



F. No. 39-762/2010 (SR)

The Under Secretary (FD-III)
University Grants Commission
New Delhi-110002

12 JAN 2011

Sub:- UGC support for the Major Research Project in Physical Sciences, Bio-Sciences, Maths, Medical, Agricultural Sciences and Engineering & Chemistry to University/College Teachers - Project entitled, "Asymmetric reductive amination of carbonyl compounds in ionic liquids"

Sir,

I am to refer to your letter forwarding the application of Dr. Boyina Rupini of your institution for financial assistance under the above scheme and to convey the Commission's approval & sanction an on account grant of Rs. 4,99,800/- (Rupees: Four lakh ninety nine thousand eight hundred only) to the Principal, Sri Venkateswara College, Dhaula Kuan, New Delhi in r/o Major Research Project of Dr. Boyina Rupini, Department of Chemistry for the period of 3 years w.e.f. 1.2.2011 as detailed below:-

S.No.	ITEMS	AMOUNT APPROVED	GRANT RELEASED AS 1st INSTALMENT
A.	Non - Recurring		
1.	Books & Journals	20,000/-	1,70,000/-
2.	Equipment (magnetic stoves, hot plate other minor equipments)	1,50,000/-	
B.	Recurring		
1.	Honorarium to Retd. Teacher @ Rs. 12, 000/- p.m.	nil	3,29,800/-
2.	Project Fellow @ 8, 000/- p.m.	2,88,000/-	
3.	Chemical/ Glassware / Consumable	1,50,000/-	
4.	Hiring Services	20,000/-	
5.	Contingency	50,000/-	
6.	Travel/Field Work	50,000/-	
7.	Overhead Charges @ Rs. 10% approved recurring Grant (Except Travel & Field Work)	50,800/-	
	Total (A + B)	7,78,800/-	4,99,800/-

The acceptance Certificate in prescribed format attached Annexure I may be sent to the undersigned within one month from the issue of the award letter failing which the project may be treated as cancelled.

If the terms & conditions are acceptable, as per guideline which are available on UGC web-site www.ugc.ac.in the Demand Draft/ Cheque being sent may be retained. Otherwise the same may be returned in original to the commission by Registered Post in variably with in 15 days from the receipt of the Demand Draft/Cheque.

Principal Investigators should ensure that the utilization Certificate to the effect that the grant has been utilized for the purpose for which it has been sanctioned shall be furnished to the University Grants Commission in time.

The final report of the project may be submitted in typed form as well as electronically i.e. in floppy/CD

1. The sanctioned amount is debitable to the Major Head 4. (i).a (31) Rs. 3,29,800/- & 4. (i).a (35) Rs. 1,70,000/- and is valid for payment during financial year 2010-11.
2. The amount of the Grant shall be drawn by the Under Secretary (drawing and Disbursing Office), University Grants Commission on the Grants-in-aid Bill and shall be disbursed to and credited to the University/College, Sri Venkateswara College, Dhaula Kuan, New Delhi through Cheque/Demand Draft/ Mail Transfer.
3. The Grants is subject to the adjustment of the basis of Utilization Certificate in the prescribed performa submitted by the University/Colleges/Institution.
4. The University/College shall maintain proper accounts of the expenditure out of the grants which shall be utilized only on approved items of expenditure.
5. The Utilization Certificate of the effect that the grant has been utilized for the purpose for which it has been sanctioned shall be furnished to the University Grants Commission as early as possible after the close of the current financial year.
6. The assets acquired wholly or substantially out of University Grant Commission's grant shall not be disposed or encumbered or utilized for the purposes other than those for which the grant was given, without proper sanction of the University Grants Commission and should, at any time the College/University ceased in function such assets shall revert to the University Grant Commission.
7. A Register of assets acquired wholly or substantially out of the grant shall be maintained by the University/College in the prescribed form.
8. The grantee institution shall ensure the utilization of grant-in-aid for which it is being sanctioned/paid. In case non-utilization/part utilization, the simple interest @ 10% per annum as amended from time to time on unutilized amount from the date of drawl to the date of refund as per provisions contained in General Financial Rules of Govt. of India will be charged.
9. The interest earned by the University/College/Institute on this grants in aid shall be treated as additional grant and may be shown in the Utilization Certificate/Statement of expenditure to be furnished by grantee institution.
10. The University/College/Institute shall follow strictly all the instructions issued by the Government of India from time to time with regard to reservation of posts for Scheduled Castes/Scheduled Tribes/OBC/PH etc.
11. The University/College shall fully implement to Official Language Policy of Union Govt. and comply with the Official Language Act, 1963 and Official Languages (Use for Official purposes of the Union) Rules, 1978 etc.
12. The sanction issues in exercise of the delegation of powers vide Commission Office Order No. 25/92 dated May 01, 1992.
13. An amount of Rs. ——— out the grant of Rs. ——— sanctioned vide letter No. F. 39-762/2010 (SR) dated has been utilized by University/College/Institution for the purpose for which it was sanctioned. Utilization Certificate for Rs. ——— has already been entered at S. No. ——— now we may enter Utilization Certificate for Rs. ——— S. No. ——— and in the U. C. Registrar at page No. ———.
14. It is also certified from the B.C.R. that the funds are available under the scheme. Entered in BCR at S.No. 2078. The above grant is sanctioned against the budget provision of Rs. ——— during the current financial year leaving a balance of Rs. ——— under the head of Account 4. (i).a (31) Rs. 3,29,800/- & 4. (i).a (35) Rs. 1,70,000/-
15. The funds to the extent are available under the Scheme.
16. The University/Institution/College is strictly following the UGC regulations on curbing the menace of ragging in Higher Educational Institutions, 2009.

(Dr. K.C. Pathak)
Joint Secretary

Copy forwarded for information and necessary action for:-

1. The Principal, Sri Venkateswara College, Dhaula Kuan, New Delhi-110021, Acknowledgement for the receipt of DD / Cheque / Mail Transfer for Rs. 4,99,800/- may be sent to the Under Secretary, Finance Division III, UGC,
2. Dr. Boyina Rupini, Principal Investigator, Department of Chemistry Sri Venkateswara College, Dhaula Kuan, New Delhi, 110021
3. office of the Director General of Audit, Central Revenues, A. G. C. R. Building, I. P. Estate, New Delhi.
4. The Registrar, University of Delhi, Delhi

(Kanta Batra)
Deputy Secretary

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
NEW DELHI – 110 002

PROFORMA FOR SUBMISSION OF INFORMATION AT THE TIME OF SENDING THE
FINAL REPORT OF THE WORK DONE ON THE PROJECT

1. NAME AND ADDRESS OF THE PRINCIPAL INVESTIGATOR: Dr.B.Rupini, Reader,
SOA, Indira Gandhi National Open University, Maidan Garhi, New Delhi-68
2. NAME AND ADDRESS OF THE INSTITUTION: IGNOU, Maidan Garhi, New Delhi-68
3. UGC APPROVAL NO. AND DATE: F.No.39-762/2010 (SR) dt: 12th Jan, 2011
4. DATE OF IMPLEMENTATION: March-2011
5. TENURE OF THE PROJECT: Three years
6. TOTAL GRANT ALLOCATED: Rs.7, 78,000
7. TOTAL GRANT RECEIVED: Rs.4, 99,800
8. FINAL EXPENDITURE: Rs. 4,24,211
9. TITLE OF THE PROJECT: "Asymmetric Reductive Amination of carbonyl
compounds in chiral ionic liquids".
10. OBJECTIVES OF THE PROJECT:
 1. *Preparation of chiral ionic liquids by changing either the anion counterpart or by the cation counterpart by the chiral one using the reported methodology.*
 2. *Preparation of chiral amines by asymmetric reductive amination of carbonyl compounds using above chiral ionic liquids.*
11. WHETHER OBJECTIVES WERE ACHIEVED (GIVE DETAILS):

80% of the objectives were achieved, remaining objectives could not accomplished due to inordinate delay in transfer of the project from Sri Venkateswara College to IGNOU by host institution. Synthesis and structural characterization of one set of chiral ionic liquids and amines have been achieved (copy enclosed). Second set of synthesis, structural characterization and biological activity of the synthesized samples are yet to be completed.
12. ACHIEVEMENTS FROM THE PROJECT: *Research Publication and Presented the work in national and international conferences.*
13. SUMMARY OF THE FINDING (IN 500 WORDS): *Copy enclosed at 'A'*

14. CONTRIBUTION TO THE SOCIETY (GIVE DETAILS)

Synthesis of chiral amines by using asymmetric reductive amination of carbonyl compounds is one of the promising synthetic method. So far reported reducing agents like NaBH_3CN , $\text{ZnCl}_2/\text{NaBH}_4$ etc., involve the use of organic solvents are hazardous in nature and pollute the environment.

Keep in view of environmental pollution the present research developed cleaner technological and environmental friendly method for the synthesis of chiral amines without using hazardous solvents as well as reducing agents by adopting green chemistry principles like selection of raw materials, reduction in waste, design of synthesis etc.

Our current study proved that the newly synthesized chiral ionic liquids could be used as green solvents, highly efficient and reusable organocatalyst. The results and observations in this study could also be useful in designing of a new type of aliphatic quaternary ammonium based IL with improved stereoselectivity. The catalytic efficiency of these new ILs can be performed for reductive amination reaction for the synthesis of chiral amines and also extendable to various other reactions (like aldol condensation and mumm reaction) which are yet to be completed.

15. WHETHER ANY PH.D. ENROLLED/PRODUCED: No
OUT OF THE PROJECT

16. NO. OF PUBLICATIONS OUT OF THE PROJECT (PLEASE
ATTACH RE-PRINTS): copies enclosed

1. Asymmetric Reductive Amination of Carbonyl Compounds by using *N,N,N*-tributylpropanaminium based Novel Chiral Ionic Liquid. **Boyina Rupini¹, Sharda Pasricha², Brijesh Rathi²** *International Journal of Organic Chemistry*, 2013, 3, 190-193
<http://dx.doi.org/10.4236/ijoc.2013.33024> Published Online September 2013.
(<http://www.scirp.org/journal/ijoc>) - A₁

2. Poster titled "Synthesis of *N,N,N*-Tributyl Propanaminium based chiral ionic liquid" presented at International conference on New Emerging Trends in Chemistry, IIS University, Jaipur- March-2013. - A₂

3. Paper titled "Benign by design -alternative pathways for pollution prevention" presented in National Conference on Environment and Bio diversity of India. Dec-2012 - A₃


(PRINCIPAL INVESTIGATOR)

Dr B Rupini

Principal Investigator

JGC Margi Research Project

(CO-INVESTIGATOR)

(REGISTRAR/PRINCIPAL)

SUMMARY OF THE FINDINGS

A green and efficient functionalized ionic liquid catalytic system was developed to facilitate the reductive amination reactions through the use of microwave heating technology as well as conventional heating. The structures of synthesized chiral and achiral ionic liquids are shown in Figure 1. We describe here the reductive amination reactions between various aromatic ketones and amines in the functionalized ionic liquids.

Our first investigation focused on the primary objective of the project that is the synthesis of chiral ionic liquids. Two novel chiral ionic liquids were prepared and found to be efficient in promoting the reductive amination of carbonyl compounds with aromatic amines to afford chiral secondary amines. These reactions are highly stereoselective in nature. The first ionic liquid, **S-(+)-2- 3-dihydroxy-N,N,N-tributylpropanaminium chloride** was obtained by the reaction of tributylamine and S-(+)- 3-chloro-1,2-propanediol at 120°C for 6 h. S-(+)-2-3-dihydroxy-N,N,N-tributylpropanaminium chloride ionic liquid is soluble in chloroform, dichloro- methane, acetone and ethanol. It is insoluble in water, diethyl ether and n-hexane. The resulting chiral ionic liquid is very stable at room temperature. The other ionic liquid, **S-(+)-3-(tributylamino)-1-hydroxy-2-propoxyborohydride** was prepared by the reaction of first IL with sodium borohydride in water.

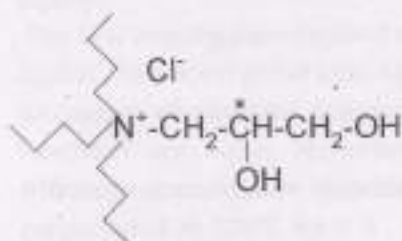
To achieve the second objective a model reaction of asymmetric reductive amination of aniline and 4-fluoro-propeophenone in first ionic liquid was carried out to standardize the reaction procedure. Interestingly, the reaction afforded in 72% yield over a period of 30 minutes. The same reaction was carried out in the presence of second ionic liquid at room temperature for ~30 minutes to secure 60% yield of the same product. In the present investigation, an extremely efficient method has been developed for the synthesis of novel chiral amine, **S-(+)-4-fluoropropeophenyl- N-benzyl-3-(2-amino-4-thiazolyl) coumarin** using one pot, three component reaction using ionic liquids as efficient reusable catalyst. Homogeneity/purity of all the products were assayed by (TLC) on alumina-coated plates (Merck). Compounds were further characterized by IR and NMR. This method has several merits such as high stereo selectivity, cost efficiency, task specific, and simple work up and usage in the synthesis of complex amines. The present investigation reports a novel procedure to synthesize chiral amines using novel chiral ionic liquids which are considerably faster with cleaner technology under neutral conditions. As a testimony to the ILs efficiency, these chiral ionic liquids could be easily recycled and reused for three times. The reductive amination reactions proceeded quickly with good yields and also can be extended successfully to the aldol reactions. The short reaction times and simple reaction conditions coupled with a broad substrate scope render the protocol particularly attractive for the efficient preparation of bioactive and medically interesting molecules.

Our current study proved that the newly synthesized chiral ionic liquids could be used as solvent, highly efficient and reusable organocatalyst for the synthesis of chiral amines in chiral IL by the method of asymmetric reductive amination of carbonyl compounds. The results and observations in this study could also be useful in designing of a new type of aliphatic quarternary ammonium based IL with improved stereo selectivity. These preliminary results are encouraging, however, the catalytic efficiency of these new ILs needs to be performed for various reductive amination reactions which are biologically important.

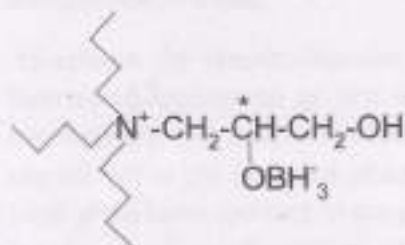
SUMMARY OF THE FINDINGS

Figure-1

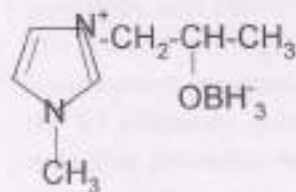
1. S-(+)-2- 3-dihydroxy-N,N,N-tributylpropanaminum chloride

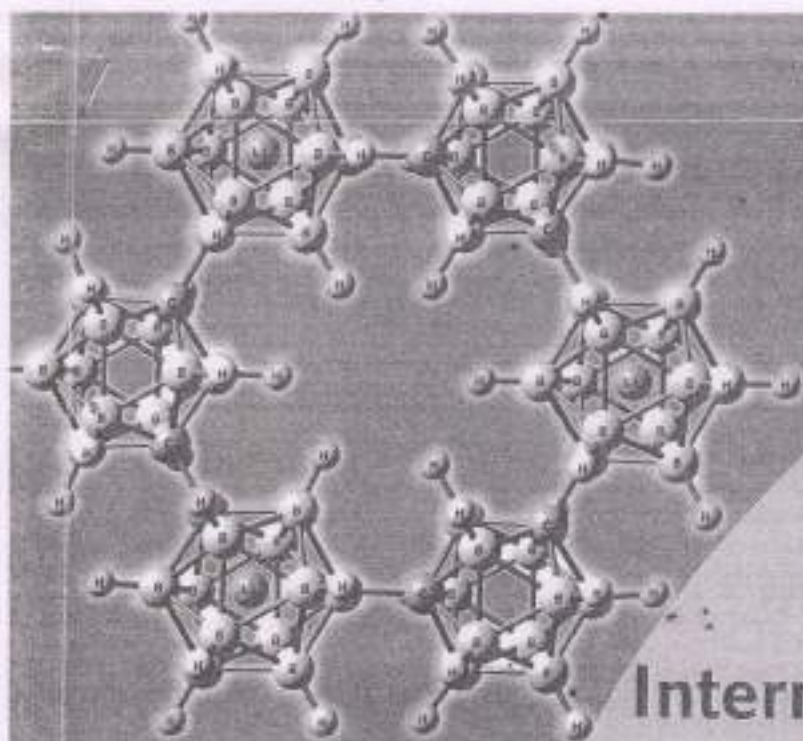


2. S-(+)-3-(tributylamino)-1-hydroxy-2-propoxyborohydride



3.(Hmim)-2-propoxy borohydride





International Conference on New Emerging Trends in Chemistry

A satellite conference of 3rd Indo-German Conference on
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3-4 March 2013

Conference Venue
ISIM Auditorium
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Department of Chemistry
The IIS University, Jaipur



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Synthesis of N,N,N-Tributyl, Propanaminium-Based Chiral Ionic Liquid

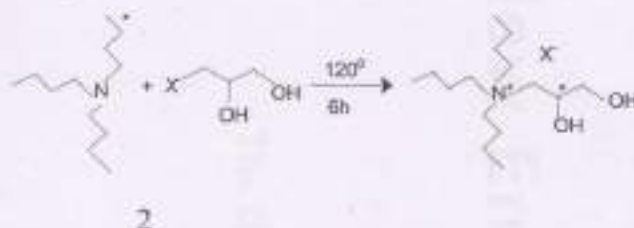
Sharda P¹, Brijesh Rathi¹, Sanjay Jena¹, Rupini B^{2*}

¹S.V. College, University of Delhi, Delhi-110 007 INDIA

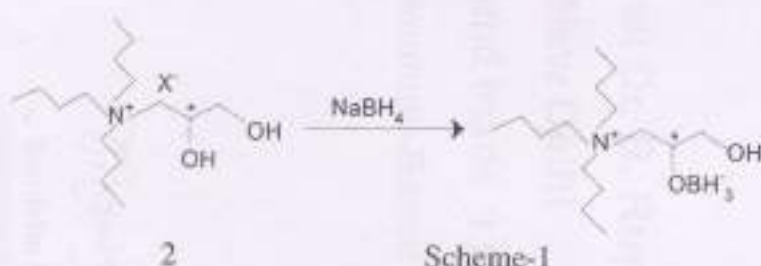
²Reader, School of Agriculture, Indira Gandhi National Open University, New Delhi-110 068 INDIA

E-mail: brupini@ignou.ac.in

In recent years, there has been emphasis on green chemistry, which implies the use of environmental benign chemicals and avoidance of the use of halogenated solvents. In this context, ionic liquids occupy a prominent place in view of their non-toxic nature and the possibility of their recycling. We have undertaken the synthesis of new chiral ionic liquids and their use in asymmetric reductive amination. We wish to report herein the



synthesis, characterization and use of N,N,N-tributylaminium based chiral ionic liquid. The reaction of S-(+)-3-chloro-1,2-propanediol and tributyl amine followed by reduction is the key step in designing a new chiral ionic liquid, which plays a dual role as a solvent and catalyst for reductive amination reaction. 2,3-dihydroxy-N,N,N-tributylpropanaminium chloride (2) was obtained from the reaction of tributyl amine and S-(+)-3-chloro-1,2-propanediol at 120°C for 6 hrs. 3-hydroxy-2-(tributyl amino) propoxy borohydride (scheme 1) was prepared from 2



Scheme-1

by the reaction with NaBH₄ in H₂O at 0-50°C. The resulting chiral IL was purified by silica gel column chromatography eluting with acetone in petroleum ether. The organic layer was dried (Na₂SO₄) and the solvent was evaporated under reduced pressure. After

drying in vacuum the product obtained as a colorless oily liquid. The product was characterized by IR, ¹HNMR, ¹³CNMR and gave satisfactory spectroscopic data in accordance with the structure. In conclusion this synthetic procedure using an easily accessible chiral IL as reagent for the reductive amination provides a novel protocol for the synthesis of chiral amines without requiring any other catalyst and solvent that can be recyclable and green alternative in mild and neutral conditions with significant atom economy.