Synthesis of Axially Chiral (Hetero)-biaryls by Enolization, *O*-Alkylation, and Nucleophilic Aromatic Substitution (S_NAr)

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by

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Department of Chemistry

Submitted

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to the



Indian Institute of Technology Delhi

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This work is dedicated to my

Family.

I am sincerely grateful to my

parents and my brother for their

love, blessings, and continuous

encouragement.

CERTIFICATE

This certifies that the thesis titled "**Synthesis of Axially Chiral (Hetero)-biaryls by Enolization, *O*-Alkylation, and Nucleophilic Aromatic Substitution (S_NAr)**," submitted by **Mr. Harish Kumar Harit** to the **Indian Institute of Technology Delhi** for the **Doctor of Philosophy** degree in Chemistry, constitutes a genuine record of his research work. **Mr. Harish Kumar Harit** has worked under my guidance and supervision, and has met the criteria for the submission of this thesis, which, to my knowledge, has attained the required level of excellence. The findings presented in this dissertation have not been submitted, in whole or in part, to any other university or institution for the conferment of any degree or diploma.

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Harish Kumar Harit

ABSTRACT

The doctoral thesis titled "**Synthesis of Axially Chiral (Hetero)-biaryls by Enolization, O-Alkylation, and Nucleophilic Aromatic Substitution (S_NAr)**" encompasses four chapters focused on the advancement of organocatalytic enantioselective synthesis of quinoline-(1H)-ones. Chapter 1 is introductory and provides a brief explanation of the terminology and importance of asymmetric catalysis. In Chapter 2, the concept of kinetic resolution *via* keto-enol isomerization is elaborated; Chapter 3 involves the phase-transfer-catalyzed synthesis of quinoline-(1*H*)-ones. In chapter 4, the organocatalytic approach to access axially chiral biquinoline-2(1*H*)-ones is described.

Chapter 1: Provides a concise overview of the importance of chirality and its practical aspects with intriguing examples. It covers methods of asymmetric induction, with special emphasis on kinetic resolution (KR), desymmetrization, and organocatalysis, followed by its brief history and modes of activation, including covalent and non-covalent organocatalysis. Then, a brief introduction to atropisomerism, asymmetric isomerization *via* proton transfer, alkylation, and asymmetric aromatic nucleophilic substitution.

Chapter 2: We describe the first asymmetric keto-enol isomerization proceeding *via* proton transfer catalysis, enabled by an unprecedented, reversed proton transfer from the carbonyl to the alcohol group. This protocol exhibits exceptional efficiency, affording axially chiral products in high yields with outstanding enantioselectivities (up to 99:1 er). Mechanistic investigations, including kinetic resolution efficiency analysis, rotational barrier calculation, reaction-time monitoring, and deuterium-shifting experiments, provide compelling evidence for the proposed asymmetric proton-transfer pathway. This innovative strategy not only expands the synthetic toolbox for accessing axially chiral molecules but also underscores the potential of asymmetric proton-transfer processes in achieving challenging stereochemical outcomes. Furthermore, scale-up synthesis and post-synthetic derivatizations highlight the practical synthetic utility of this method.

Chapter 3: In this chapter, we present our efforts in PTC-catalyzed enantioselective synthesis of atropoisomeric biaryls quinolin-2(1*H*)-one by a cation-directed *O*-alkylation. This innovative technique demonstrates the transfer of ions across boundaries in a biphasic reaction

system, serving as an effective pathway for generating atropoisomeric quinolin-2(1*H*)-ones. Impressively, our method delivers these axially chiral motifs in consistently good to high yields and enantioselectivities. A series of axially chiral quinolin-2(1*H*)-one bearing axial chirality has been synthesized successfully with excellent yield (up to 50%) and stereoselectivity (er up to 96:4).

Chapter 4: Here, we have developed the first enantioselective synthesis of axially chiral 4,8'-biquinoline-2(1*H*)-one, guided by nucleophilic aromatic substitution reaction, is showcased. By employing catalytic amount of chiral quinine-derived urea chiral bifunctional organocatalyst, this reaction enables stereocontrolled substitution at electron-deficient aromatic fluorobenzenes to access atropisomeric quinoline-2(1*H*)-ones. The products can be derivatized to a range of atropisomeric 4,8'-biquinoline-2(1*H*)-one without any loss in enantioselectivity, thus offering access to an unexplored axially chiral biquinolinone system.

पीएच.डी. शोधकार्य का सारांश

डॉक्टोरल शोध प्रबंध *"एनोलीकरण, ओ-एल्किलीकरण और न्यूक्लियोफिलिक एरोमैटिक प्रतिस्थापन द्वारा अक्षीय रूप से चिरल (हेटेरो)-बायेरिल्स का संश्लेषण (SNAr)"* इसमें क्विनोलिन-(1H)-ओन्स के ऑर्गेनोकैटेलिटिक एनेंटियोसेलेक्टिव संश्लेषण की उन्नति पर केंद्रित चार अध्याय शामिल हैं।

अध्याय 1: काइरालिटी के महत्व और इसके व्यावहारिक पहलुओं का रोचक उदाहरणों सहित संक्षिप्त अवलोकन प्रस्तुत करता है। इसमें असममित प्रेरण की विधियों को शामिल किया गया है, विशेष रूप से गतिज संकल्प (केआर), विषमरूपीकरण और ऑर्गेनोकैटलिसिस पर जोर दिया गया है। इसके बाद, इसके संक्षिप्त इतिहास और सक्रियण के तरीकों, जिनमें सहसंयोजक और गैर-सहसंयोजक ऑर्गेनोकैटलिसिस शामिल हैं, का वर्णन किया गया है। फिर, एट्रापआइसोमेरिज्म, प्रोटॉन स्थानांतरण द्वारा असममित आइसोमेराइजेशन, एल्किलीकरण और असममित एरोमैटिक न्यूक्लियोफिलिक प्रतिस्थापन का संक्षिप्त परिचय दिया गया है।

अध्याय 2: हम एक अभूतपूर्व, विपरीत प्रोटॉन स्थानांतरण द्वारा संभव बनाए गए प्रोटॉन स्थानांतरण के माध्यम से होने वाले पहले असममित कीटो-एनोल आइसोमेराइज़ेशन का वर्णन करते हैं, जो कार्बोनिल से अल्कोहल समूह में होता है। यह प्रोटोकॉल असाधारण दक्षता प्रदर्शित करता है, जिससे उच्च उपज और उत्कृष्ट एनेंटियोसेलेक्टिविटी (99:1 er तक) के साथ अक्षीय रूप से काइरल उत्पाद प्राप्त होते हैं। क्रियाविधि संबंधी जांच, जिसमें गतिज संकल्पन दक्षता विश्लेषण, घूर्णी अवरोध गणना, अभिक्रिया-समय निगरानी और ड्यूटेरियम-स्थानांतरण प्रयोग शामिल हैं, प्रस्तावित असममित प्रोटॉन-स्थानांतरण मार्ग के लिए ठोस प्रमाण प्रदान करते हैं। यह अभिनव रणनीति न केवल अक्षीय रूप से काइरल अणुओं तक पहुँचने के लिए सिंथेटिक टूलबॉक्स का विस्तार करती है, बल्कि चुनौतीपूर्ण स्टीरियोकेमिकल परिणामों को प्राप्त करने में असममित प्रोटॉन-स्थानांतरण प्रक्रियाओं की क्षमता को भी रेखांकित करती है। इसके अलावा, स्केल-अप संश्लेषण और पोस्ट-सिंथेटिक व्युत्पन्न इस विधि की व्यावहारिक सिंथेटिक उपयोगिता को उजागर करते हैं।

अध्याय 3: इस अध्याय में, हम धनायन-निर्देशित O-एल्किलीकरण द्वारा एट्रापोआइसोमेरिक बायएरिल क्विनोलिन-2(1*H*)-वन के पीटीसी-उत्प्रेरित एनेंटियोसेलेक्टिव संश्लेषण में अपने प्रयासों को प्रस्तुत करते हैं। यह अभिनव तकनीक एक द्विचरणीय अभिक्रिया प्रणाली में सीमाओं के पार आयनों के स्थानांतरण को प्रदर्शित करती है, जो एट्रापोआइसोमेरिक क्विनोलिन-2(1H)-वन उत्पन्न करने के लिए एक प्रभावी मार्ग के रूप में कार्य करती है। उल्लेखनीय रूप से, हमारी विधि इन अक्षीय रूप से काइरल रूपांकनों

को लगातार अच्छी से उच्च उपज और एनेंटियोसेलेक्टिविटी के साथ प्रदान करती है। अक्षीय काइरालिटी युक्त अक्षीय रूप से काइरल क्विनोलिन-2(1H)-वन की एक श्रृंखला को उत्कृष्ट उपज (50% तक) और स्टीरियोसेलेक्टिविटी (er 96:4 तक) के साथ सफलतापूर्वक संश्लेषित किया गया है।

अध्याय 4: यहाँ, हमने नाभिकीय सुगंधित प्रतिस्थापन अभिक्रिया द्वारा निर्देशित अक्षीय रूप से काइरल 4,8'-बाइक्विनोलिन-2(1H)-वन के पहले एनेंटियोसेलेक्टिव संश्लेषण को विकसित किया है। काइरल क्विनिन-व्युत्पन्न यूरिया काइरल बाइफंक्शनल ऑर्गेनोकैटलिस्ट की उत्प्रेरक मात्रा का उपयोग करके, यह अभिक्रिया इलेक्ट्रॉन-कमी वाले सुगंधित फ्लोरोबेंजीन पर स्टीरियोकंट्रोल्ड प्रतिस्थापन को सक्षम बनाती है, जिससे एट्रापिसोमेरिक क्विनोलिन-2(1H)-वन प्राप्त होते हैं। उत्पादों को एनेंटियोसेलेक्टिविटी में बिना किसी हानि के एट्रापिसोमेरिक 4,8'-बाइक्विनोलिन-2(1H)-वन की एक श्रृंखला में व्युत्पन्न किया जा सकता है, इस प्रकार एक अनन्वेषित अक्षीय रूप से काइरल बाइक्विनोलिनोन प्रणाली तक पहुँच प्रदान होती है।

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List of Abbreviations

Abbreviation	Full form
Ac	Acetate
Ar	Aryl
Bn	Benzyl
Boc	*tert*-butyloxycarbonyl
^{t}Bu	*tert*-Butyl
BuLi	Butyllithium
$CDCl_3$	Deuterated chloroform
Cs_2CO_3	Cesium carbonate
CH_2Cl_2	Dichloromethane
CPME	Cyclopentyl methyl ether
CPA	Chiral Phosphoric Acid
CF_3Ph	Trifluoro toluene
CuI	Copper iodide
cm^{-1}	Wavenumbers
°C	Degrees Celsius
dr	Diastereomeric ratio
DET	Diethyl tartrate
DIPEA	*N*, *N*-Diisopropylethylamine
DBU	1,8-Diazabicyclo[5.4.0]undec-7-ene
DMF	Dimethylformamide
DMSO	Dimethylsulfoxide
d	Doublet
dd	Doublet of doublets
d_6-DMSO	Deuterated dimethyl sulfoxide
δ	Chemical shift
er	Enantiomeric ratio
ee	Enantiomeric excess
ESI-TOF	Electrospray ionization - Time-of-flight
EtOAc	Ethyl acetate
Et_2O	Diethyl ether
EtOH	Ethanol

Abbreviation	Full form
FTIR	Fourier transform infrared
H_2O	Water
h	Hour
HOMO	Highest occupied molecular orbital
HPLC	High-performance liquid chromatography
J	Coupling constant
λ	Wavelength
LUMO	Lowest unoccupied molecular orbital
LDA	Lithium diisopropylamide
min	Minute
MTBE	Methyl *tert*-butyl ether
m	Multiplet
MHz	Megahertz
mL	Millilitre
NBS	*N*-Bromosuccinimide
μL	Microlitre
p-TsOH	*p*ara-Toluenesulfonic acid
s	Singlet
SOMO	Singly unoccupied molecular orbital
TFA	Trifluoroacetic acid
TfOH	Triflic acid
TCA	Trichloroacetic acid
THF	Tetrahydrofuran
TMS	Tetramethylsilane
TBAF	Tetra-*N*-butylammonium floride
UV	Ultraviolet