



Asymmetric addition of trimethylsilylcyanide to *N*-benzylimines catalyzed by recyclable chiral dimeric V(V) salen complex

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ABSTRACT

Chiral dimeric vanadium (V) salen complex (10 mol%) derived from 5,5-Methylene di-[(*S,S*)-{*N*-(3-*tert*-butyl salicylidene)-*N'*-(3',5'-di-*tert*-butyl salicylidene)}-1,2-cyclohexanediamine] with vanadyl sulphate followed by auto oxidation was used as efficient catalyst for enantioselective Strecker reaction of *N*-benzylimines with TMSCN at $-30\text{ }^{\circ}\text{C}$. Excellent yield (92%) of α -aminonitrile and high chiral induction was achieved (ee up to 94%) in case of 2-methoxy substituted *N*-benzylimines in 10 h. The catalytic system worked well up to four cycles with retention of enantioselectivity.

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1. Introduction

The catalytic asymmetric Strecker reaction represents simple and efficient method for the synthesis of optically pure α -amino acid derivatives [1], nucleic acids [2], various nitrogen and sulfur containing heterocycles and pharmaceuticals [3]. The classical Strecker reaction reported in 1850 comprises a condensation of an aldehyde, ammonia and a cyanide source, followed by the hydrolysis of the resulting α -amino nitrile [1a]. However, the classical Strecker reaction yields racemic products, which after hydrolysis yield α -amino acids in racemic form [4]. Asymmetric version of this synthetically very useful reaction reportedly used a variety of chiral catalyst such as organocatalysts [5], Jacobsen's Schiff base complexes [6], bi-functional catalyst [7] and metal based catalysts [4,8]. Among these, Jacobsen salen-metal complexes have emerged as an efficient catalyst for the asymmetric Strecker reaction [8a]. Since chiral ligands are expensive, the recycling of chiral catalyst is of immense value. In this context, our group has reported dimeric salen complexes as recyclable chiral catalysts for various asymmetric organic transformations [9]. These dimeric complexes due to relatively high molecular weight are less soluble in solvents like hexane and hence can be easily precipitated out from the reaction mixture in a post-work up step. Previously we have reported dimeric (V) salen complex as an efficient catalyst for the cyanation of aldehydes [9a]. Herein, we are extending the application of

dimeric V(V) salen complex for the asymmetric addition of TMSCN to various *N*-benzylimines.

2. Results and discussion

Chiral vanadium (V) salen complex **1** (Fig. 1) was synthesized by the reaction of ligand 5,5-methylene di-[(*S,S*)-{*N*-(3-*tert*-butyl salicylidene)-*N'*-(3',5'-di-*tert*-butyl salicylidene)}-1,2-cyclohexanediamine] with vanadyl sulfate by the reported method [9a]. Our systemic study for the optimization of the reaction condition for the Strecker reaction (Fig. 2) was initiated by the use of 2-methoxy imine as a model substrate with TMSCN as a source of cyanide using chiral dimeric V(V) salen complex **1** as a recyclable catalyst. As the temperature plays a crucial role to achieve high chiral induction in asymmetric catalysis, we first varied the reaction temperature in a stepwise manner from room temperature (RT) to $-40\text{ }^{\circ}\text{C}$ (Table 1, entries 1–5). On decreasing the temperature from RT to $-30\text{ }^{\circ}\text{C}$ (entries 1–5), the yield of α -aminonitrile remained nearly same, however there was an increase in reaction time and improvement in enantioselectivity. A further decrease in the reaction temperature from -30 to $-40\text{ }^{\circ}\text{C}$ adversely affects the reactivity and enantioselectivity (entry 6). Hence, $-30\text{ }^{\circ}\text{C}$ was taken as optimum reaction temperature for the present protocol for the synthesis of α -aminonitrile. Next, to find out the optimum catalyst loading, we carried out the Strecker reaction with 5, 10 and 20 mol% of the catalyst (Table 1, entries 4, 6, and 7) at $-30\text{ }^{\circ}\text{C}$. In the case of 5 mol% catalyst loading 89% yield with 82% ee was achieved within 10 h. When we increase the catalyst loading from

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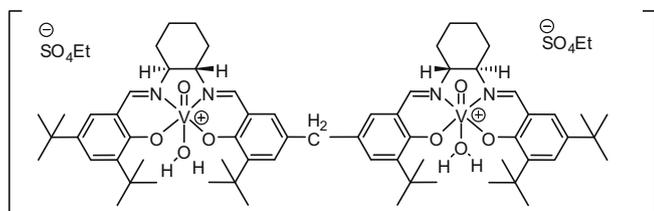
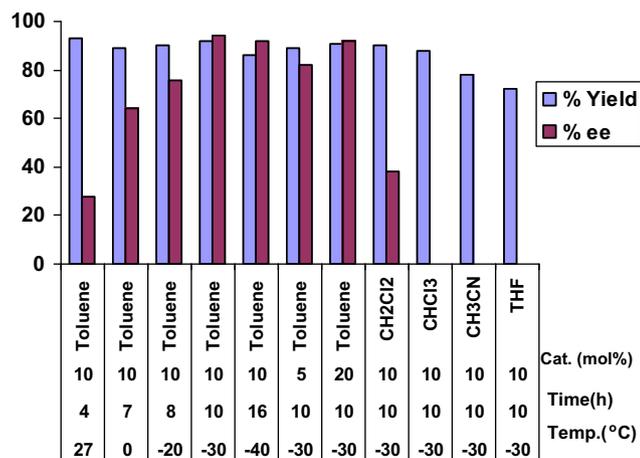
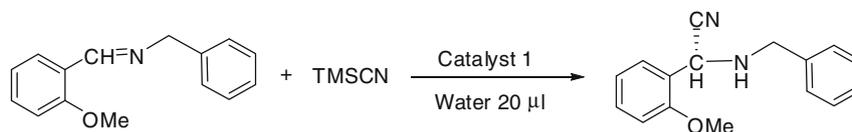


Fig. 1. Structure of catalyst 1.

Fig. 2. Optimization of the reaction conditions for Strecker reaction of *N*-benzylimines with TMSCN.

5 to 10 mol% there was significant improvement in the yield (92%) and ee (94%) of the product, however a further increase in the catalyst loading (20 mol%) was inconsequential. Having optimized the reaction temperature ($-30\text{ }^{\circ}\text{C}$) and catalyst loading (10 mol%), next we optimized reaction medium bearing in mind that the reactivity and enantioselectivity of the Strecker reaction is strongly dependent on the nature of the solvent used [8a]. The solvents e.g., toluene, dichloromethane, chloroform, acetonitrile and THF (Table 1, entries 4, 8–11) were screened for their effect on the yield and ee of the product. A good yield (90%) with moderate ee (38%)

Table 1
Data for the optimization of reaction conditions.^a



Entry	Catalyst loading (mol%)	Temp ($^{\circ}\text{C}$)	Time (h)	Solvent	Yield ^b (%)	Ee ^c (%)
1	10	RT	4	Toluene	93	28
2	10	0	7	Toluene	89	64
3	10	-20	8	Toluene	90	76
4	10	-30	10	Toluene	92	94
5	10	-40	16	Toluene	86	92
6	5	-30	10	Toluene	89	82
7	20	-30	10	Toluene	91	92
8	10	-30	10	CH ₂ Cl ₂	90	38
9	10	-30	10	CHCl ₃	88	Racemic
10	10	-30	10	CH ₃ CN	78	Racemic
11	10	-30	10	THF	72	Racemic

^a All reactions were carried out using 1.5 equiv of TMSCN.

^b Isolated yield.

^c Ee were determined using chiral OD-H column.

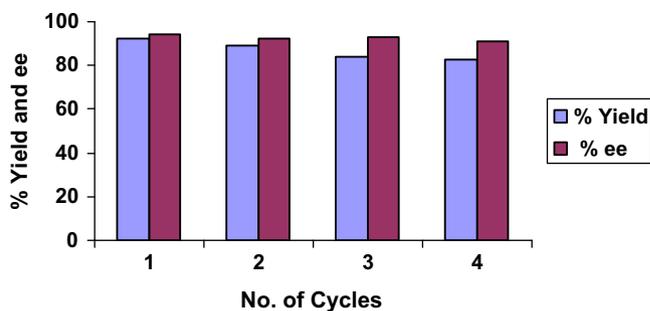
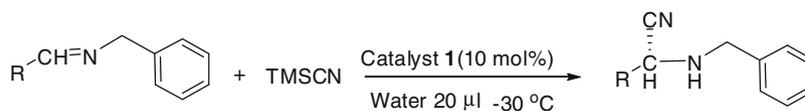


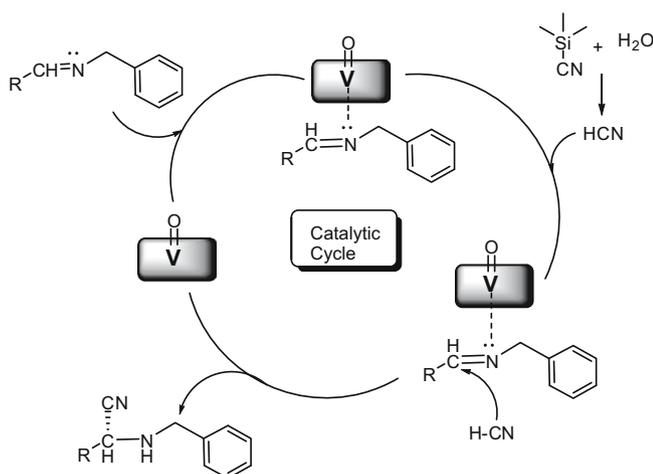
Fig. 3. Recycling of the catalyst 1.

was obtained with dichloromethane. However, on using chloroform, acetonitrile, THF as a solvent although the product yield was 72–88% but the reaction took non chiral path. The best results in terms of yield and ee of the product for the model reaction was achieved with toluene as a solvent (Table 1, entry 4). In all catalytic runs the 1*S*, 2*S* chiral dimeric V(V) salen complex gives the *R*-form of α -aminonitrile.

After optimizing the reaction conditions (Table 1, entry 4) this catalytic protocol was implemented on various *N*-benzylimines in order to see the generality of the catalytic system and the results are summarized in Table 2. Both the electron donating and electron withdrawing substituent present on imine had some effect on the asymmetric Strecker reaction. *N*-Benzylimines with no substituent gave 89% isolated yield with 80% ee. The electron donating ($-\text{Me}$, $-\text{OMe}$) groups at 2- and 4-positions of *N*-benzylimines gave higher yields as well as ee (Table 2, entries 2, 4, 5, 7) than in the cases where these substituents were on 3-position (Table 2, entries 3, 6). Among the methyl and methoxy substituent, the methoxy substituent showed better results in terms of product yield and ee (Table 2, entries 4, 7). The presence of electron withdrawing group on the aromatic ring of imines gave moderate yield and ee (Table 2, entries 8, 9). Furthermore, on conducting the same reaction with *N*-benzylimines derived from aliphatic aldehydes and benzyl amine we could achieve a poor yield (65%) and ee (22%) in the case of crotonaldehyde (Table 2, entry 11) whereas with trimethyl acetaldehyde better yield (78%) and ee (75%) was achieved (Table 2, entry 10).

Table 2Enantioselective addition of TMSCN to various *N*-benzylimines catalyzed by dimeric V(V) salen complex **1**.^a

S. no.	Imine	Yield ^b (%)	Ee ^c (%)
1	C ₆ H ₅ CH=NCH ₂ Ph	89	80
2	4-MeC ₆ H ₄ CH=NCH ₂ Ph	82	83
3	3-MeC ₆ H ₄ CH=NCH ₂ Ph	68	76
4	2-MeC ₆ H ₄ CH=NCH ₂ Ph	75	79
5	4-MeOC ₆ H ₄ CH=NCH ₂ Ph	90	89
6	3-MeOC ₆ H ₄ CH=NCH ₂ Ph	78	60
7	2-MeOC ₆ H ₄ CH=NCH ₂ Ph	92	94
8	4-FC ₆ H ₄ CH=NCH ₂ Ph	88	75
9	4-ClC ₆ H ₄ CH=NCH ₂ Ph	72	62
10	(CH ₃) ₃ CCH=NCH ₂ Ph	78	75
11	CH ₃ CH=CHCH=NCH ₂ Ph	65	22

^a Reaction carried out at -30 °C in toluene using 10 mol% of catalyst **1** using 1.5 equiv of TMSCN.^b Isolated yield.^c Ee were determined using chiral OD and OD-H column.**Scheme 1.** Proposed catalytic cycle for asymmetric Strecker reaction.

Scheme 1 depicted the probable catalytic cycle for the enantioselective Strecker reaction, where the imine was polarized by the weak interaction through the lone pair of electrons present on it with metal center of the catalyst followed by the nucleophilic addition of cyanide ion (HCN was generated by the reaction of H₂O and TMSCN) to the polarized carbon of imines, resulting in the formation of the product and liberated the catalyst to takes part in another catalytic cycle.

Catalyst recycling studies were carried out by precipitating the catalyst by the addition of hexane to the post catalytic reaction mixture. The recovered catalyst was then charged with fresh substrate and reactants and the catalytic run was conducted in the similar manner as in the case of fresh catalyst. The data of four-

Table 3Catalyst recycling data for the enantioselective addition of TMSCN to 2-methoxy substituted *N*-benzylimine.

Run	1	2	3	4
Time (h)	10	10	12	14
Yield	92	89	84	83
Ee	94	92	93	91

time use of the same catalyst is given in **Table 3**. The activity of the recycled catalysts gradually decreased upon successive use possibly due to some physical loss of the catalyst with retention of enantioselectivity (**Fig. 3**).

3. Conclusion

In conclusion chiral dimeric vanadium (V) salen complex was used for the asymmetric addition of TMSCN to *N*-benzylimines. Excellent yield (92%) of α-aminonitrile and high chiral induction was achieved (ee up to 94%) in case of 2-methoxy substituted *N*-benzylimines. Besides, V(V) salen complex turned to be the most efficient recyclable system reported so far in the literature.

4. Experimental

4.1. Materials and methods

NMR spectra were obtained with a Bruker F113 V spectrometer (500 MHz and 125 MHz for ¹H and ¹³C, respectively) and are referenced internally with TMS. FTIR spectra were recorded on Perkin Elmer Spectrum GX spectrophotometer in KBr window. High-resolution mass spectra were obtained with a LC-MS (Q-TOF) LC (Waters), MS (Micromass) instruments. Optical rotations were measured with a Digipol 781 Automatic Polarimeter Rudolph Instruments. Enantiomeric excess (ee) were determined by HPLC (Shimadzu SCL-10AVP) using Daicel Chiralpak OD-H and OD chiral columns with 2-propanol/hexane as eluent. For the product purification flash chromatography was performed using silica gel 100–200 mesh, Vanadyl sulphate penta hydrate (VOSO₄·5H₂O) purchased from s.d. Fine-Chem Limited Mumbai (India). TMSCN, Benzaldehyde, 2-methoxy benzaldehyde, 3-methoxy benzaldehyde, 4-methoxy benzaldehyde, 4-fluoro benzaldehyde, 4-chlorobenzaldehyde, trimethylacetaldehyde, crotonaldehyde (Aldrich Chemicals) 2-methyl benzaldehyde, 3-methyl benzaldehyde, 4-methyl benzaldehyde, benzylamine (Merck Chemicals) and used as received. The recyclable dimeric V(V) salen complex **1** derived from 5,5-Methylene di-[(*S,S*)-*N*-(3-*tert*-butyl salicylidine)-*N'*-(3',5'-di-*tert*-butyl salicylidene)]-1,2-cyclohexanediamine] with vanadyl sulphate followed by auto oxidation was synthesized by our previously reported method [9a]. All the solvents were dried using standard procedures [10], distilled and stored under nitrogen.

4.1.1. Synthesis of complex 1

The complex **1** was synthesized by the reported procedure [9a]. The solution of 5,5-Methylene di-[(*S,S*)-{*N*-(3-*tert*-butyl salicylidene)-*N'*-(3',5'-di-*tert*-butyl salicylidene)]-1,2-cyclohexanediamine] (0.250 g, 0.252 mmol) was dissolved in a mixed solvent system-ethanol:CH₂Cl₂ (3:2, 15 ml) to which an aqueous solution of vanadyl sulphate penta hydrate VOSO₄·5H₂O (0.128 g, 0.504 mmol in 2 ml water) was added drop-wise at room temperature in an inert atmosphere. The resulting solution was refluxed for 4 h and then cooled to room temperature with an extended stirring for 12 h while opening the side arm of the reaction flask for aerial oxidation. Solvent was completely evaporated from the reaction mixture and the residue was dissolved in CH₂Cl₂ (10 ml), washed with water (3 × 5 ml). The organic layer was dried over anhydrous Na₂SO₄, filtered and evaporated to give dark green dimeric V(V) complex.

4.1.2. Typical experimental procedure for the synthesis of *N*-benzylimines [8a]

To a stirred solution of the aldehyde (1 equiv) and anhydrous magnesium sulfate (2 g) in dichloromethane (10 ml), under nitrogen at room temperature, was added benzyl amine (1 equiv). The reaction mixture was stirred for 21 h, then the magnesium sulfate was removed by filtration and the solvent removed in vacuum to give the *N*-benzyl imine as a pale yellow oil or solid. The imines were sufficiently pure for use without further purification.

4.1.3. Typical experimental procedure for addition of TMSCN to *N*-benzylimines

The chiral V(V) dimeric salen complex (10 mg, 0.009 mmol) was dissolved in dry toluene (3 ml) and the solution was cooled to -30 °C under N₂ atmosphere. To the cooled solution *N*-benzylimine (0.09 mmol) was added which was followed by the addition of TMSCN (0.75 equiv) in a drop wise manner over 12 min with stirring. To this stirred solution, H₂O (20 μl) was added and an additional quantity of TMSCN (0.75 equiv) over a period of 30 min. The reaction was monitored on TLC using hexane/ethyl acetate (90/10) as eluent. The product was purified by flash column chromatography on silica gel (eluted with hexane/ethyl acetate = 90:10). The purified products were characterized by ¹H were in agreement with the reported values [8a].

4.1.4. Characterization data

4.1.4.1. *N*-Benzyl (*S*)-2-amino-phenylacetoneitrile (Table 2, entry 1). ¹H NMR (500 MHz, CDCl₃) δ = 1.86 (br s, 1H), 3.95 (d, *J* = 13.0 Hz, 1H), 4.06 (d, *J* = 13.0 Hz, 1H), 4.76 (s, 1H), 7.3–7.6 (m, 10H) HPLC analysis: CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.8 ml/min, *t*_{r1}(minor) = 29.06 min, *t*_{r2}(major) = 31.50 min.

4.1.4.2. *N*-Benzyl (*S*)-2-amino-(4-methylphenyl) acetoneitrile (Table 2, entry 2). ¹H NMR (500 MHz, CDCl₃) δ = 1.83 (1H, br s), 2.36 (3H, s), 3.95 (d, *J* = 13.0 Hz, 1H), 4.06 (d, *J* = 13.0 Hz, 1H), 4.71 (s, 1H), 7.2–7.4 (m, 9H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 26.60 min, *t*_{r2}(major) = 29.05 min.

4.1.4.3. *N*-Benzyl (*S*)-2-amino-(3-methylphenyl) acetoneitrile (Table 2, entry 3). ¹H NMR (500 MHz, CDCl₃) δ = 1.53 (br s, 1H), 2.31 (s, 3H), 3.89 (d, *J* = 13.0 Hz, 1H), 4.01 (d, *J* = 13.0 Hz, 1H), 4.64 (s, 1H), 7.2–7.4 (m, 9H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 25.81 min, *t*_{r2}(major) = 28.28 min.

4.1.4.4. *N*-Benzyl (*S*)-2-amino-(2-methylphenyl) acetoneitrile (Table 2, entry 4). ¹H NMR (500 MHz, CDCl₃) δ = 1.6 (br s, 1H), 2.22 (s, 3H), 3.89 (d, *J* = 13.0 Hz, 1H), 4.04 (d, *J* = 13.0 Hz, 1H), 4.69 (s, 1H),

7.1–7.5 (m, 9H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 24.89 min, *t*_{r2}(major) = 34.50 min.

4.1.4.5. *N*-Benzyl (*S*)-2-amino-(4-methoxyphenyl) acetoneitrile (Table 2, entry 5). ¹H NMR (500 MHz, CDCl₃) δ = 1.62 (br s, 1H), 3.81 (s, 3H), 3.95 (d, *J* = 13.0 Hz, 1H), 4.06 (d, *J* = 13.0 Hz, 1H), 4.69 (s, 1H), 6.92 (d, *J* = 8.5 Hz, 2H), 7.3–7.5 (m, 7H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 37.34 min, *t*_{r2}(major) = 41.19 min.

4.1.4.6. *N*-Benzyl (*S*)-2-amino-(3-methoxyphenyl) acetoneitrile (Table 2, entry 6). ¹H NMR (500 MHz, CDCl₃) δ = 1.84 (br s, 1H), 3.82 (s, 3H), 3.95 (d, *J* = 13.0 Hz, 1H), 4.04 (d, *J* = 13.0 Hz, 1H), 4.72 (s, 1H), 7.3–7.5 (m, 9H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 38.62 min, *t*_{r2}(major) = 42.22 min.

4.1.4.7. *N*-Benzyl (*S*)-2-amino-(2-methoxyphenyl) acetoneitrile (Table 2, entry 7). ¹H NMR (500 MHz, CDCl₃) δ = 2.0 (br s, 1H), 3.77 (s, 3H), 3.86 (d, *J* = 13.0 Hz, 1H), 3.99 (d, *J* = 13.0 Hz, 1H), 4.72 (s, 1H), 6.84 (d, *J* = 8 Hz, 2H) 7.2–7.4 (m, 7H). CHIRALCEL OD-H column, hexane/2-propanol = 99:1, flow rate 0.8 ml/min, *t*_{r1}(minor) = 57.56 min, *t*_{r2}(major) = 59.42 min.

4.1.4.8. *N*-Benzyl (*S*)-2-amino-(4-chlorophenyl) acetoneitrile (Table 2, entry 8). ¹H NMR (500 MHz, CDCl₃) δ = 1.6 (br s, 1H), 3.95 (d, *J* = 13.0 Hz, 1H), 4.05 (d, *J* = 13.0 Hz, 1H), 4.72 (s, 1H), 7.2–7.5 (m, 9H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 30.63 min, *t*_{r2}(major) = 34.77 min.

4.1.4.9. *N*-Benzyl (*S*)-2-amino-(4-fluorophenyl) acetoneitrile (Table 2, entry 9). ¹H NMR (500 MHz, CDCl₃) δ = 1.78 (br s, 1H), 3.88 (d, *J* = 13.0 Hz, 1H), 3.98 (d, *J* = 13.0 Hz, 1H), 4.64 (s, 1H), 6.9–7.4 (m, 9H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 35.26 min, *t*_{r2}(major) = 39.17 min.

4.1.4.10. *N*-Benzyl (*S*)-2-amino-3,3-dimethyl butanonitrile (Table 2, entry 10). ¹H NMR (500 MHz, CDCl₃) δ = 1.18 (s, 9H), 3.03 (s, 1H), 3.88 (d, *J* = 13.0 Hz, 1H), 4.01 (s, 1H), 7.2–7.4 (m, 5H). CHIRALCEL OD column, hexane/2-propanol = 95:5, flow rate 0.5 ml/min, *t*_{r1}(minor) = 34.82 min, *t*_{r2}(major) = 36.38 min.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.01.018.

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