Ring Opening of Epoxides and Aziridines with Sodium Azide using Oxone® in Aqueous Acetonitrile: A Highly Regioselective Azidolysis Reaction

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Received 16 March 2002; revised 26 July 2002

Abstract: A wide variety of epoxides and aziridines were converted to the corresponding β-azido alcohols and β-azido amines with sodium azide using Oxone® in aqueous acetonitrile. The reactions were highly regioselective and efficient with excellent yields at room temperature under mild reaction conditions.

Keywords: Oxone®, epoxides, aziridines, azides, regioselectivity

Epoxides2 and aziridines3 are among the most useful synthetic intermediates in organic synthesis. Due to their three membered ring strain, they undergo facile, regio- and stereoselective ring-opening reactions with various nucleophiles, yielding a broad range of valuable products.4,5 Among these, the azidolysis of epoxides and aziridines enjoys a prominent position for the preparation of azido alcohols6 and azido amines,7 respectively. The vicinal azido alcohols are precursors of amino alcohols8 which are well known as β-blockers and also as a structural component in a vast group of natural products.9 Further, they have also been utilized in carbohydrate chemistry10 or in the chemistry of nucleosides.11 The azido amines obtained upon ring opening are easily transformed to valuable vicinal diamines.12 The classical reagents for azidohydrin synthesis are the combined use of TMSN₃ or NaN₃ and a Lewis acid or a transition metal complex.13 In most of epoxide ring-opening reactions with NaN₃ under either alkaline or acidic conditions, suffer from high temperatures or long reaction times. In addition to these, side reactions, isomerizations, epimerization and rearrangements have also been observed by the alkaline conditions of the reaction. It has been found that NaN₃ impregnated on a calcium cation exchange Y-type Zeolite induces the nucleophilic ring opening of epoxides in protic solvents affording azidohydrins.14 Other reported reagents are tributyltinazide and dibutyltinazide, in DMF.15 Use of phase transfer catalyst16 has also been reported recently for the preparation of azidohydrins. Even though several procedures have appeared for the ring opening of epoxides, a limited number of methods have been available for the ring-opening reactions of aziridines. The most straightforward route to azido amines involves the regioselective ring opening of aziridines with TMSN₃ in the presence of a promoter. Tetrabutylammonium fluoride was found to promote the ring-opening reaction of aziridines with TMSN₃.12 Yeung and co-workers17 reported the chromium complex mediated ring-opening reaction of aziridines with TMSN₃. Transition metal-based complexes18 were also used when N-benzoylaziridines were the substrates, but the rearrangement to oxazolines took place. As part of a programme aimed at the synthesis of epoxides and aziridines and their applications in organic synthesis, we have studied the azidolysis of these small heterocyclic compounds.19

We recently found that potassium peroxymonosulfate, commonly sold as Oxone® (2 KHSO₅, KHSO₄, K₂SO₄) catalyze in an extraordinarily effective way the alcoholysis20 of epoxides and aziridines in aqueous methanol. On this basis, we looked for an analogous catalysis, which could be efficient in the azidolysis of epoxides and aziridines, since Oxone®21 is inexpensive, safe and readily available oxidizing agent. Recently we have also found a very useful method22 for easy azidolysis of epoxides and aziridines with NaN₃ in the presence of CeCl₃. Herein, we wish to report a novel, convenient and highly efficient method for the regioselective azidolysis of epoxides and aziridines with NaN₃ in the presence of Oxone® in aqueous acetonitrile (Schemes 1 and 2).

The reaction of styrene oxide (Table 1, Entry 8) with NaN₃ in aqueous acetonitrile was studied at room temperature in the presence of Oxone®. The reaction was found...
to be completed within 30 minutes and it proceeded in a regiospecific manner, whereby azide ion attacked exclusively at benzylic position as expected. This reaction also produced a very small percentage of the other isomer. The ring opening reactions of 3-phenoxy-1,2-epoxy propanes (Table 1, Entries 9–12), epichlorohydrin and isopropyl glycidyl ether were found to be highly regioselective affording single products resulting from the terminal attack of azide ion. In the case of aliphatic terminal epoxides (Table 1, Entries 2 and 3) the attack appears predominantly at the terminal carbon atom of the epoxide ring. The azido alcohols obtained using cycloalkene oxides (Table 1, Entries 5, 6 and 7) were shown to possess a trans configuration.

Similarly, treatment of styrene N-tosylaziridine (Table 2, Entry 1) with NaN₃ in aqueous acetonitrile in the presence of Oxone® afforded a major isomer resulting by the attack of azide ion at the benzylic position, with a small amount of the other isomer. The reaction was found to be highly efficient and completed within 90 minutes at room temperature giving a overall yield of 98%. As in the case of epoxides, the terminal aliphatic aziridines (Table 2, Entries 8–10) yielded a mixture of products. The formation of the major isomer resulting from the attack of azide ion at terminal carbon atom and the minor isomer obtained by internal attack. These isomers could not be separated using column chromatography. Under similar conditions, cyclic aziridines (Table 2, Entries 5–7) gave trans products and the structures were confirmed by coupling constants in ¹H NMR spectroscopy. The azidolysis reaction of 1-vinylcyclohexyl N-tosylaziridine (Table 2, Entry 4) was found to be highly regioselective and gave only one isomer exclusively by attack of azide ion at the terminal carbon atom of the aziridine ring.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>Regioselective Ring Opening of Epoxides with NaN₃ in the Presence of Oxone®</th>
</tr>
</thead>
<tbody>
<tr>
<td>Entry</td>
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<tr>
<td>1</td>
<td>Cl O</td>
</tr>
<tr>
<td>2</td>
<td>Cl O</td>
</tr>
<tr>
<td>3</td>
<td>Cl O</td>
</tr>
<tr>
<td>4</td>
<td>O O</td>
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<td>O O</td>
</tr>
<tr>
<td>12</td>
<td>O O</td>
</tr>
<tr>
<td>13</td>
<td>O O</td>
</tr>
</tbody>
</table>

The products obtained were characterized by IR, ¹H NMR and mass spectra.

Yield refers to the isolated pure products after column chromatography. Yields in parantheses corresponds to the other regioisomer (determined by their crude NMR spectrum).

<table>
<thead>
<tr>
<th>Table 2</th>
<th>Regioselective Ring Opening of Aziridines with NaN₃ in the Presence of Oxone®</th>
</tr>
</thead>
<tbody>
<tr>
<td>Entry</td>
<td>Aziridine</td>
</tr>
<tr>
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<tr>
<td>2</td>
<td>N Ts</td>
</tr>
<tr>
<td>3</td>
<td>N Ts</td>
</tr>
<tr>
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<tr>
<td>10</td>
<td>N Ts</td>
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</tbody>
</table>

The products obtained were characterized by IR, ¹H NMR and mass spectra.

Yield refers to the isolated pure products after column chromatography. Yields in parantheses corresponds to the other regioisomer (determined by their crude NMR spectrum).
In all the cases, 0.5 equivalents of Oxone® was required for the complete conversion of the epoxides and aziridines into the corresponding azido alcohols and azido amines within a short period of time. Reducing the equivalents of Oxone® resulted in isolation of low yields of the products along with the recovery of starting materials even after prolonged reaction times. Ring opening reactions of epoxides and aziridines with NaN₃ carried out at room temperature in the presence of KHSO₅ and K₂SO₄ did not give ring opened products, instead giving recovery of the starting materials. This clearly demonstrates the necessity of KHSO₅ to promote azidolysis. It was also found that the present reaction is also applicable to the substrates having oxidation sensitive functionalities like alkenes to afford the product in excellent yields without any side products (Table 1, Entry 13). Even though at present the mechanism of the cleavage reaction is not known, it may be attributed to the mild acidic nature of the Oxone® in aqueous acetonitrile which may be coordinated with oxygen or nitrogen and facilitate the nucleophilic ring opening of epoxides and aziridines.

In conclusion, Oxone® proved to be an excellent promoter for highly regioselective ring opening of epoxides and aziridines to prepare azidohydrins and azido amines. The advantages of the present protocol are the use of inexpensive reagent, operational simplicity, easy work-up procedures, high regioselectivity and isolation of pure products. Further the short reaction times and the equal reaction efficiency for both epoxides and aziridines at room temperature may open new entry into the ring opening reactions in the field of synthetic organic chemistry.

Regioselective Opening of Epoxides and Aziridines Giving β-Azido Alcohols and β-Azido Amines; General Procedure
A mixture of epoxide or aziridine (1 mmol) in H₂O–MeCN (1:9, v/v), and Oxone (0.5 mmol) was stirred for 5 min at r.t., then NaN₃ (1 mmol) was added and the reaction mixture was stirred at r.t. for a specified time (see Tables 1 and 2). After completion, as indicated by TLC, the solvents were removed under reduced pressure and the residue was purified by column chromatography (silica gel: Merck, 100–200 mesh) to afford the corresponding pure β-azido alcohol or β-azido amine.

1-Azido-2-chloro-2-propanol (Table 1, Entry 1)
Liquid.
IR (neat): 2100, 3385 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 2.40 (br s, 1 H, CH₂O), 3.28–3.45 (m, 4 H), 3.86 (m, 4 H, 1 CH₂OH).
MS (EI): m/z = 59 (M – CH₂N₃)⁺, 79 (M – CH₂N₃)⁺.

1-Azido-2-butanol (Table 1, Entry 2)
Liquiıldı.
IR (neat): 2090, 3390 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 0.98 (t, J = 7.0 Hz, 3 H, CH₃), 1.40–1.65 (m, 2 H, CH₂), 1.96 (br s, 1 H, OH), 3.15–3.45 (m, 2 H, CH₂N₃), 3.58–3.76 (m, 1 H, CH₂OH).

1-Azido-2-hexanol (Table 1, Entry 3)
Liquid.
IR (neat): 2090, 3380 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 0.80 (t, J = 7.2 Hz, 3 H, CH₃), 1.10–1.60 (m, 6 H, CH₂), 1.85 (br s, 1 H, OH), 3.16–3.30 (m, 2 H, CH₂N₃), 3.55–3.80 (m, 1 H, CH₂OH).
MS (EI): m/z = 87 (M – CH₂N₃)⁺.

1-Azido-2-hexyl-1-ethanol (Table 1, Entry 4)
Liquid.
IR (neat): 2100, 3385 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 1.24 (br s, 1 H, OH), 1.50–2.40 (m, 6 H, CH₂), 3.65–3.75 (q, 1 H, CH₂N₃), 4.08–4.28 (q, 1 H, CH₂OH).
MS (EI): m/z = 99 (M – N₃)⁺.

1-Azido-2-phenyl-1-ethanol (Table 1, Entry 5)
Liquid.
IR (neat): 2105, 3390 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 1.20–1.55 (m, 3 H), 1.60–1.80 (m, 2 H), 1.90–2.30 (m, 3 H), 3.10–3.25 (m, 1 H), 3.30–3.45 (m, 1 H).
MS (EI): m/z = 127 (M – N₃)⁺.

1-Azido-2-phenyl-1-propanol (Table 1, Entry 6)
Liquid.
IR (neat): 2100, 3385 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 1.20–1.55 (m, 3 H), 1.60–1.80 (m, 6 H), 1.90–2.30 (m, 3 H), 3.06–3.20 (m, 1 H), 3.24–3.35 (m, 1 H).
MS (EI): m/z = 127 (M – N₃)⁺.

1-Azido-2-phenyl-1-cyclohexanol (Table 1, Entry 7)
Liquid.
IR (neat): 2095, 3390 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 2.00 (br s, 1 H, OH), 3.75 (d, J = 6.5 Hz, 2 H, CH₂OH), 4.70 (t, J = 6.5 Hz, 1 H, CH₂N₃), 7.20–7.40 (m, 5 H, ArH).
MS (EI): m/z = 162 (M – 1)⁺.

1-Azido-2-phenyl-1-ethanol (Table 1, Entry 8)
Liquid.
IR (neat): 2100, 3380 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 2.55 (br s, 1 H, OH), 3.35–3.50 (dd, J = 7.4, 5.5 Hz, 2 H, CH₂N₃), 4.75–4.96 (m, 1 H, CH₂OH), 7.20–7.40 (m, 5 H, ArH).
MS (EI): m/z = 163 (M – 1)⁺.

1-Azido-3-phenoxy-2-propanol (Table 1, Entry 9)
Liquid.
IR (neat): 2100, 3390 cm⁻¹.
¹H NMR (200 MHz, CDCl₃): δ = 0.98 (t, J = 7.0 Hz, 3 H, CH₃), 1.40–1.65 (m, 2 H, CH₂), 1.96 (br s, 1 H, OH), 3.15–3.45 (m, 2 H, CH₂N₃), 3.58–3.76 (m, 1 H, CH₂OH).
1H NMR (200 MHz, CDCl3): δ = 1.55 (br s, 1 H, OH), 3.70 (d, J = 6.5 Hz, 2 H, CH2N3), 4.20 (d, J = 6.5 Hz, 2 H, OCH3), 4.65 (t, J = 6.5 Hz, 1 H, CHOH), 7.20–7.40 (m, 5 H, ArH).

MS (EI): m/z = 192 (M – 1)+.

1-Azido-3-[3,5-dimethylphenoxy]-2-propanol (Table 1, Entry 10)
Liquid.
IR (neat): 2100, 3390 cm–1.

1H NMR (200 MHz, CDCl3): δ = 1.48 (br s, 1 H, OH), 2.30 (s, 6 H, ArCH3), 3.50 (dd, J = 4.2, 4.6 Hz, 2 H, CH2N3), 4.00 (d, J = 6.5 Hz, 2 H, OCH2), 4.05–4.20 (m, 1 H, OCH2), 6.55 (s, 2 H, ArH), 7.00 (s, 1 H, ArH).

MS (EI): m/z = 220 (M – 1)+.

1-Azido-3-[3,5-dichlorophenoxy]-2-propanol (Table 1, Entry 11)
Liquid.
IR (neat): 2100, 3555 cm–1.

1H NMR (200 MHz, CDCl3): δ = 2.50 (br s, 1 H, OH), 3.55 (t, J = 4.6 Hz, 1 H, CHN), 3.85 (t, J = 6.4 Hz, 1 H, CHN), 4.10 (d, J = 6.5 Hz, 2 H, OCH2), 4.15–4.30 (m, 1 H, CHOH), 6.86 (m, 1 H, ArH), 7.21 (m, 1 H, ArH), 7.38 (m, 1 H, ArH).

MS (EI): m/z = 242 (M – 1)+.

1-Azido-3-[2-naphthoxy]-2-propanol (Table 1, Entry 12)
Liquid.
IR (neat): 2100, 3385 cm–1.

1H NMR (200 MHz, CDCl3): δ = 2.45 (br s, 1 H, OH), 3.39 (dd, 2 H, ArH), 2.9–3.1 (m, 1 H, OH, olefinic), 6.60 (dd, 1 H, ArH), 15.9 Hz, olefinic), 7.22–7.42 (m, 3 H, ArH).

MS (EI): m/z = 261 (M – 1)+.

1-Azido-3-[3-phenyl-(E)-2-propenoyloxy]-2-propanol (Table 1, Entry 13)
Liquid.
IR (neat): 2100, 3390 cm–1.

1H NMR (200 MHz, CDCl3): δ = 2.45 (br s, 1 H, OH), 3.39 (dd, 2 H, ArH), 2.9–3.1 (m, 1 H, OH, olefinic), 6.60 (dd, 1 H, ArH), 15.9 Hz, olefinic), 7.22–7.42 (m, 5 H, ArH).

MS (EI): m/z = 160 (M – 1)+.

1-(2-Azido-2-phenethyl)-4-methyl-1-benzenesulfonamide (Table 2, Entry 1)
Liquid.
IR (neat): 2100, 3270 cm–1.

1H NMR (200 MHz, CDCl3): δ = 2.45 (s, 3 H, ArCH3), 2.95–3.10 (m, 1 H, CHNH), 3.10–3.30 (m, 1 H, CHNH), 4.55–4.65 (dd, J = 7.7, 5.0 Hz, 1 H, CHN), 5.30–5.50 (m, 1 H, NH), 7.05–7.40 (m, 7 H, ArH), 7.75 (d, J = 8.0 Hz, 2 H, ArH).

MS (EI): m/z = 274 (M – N3)+.

1-(2-Azido-1-phenethyl)-4-methyl-1-benzenesulfonamide (Table 2, Entry 2)
Liquid.
IR (neat): 2100, 3370 cm–1.

1H NMR (200 MHz, CDCl3): δ = 2.35 (s, 3 H, ArCH3), 2.45 (s, 3 H, ArCH3), 2.94–3.30 (m, 2 H, CH2N3), 4.50–4.60 (dd, J = 7.8, 5.2 Hz, 1 H, CHNH), 4.90 (m, 1 H, NH), 7.10–7.20 (m, 4 H, ArH), 7.30 (d, J = 8.4 Hz, 2 H, ArH), 7.75 (d, J = 8.4 Hz, 2 H, ArH).

MS (EI): m/z = 288 (M – N3)+.

1-(2-Azido-2-[4-methylphenyl]ethyl)-4-methyl-1-benzenesulfonamide (Table 2, Entry 3)
Liquid.
IR (neat): 2100, 3280 cm–1.

1H NMR (200 MHz, CDCl3): δ = 2.45 (s, 3 H, ArCH3), 3.05–3.15 (m, 1 H, CH), 3.15–3.30 (m, 1 H, CH), 4.55–4.60 (dd, J = 11.9, 5.9 Hz, 1 H, CHNH), 4.90 (m, 1 H, NH), 7.00–7.15 (m, 4 H, ArH), 7.35 (d, J = 8.0 Hz, 2 H, ArH), 7.80 (d, J = 8.0 Hz, 2 H, ArH).

MS (EI): m/z = 308 (M – N3)+.

1-(2-Azido-1-cyclohexylethyl)-4-methyl-1-benzenesulfonamide (Table 2, Entry 4)
Liquid.
IR (neat): 2100, 3380 cm–1.

1H NMR (200 MHz, CDCl3): δ = 0.60–1.80 (m, 1 H, cyclohexyl), 2.40 (s, 3 H, CH3), 3.26 (dd, 1 H, J = 3.2, 9.4 Hz, CH3N), 3.30 (dd, 1 H, J = 3.2, 9.4 Hz, CH3N), 5.20 (m, 1 H, NH), 7.30 (d, J = 7.5 Hz, ArH), 7.80 (d, J = 7.5 Hz, ArH).

MS (EI): m/z = 101 (M – CH2N3)+.

N-(2-Azidocyclohexyl)-4-methylbenzenesulfonamide (Table 2, Entry 5)
Liquid.
IR (neat): 2100, 3320 cm–1.

1H NMR (400 MHz, CDCl3): δ = 1.20–1.40 (m, 4 H), 1.60–1.75 (m, 2 H), 2.00–2.15 (m, 2 H), 2.45 (s, 3 H, ArCH3), 2.85–2.95 (m, 1 H, CHNH), 3.00–3.10 (m, 1 H, CHNH), 4.80 (d, J = 5.4 Hz, 1 H, NH), 7.30 (d, J = 8.1 Hz, 2 H, ArH), 7.80 (d, J = 8.1 Hz, 2 H, ArH).

MS (EI): m/z = 294 (M+).

N-(2-Azidocyclopentyl)-4-methylbenzenesulfonamide (Table 2, Entry 6)
Liquid.
IR (neat): 2105, 3270 cm–1.

1H NMR (400 MHz, CDCl3): δ = 1.30–1.50 (m, 1 H), 1.60–1.75 (m, 3 H), 1.95–2.10 (m, 2 H), 2.45 (s, 3 H, ArCH3), 3.30–3.50 (m, 1 H,
CHN\n\n0.36–3.70 (m, 1 H, CHNH), 4.85 (d, \( J = 6.5 \) Hz, 1 H, NH), 7.30 (d, \( J = 8.0 \) Hz, 2 H, ArH), 7.80 (d, \( J = 8.0 \) Hz, 2 H, ArH).

MS (EI): \( m/z = 281 \) (MH\(^+\)).

N-(2-Azidocyclooctyl)-4-methylbenzenesulfonamide (Table 2, Entry 7)

Liquid.

IR (neat): 2100, 3275 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta = 1.10–1.50 \) (m, 8 H, 2 H), 1.93–2.20 (m, 2 H), 2.40 (s, 3 H, ArCH\(_3\)), 2.90–3.00 (m, 1 H, CHN\(_3\)), 3.05–3.10 (m, 1 H, CHNH), 4.85 (d, \( J = 5.8 \) Hz, 1 H, NH), 7.30 (d, \( J = 8.0 \) Hz, 2 H, ArH), 7.70 (d, \( J = 8.0 \) Hz, 2 H, ArH).

MS (EI): \( m/z = 323 \) (MH\(^+\)).

N-(2-Azidomethylpenty1)-4-methyl-1-benzenesulfonamide (Table 1, Entry 8)

Liquid.

IR (neat): 2095, 3380 cm\(^{-1}\).

\(^1\)H NMR (200 MHz, CDCl\(_3\)): \( \delta = 0.80 \) (t, \( J = 7.0 \) Hz, 3 H, CH\(_3\)), 1.10–1.30 (m, 4 H, CH\(_2\)), 1.35–1.50 (m, 2 H, CH\(_2\)), 2.50 (s, 3 H, ArCH\(_3\)), 3.25–3.40 (m, 3 H, CH\(_3\), CH\(_2\)NH), 4.60 (d, 1 H, \( J = 7.5 \) Hz, NH), 7.35 (d, \( J = 8.2 \) Hz, 2 H, ArH), 7.75 (d, \( J = 8.4 \) Hz, 2 H, ArH).

MS (EI): \( m/z = 240 \) (M – CH\(_2\)N\(_3\)).

N-(2-Azidocycloctyl)-4-methylbenzenesulfonamide (Table 2, Entry 9)

Liquid.

IR (neat): 2105, 3280 cm\(^{-1}\).

\(^1\)H NMR (200 MHz, CDCl\(_3\)): \( \delta = 0.85 \) (t, \( J = 7.1 \) Hz, 3 H, CH\(_3\)), 1.00–1.35 (m, 7 H, CH\(_2\)), 1.35–1.60 (m, 3 H, CH\(_2\)), 2.40 (s, 3 H, ArCH\(_3\)), 3.20–3.40 (m, 3 H, CH\(_2\)NH), 4.65 (d, 1 H, \( J = 7.7 \) Hz, NH), 7.35 (d, \( J = 7.9 \) Hz, 2 H, ArH), 7.80 (d, \( J = 8.1 \) Hz, 2 H, ArH).

MS (EI): \( m/z = 268 \) (M – CH\(_2\)N\(_3\)).

1-N-(1-Azidomethyl)1-n-propyl-4-methyl-1-benzenesulfonamide (Table 2, Entry 10)

Liquid.

IR (neat): 2100, 3390 cm\(^{-1}\).

\(^1\)H NMR (200 MHz, CDCl\(_3\)): \( \delta = 0.85 \) (t, \( J = 7.2 \) Hz, 3 H, CH\(_3\)), 1.05–1.30 (m, 6 H, CH\(_2\)), 1.32–1.60 (m, 2 H, CH\(_2\)), 2.45 (s, 3 H, ArCH\(_3\)), 3.20–3.40 (m, 3 H, CH\(_3\), CH\(_2\)NH), 4.95 (d, 1 H, \( J = 7.6 \) Hz, NH), 7.30 (d, \( J = 8.5 \) Hz, 2 H, ArH), 7.80 (d, \( J = 8.6 \) Hz, 2 H, ArH).

MS (EI): \( m/z = 296 \) (M – CH\(_2\)N\(_3\)).

Acknowledgments

RSB and MSKR thank CSIR New Delhi for the award of fellowships.

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(1) ICT communication No. 03/02/02.