at room temperature for 30 min. After ageing for 5 h, the
obtained black powder (Fe$_3$O$_4$) was washed several times with distilled water, and dried under vacuum.

**Preparation of Fe$_3$O$_4$-OSO$_3$H catalyst:** In a two neck round bottom flask, one neck was equipped with a dropping funnel and other neck is fixed with water vacuum to suck HCl gas generated during the reaction progress. Magnetite (Fe$_3$O$_4$) (3 g) was then poured into round bottom flask and neat chlorosulfonic acid was added (1.0 mL) drop by drop over a period of 10 min at room temperature. HCl gas generated immediately from the reaction flask. After complete addition of chlorosulfonic acid, the mixture was stirred vigorously for 30 min and solid brown magnetic sulfonic acid (Nanocat-Fe-OSO$_3$H) (3.45 g) was collected.

![Fig. S1: IR spectrum of Fe$_3$O$_4$ and Fe$_3$O$_4$-OSO$_3$H](image-url)
1H NMR and 13C NMR Spectra

$^{1}H$ NMR of Compound (±)-$2a/2a'$ in $D_6$-DMSO

$^{13}C$ NMR of Compound (±)-$2a/2a'$ in $D_6$-DMSO
DEPT-135 of Compound (±)-2a/2a’ in D₆-DMSO

¹H NMR of Compound (±)-2b/2b’ in D₆-DMSO
$^{13}$C NMR of Compound (±)-2b/2b' in D$_6$-DMSO

$^1$H NMR of Compound (±)-2c/2c' in D$_6$-DMSO
$^{13}$C NMR of Compound (±)-2c/2c' in D$_6$-DMSO

$^1$H NMR of Compound (±)-2d/2d' in D$_6$-DMSO
$^{13}$C NMR of Compound (±)-2d/2d′ in D$_{6}$-DMSO

$^1$H NMR of Compound (±)-2e/2e′ in D$_{6}$-DMSO
\[ ^{13}\text{C NMR of Compound (±)-2e/2e'} \text{ in D}_6\text{-DMSO} \]

\[ ^{1}\text{H NMR of Compound (±)-2f/2f'} \text{ in D}_6\text{-DMSO} \]
$^{13}$C NMR of Compound (±)-2f/2f' in D$_6$-DMSO

DEPT-135 of Compound (±)-2f/2f' in D$_6$-DMSO
$^1$H NMR of Compound (±)-2g/2g’ in D$_6$-DMSO

$^{13}$C NMR of Compound (±)-2g/2g’ in D$_6$-DMSO
$^1$H NMR of Compound (±)-2h/2h' in D$_6$-DMSO

$^{13}$C NMR of Compound (±)-2h/2h' in D$_6$-DMSO
$^{1}\text{H NMR of Compound (±)-2i/2i'}$ in $D_6$-DMSO

$^{13}\text{C NMR of Compound (±)-2i/2i'}$ in $D_6$-DMSO
$^1$H NMR of Compound (±)-2j/2j’ in D$_6$-DMSO

$^{13}$C NMR of Compound (±)-2j/2j’ in D$_6$-DMSO
$^{1}H$ NMR of Compound ($\pm$)-2k/2k’ in $D_6$-DMSO

$^{13}C$ NMR of Compound ($\pm$)-2k/2k’ in $D_6$-DMSO
$^1$H NMR of Compound (±)-2l/2l' in D$_6$-DMSO

$^{13}$C NMR of Compound (±)-2l/2l' in D$_6$-DMSO
DEPT-135 of Compound (±)-21/21' in D$_6$-DMSO

$^1$H NMR of Compound (±)-2m/2m' in D$_6$-DMSO
$^{13}$C NMR of Compound (±)-2m/2m' in D$_6$-DMSO

$^1$H NMR of Compound (±)-2n/2n' in D$_6$-DMSO
$^{12}$C NMR of Compound (±)-2n/2n' in D$_6$-DMSO

$^1$H NMR of Compound (±)-2o/2o' in D$_6$-DMSO
$^{13}$C NMR of Compound (±)-2o/2o′ in D$_6$-DMSO

$^1$H NMR of Compound (±)-2p/2p′ in D$_6$-DMSO
$^{13}$C NMR of Compound (±)-2p/2p' in D$_6$-DMSO

$^1$H NMR of Compound (±)-2q/2q' in D$_6$-DMSO
$^{13}$C NMR of Compound (±)-2q/2q' in D$_6$-DMSO

DEPT-135 of Compound (±)-2q/2q' in D$_6$-DMSO
$^{1}$H NMR of Compound (±)-2r/2r′ in D$_6$-DMSO

$^{13}$C NMR of Compound (±)-2r/2r′ in D$_6$-DMSO
DEPT-135 of Compound (±)-2r/2r′ in D₆-DMSO