

**STEREOSELECTIVE TOTAL SYNTHESSES OF CYCLOALKENONE
NATURAL PRODUCTS AND CALLYSPONGIDIC ACIDS.**

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**DEPARTMENT OF CHEMISTRY
INDIAN INSTITUTE OF TECHNOLOGY DELHI
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NATURAL PRODUCTS AND CALLYSPONGIDIC ACIDS.**

by

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Submitted

In fulfillment of the requirements of the degree of DOCTOR OF PHILOSOPHY

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

JULY 2021

Dedicated to
My Beloved Family

CERTIFICATE

This is to certify that thesis entitled “**Stereoselective total syntheses of cycloalkenone natural products and callispongic acids.**”, being submitted by **SAYANI DAS** to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of bonafide research work carried out by her. **Ms Sayani Das** has worked under my supervision and guidance and has fulfilled all the requirements for the submission of a Ph.D. thesis, which to my knowledge, has reached the requisite standard and is worthy of consideration for the award of PhD degree.

The work embodied in this thesis has not been submitted, in part or full, to another University or Institute for the award of any degree or diploma.

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ABSTRACT

The thesis titled “**Stereoselective total syntheses of cycloalkenone natural products and callyspongidic acids.**” presents the work carried out on the total synthesis of a chiral cyclohexenone, cyclopentenone bioactive natural products and a family of amphiphilic lipidic tyrosine derivatives named callyspongidic acids.

Chapter 1 describes the stereoselective total syntheses of anti-fungal cyclohexenones bioactive natural products (–)-Pseudohygrophorone A¹² and (–)-Pseudohygrophorone B¹² starting from commercially available inexpensive chiral starting material (–)-quinic acid. The salient features of these syntheses are the diastereoselective addition of dodecyl magnesium bromide to highly oxygenated ketone derived from (–)-quinic acid and α -oxidation of the enone.

Chapter 2 describes the stereoselective total syntheses of anti-fungal cyclopentenones natural products (–)-Hygrophorone A¹², (–)-4-O-Acetyl-hygrophorone A¹² and (+)-Hygrophorone B¹² in high overall yield from D-(–)-tartaric acid. The key step of this syntheses is an aqueous KOH mediated diastereoselective intramolecular aldol reaction to form β -hydroxy ketone with three contiguous chiral centres, which was further elaborated to (–)-hygrophorone A¹² and (+)-hygrophorone B¹².

Chapter 3 describes the first short and expeditious total syntheses of (±)-Callyspongidic acids and 2-*epi*-(±)-Callyspongidic acids from epoxy ester derived from L-tartaric acid. The notable features of these syntheses are the stereoselective opening of epoxide with organocuprates and chemoselective addition of Grignard reagent to ketone in the presence of ester.

सार

थीसिस शीर्षक "साइक्लोएलकेनोन प्राकृतिक उत्पादों और कैलीस्पोन्गिडिक एसिड के स्टीरियोसेलेक्टिव कुल संश्लेषण।" एक चिरल साइक्लोहेक्सेनोन, साइक्लोपेंटेनोन बायोएक्टिव प्राकृतिक उत्पादों और एम्फीफिलिक लिपिडिक टाइरोसिन डेरिवेटिव्स परिवार के कैलीस्पोन्गिडिक एसिड के कुल संश्लेषण पर किए गए कार्य को प्रस्तुत करता है।

अध्याय 1 व्यावसायिक रूप से उपलब्ध सस्ती चिरल प्रारंभिक सामग्री (-)-क्विनिक एसिड से शुरू होने वाले स्टीरियोसेलेक्टिव कुल संश्लेषण से एंटी-फंगल साइक्लोहेक्सेनोन बायोएक्टिव प्राकृतिक उत्पादों (-)-स्यूडोहाइग्रोफोरोन ए¹² और (-)-स्यूडोहाइग्रोफोरोन बी¹² का वर्णन करता है। इन संश्लेषणों की मुख्य विशेषताएं डोडेसिल मैग्नीशियम ब्रोमाइड का डायस्टेरियोसेलेक्टिव जोड़ हैं जो अत्यधिक ऑक्सीजन युक्त कीटोन से (-)-क्विनिक एसिड और एनोन के α -ऑक्सीकरण से प्राप्त होता है।

अध्याय 2 में एंटी-फंगल साइक्लोपेंटेनोन प्राकृतिक उत्पादों (-)-हाइग्रोफोरोन ए¹², (-)-4-ओ-एसिटिल-हाइग्रोफोरोन ए¹² और (+)-हाइग्रोफोरोन बी¹² के डी-(-)-टारटरिक एसिड से उच्च समग्र उपज के स्टीरियोसेलेक्टिव कुल संश्लेषण का वर्णन किया गया है। इस संश्लेषण का मुख्य चरण एक जलीय KOH मध्यस्थता डायस्टेरियोसेलेक्टिव इंट्रामोलेक्युलर एल्डोल प्रतिक्रिया है जो तीन सन्निहित चिरल केंद्रों के साथ β -हाइड्रॉक्सी कीटोन बनाता है, जिसे आगे (-)-हाइग्रोफोरोन ए¹² और (+)-हाइग्रोफोरोन बी¹² के लिए विस्तृत किया गया था।

अध्याय 3 एल-टार्टरिक एसिड से प्राप्त एपॉक्सी एस्टर से ($\pm$)-कैलीस्पोन्गिडिक एसिड और 2-एपी-($\pm$)-कैलीस्पोन्गिडिक एसिड के पहले लघु और त्वरित कुल संश्लेषण का वर्णन करता है। इन संश्लेषणों की उल्लेखनीय विशेषताएं एपॉक्साइड के स्टीरियोसेलेक्टिव ओपनिंग के साथ ऑर्गनोक्यूप्रेट्स और एस्टर की उपस्थिति में कीटोन के लिए ग्रिग्नार्ड अभिकर्मक के कीमोसेलेक्टिव जोड़ हैं।

TABLE OF CONTENTS

	Page No.
CERTIFICAT	i
ACKNOWLEDGEMENTS	ii
ABSTRACT	iv
TABLE OF CONTENTS	vi
LIST OF FIGURES	viii
LIST OF TABLES	ix
GENERAL EXPERIMENTAL CONSIDERATIONS	xvi
COMMON ABBREVIATIONS	xviii

CHAPTER 1:

STEREOSELECTIVE TOTAL SYNTHESIS OF (–)-PSEUDOHYGROPHORONE

A¹² AND (–)-PSEUDOHYGROPHORONE B¹²

1.1	Introduction	2
1.2	Objectives	6
1.3	Results and discussion	7
1.4	Conclusions	12
1.5	Experimental	13
1.6	References	24
1.7	Copies of NMR spectra of synthesized compounds	26

CHAPTER 2:

STEREOSELECTIVE TOTAL SYNTHESIS OF (–)-HYGROPHORONE A¹², (+)-HYGROPHORONE B¹² AND 4-O-ACETYL-HYGROPHORONE A¹²

2.1	Introduction	36
2.2	Objectives	41
2.3	Results and discussion	42
2.4	Conclusions	51
2.5	Experimental	52
2.6	References	72
2.7	Copies of NMR spectra of synthesized compounds	74
2.8	Crystallographic data for (+)-hygrophorone A ¹² <i>ent</i> -6	93

CHAPTER 3:

A SHORT AND EXPEDITIOUS TOTAL SYNTHESSES OF (±)-CALLYSPONGIDIC ACIDS AND 2-EPI-(±)-CALLYSPONGIDIC ACIDS

3.1	Introduction	97
3.2	Objectives	100
3.3	Results and discussion	102
3.4	Conclusions	107
3.5	Experimental	108
3.6	References	126
3.7	Copies of NMR spectra of synthesized compounds	128
	List of publications	154
	A BRIEF BIODATA OF THE AUTHOR	155

LIST OF FIGURES

CHAPTER 1

Figure	Title	Page No.
1.1	Structures of bioactive naturally occurring oxygenated cyclohexenones	3
1.2	Structures of bioactive naturally occurring pseudohygrophorones	4
1.3	^1H NMR of compound 20	26
1.4	^{13}C NMR of compound 20	26
1.5	^1H NMR of compound 21	27
1.6	^{13}C NMR of compound 21	27
1.7	^1H NMR of compound 15	28
1.8	^{13}C NMR of compound 15	28
1.9	^1H NMR of compound 22	29
1.10	^{13}C NMR of compound 22	29
1.11	^1H NMR of compound 14	30
1.12	^{13}C NMR of compound 14	30
1.13	^1H NMR of mixture of compounds 23 and 24	31
1.14	^{13}C NMR of compound 24	31
1.15	^1H NMR of compound of 25	32
1.16	^1H NMR of (–)-pseudohygrophorone A ¹² (<i>ent</i> - 6)	33
1.17	^{13}C NMR of (–)-pseudohygrophorone A ¹² (<i>ent</i> - 6)	33
1.18	^1H NMR of (–)-pseudohygrophorone B ¹² (<i>ent</i> - 7)	34
1.19	^{13}C NMR of (–)-pseudohygrophorone B ¹² (<i>ent</i> - 7)	34

CHAPTER 2

Figure	Title	Page No.
2.1	Representative naturally occurring chiral cyclopentenone molecules	36
2.2	Structures of naturally occurring hygrophorones	37
2.3	Molecular structure of (+)-hygrophorone A ¹² <i>ent</i> -6 from X-ray crystallography.	48
2.4	¹ H NMR of compound 32	74
2.5	¹³ C NMR of compound 32	74
2.6	¹ H NMR of compound 33	75
2.7	¹³ C NMR of compound 33	75
2.8	¹ H NMR of compound 34	76
2.9	¹³ C NMR of compound 34	76
2.10	¹ H NMR of compound 35	77
2.11	¹³ C NMR of compound 35	77
2.12	¹ H NMR of compound 27	78
2.13	¹³ C NMR of compound 27	78
2.14	¹ H NMR of compound 36	79
2.15	¹³ C NMR of compound 36	79
2.16	¹ H NMR of compound 37	80
2.17	¹³ C NMR of compound 37	80
2.18	¹ H NMR of compound 26	81
2.19	¹³ C NMR of compound 26	81
2.20	¹ H NMR of compound 25	82
2.21	¹³ C NMR of compound 25	82
2.22	¹ H NMR of compound 38	83

2.23	^{13}C NMR of compound 38	83
2.24	^1H NMR of (–)-hygrophorone A ¹² 6	84
2.25	^{13}C NMR of (–)-hygrophorone A ¹² 6	84
2.26	^1H NMR of (+)-hygrophorone A ¹² <i>ent</i> - 6	85
2.27	^{13}C NMR of (+)-hygrophorone A ¹² <i>ent</i> - 6	85
2.28	^1H NMR of compound 39	86
2.29	^{13}C NMR of compound 39	86
2.30	^1H NMR of compound 40	87
2.31	^{13}C NMR of compound 40	87
2.32	^1H NMR of 4- <i>O</i> -acetyl-hygrophorone A ¹² 7	88
2.33	^{13}C NMR of 4- <i>O</i> -acetyl-hygrophorone A ¹² 7	88
2.34	^1H NMR of compound 41	89
2.35	^{13}C NMR of compound 41	89
2.36	^1H NMR of compound 42	90
2.37	^{13}C NMR of compound 42	90
2.38	^1H NMR of compound 43	91
2.39	^{13}C NMR of compound 43	91
2.40	^1H NMR of (+)-hygrophorone B ¹² 8	92
2.41	^{13}C NMR of (+)-hygrophorone B ¹² 8	92
2.42	Molecular structure of (+)-hygrophorone A ¹² <i>ent</i> - 6	94

CHAPTER 3

Figure	Title	Page No.
3.1	Structures of bioactive molecules isolated from marine sponge.	98
3.2	^1H NMR of compound 15	128
3.3	^{13}C NMR of compound 15	128
3.4	^1H NMR of compound 19	129
3.5	^{13}C NMR of compound 19	129
3.6	^1H NMR of compound 14	130
3.7	^{13}C NMR of compound 14	130
3.8	^1H NMR of the mixture of compounds 13 and 21	131
3.9	^{13}C NMR of the mixture of compounds 13 and 21	131
3.10	^1H NMR of compound 22	132
3.11	^{13}C NMR of compound 22	132
3.12	^1H NMR of compound 23	133
3.13	^{13}C NMR of compound 23	133
3.14	^1H NMR of ($\pm$)-Callysopngidic Acid 1	134
3.15	^{13}C NMR of ($\pm$)-Callyspongidic Acid 1	134
3.16	^1H - ^1H COSY NMR of ($\pm$)-Callysopngidic Acid 1	135
3.17	^1H - ^1H NOESY NMR of ($\pm$)-Callysopngidic Acid 1	135
3.18	^1H - ^{13}C HMBC NMR of ($\pm$)-Callysopngidic Acid 1	136
3.19	^1H - ^{13}C HSQC NMR of ($\pm$)-Callysopngidic Acid 1	136
3.20	^1H NMR of 2- <i>epi</i> -($\pm$)-Callyspongidic Acid 1	137
3.21	^{13}C NMR of 2- <i>epi</i> -($\pm$)-Callyspongidic Acid 1	137
3.22	^1H - ^1H COSY NMR of 2- <i>epi</i> -($\pm$)-Callysopngidic Acid 1	138
3.23	^1H - ^1H NOESY NMR of 2- <i>epi</i> -($\pm$)-Callysopngidic Acid 1	138

3.24	^1H - ^{13}C HMBC NMR of 2- <i>epi</i> -($\pm$)-Callysopngidic Acid 1	139
3.25	^1H - ^{13}C HSQC NMR of 2- <i>epi</i> -($\pm$)-Callysopngidic Acid 1	139
3.26	^1H NMR of compound 19a	140
3.27	^{13}C NMR of compound 19a	140
3.28	^1H NMR of compound 14a	141
3.29	^{13}C NMR of compound 14a	141
3.30	^1H NMR of the mixture of compounds 13a and 21a	142
3.31	^{13}C NMR of the mixture of compounds 13a and 21a	142
3.32	^1H NMR of compound 22a	143
3.33	^{13}C NMR of compound 22a	143
3.34	^1H NMR of compound 23a	144
3.35	^{13}C NMR of compound 23a	144
3.36	^1H NMR of ($\pm$)-Callysopngidic Acid 2	145
3.37	^{13}C NMR of ($\pm$)-Callyspongidic Acid 2	145
3.38	^1H NMR of 2- <i>epi</i> -($\pm$)-Callyspongidic Acid 2	146
3.39	^{13}C NMR of 2- <i>epi</i> -($\pm$)-Callyspongidic Acid 2	146
3.40	^1H NMR of compound 19b	147
3.41	^{13}C NMR of compound 19b	147
3.42	^1H NMR of compound 14b	148
3.43	^{13}C NMR of compound 14b	148
3.44	^1H NMR of the mixture of compounds 13b and 21b	149
3.45	^{13}C NMR of the mixture of compounds 13b and 21b	149
3.46	^1H NMR of compound 22b	150
3.47	^{13}C NMR of compound 22b	150
3.48	^1H NMR of compound 23b	151

3.49	^{13}C NMR of compound 23b	151
3.50	^1H NMR of ($\pm$)-Callysopngidic Acid 3	152
3.51	^{13}C NMR of ($\pm$)-Callyspongidic Acid 3	152
3.52	^1H NMR of 2- <i>epi</i> -($\pm$)-Callyspongidic Acid 3	153
3.53	^{13}C NMR of 2- <i>epi</i> -($\pm$)-Callyspongidic Acid 3	153

LIST OF TABLES

CHAPTER 1	Page No.
Table 1.1 Comparison of ^1H and ^{13}C NMR data of natural (+)-pseudohygrophorone A ¹² 6 with synthetic (–)-pseudohygrophorone A ¹² <i>ent</i> - 6 in CDCl ₃	13
Table 1.2 Comparison of ^1H and ^{13}C NMR data of natural (+)-pseudohygrophorone B ¹² 7 with synthetic (–)-pseudohygrophorone B ¹² <i>ent</i> - 7 in CDCl ₃	14
CHAPTER 2	Page No.
Table 2.1 Comparison of ^1H and ^{13}C NMR spectral data of natural (–)-hygrophorone A ¹² 6 with synthetic (–)-hygrophorone A ¹² 6 in CDCl ₃	52
Table 2.2 Comparison of ^1H and ^{13}C NMR data of natural 4- <i>O</i> -acetyl-hygrophorone A ¹² 7 and synthetic 4- <i>O</i> -acetyl-hygrophorone A ¹² 7 in CDCl ₃	53
Table 2.3 Comparison of ^1H and ^{13}C NMR data of natural (+)-hygrophorone B ¹² 8 and synthetic (+)-hygrophorone B ¹² 8 in CDCl ₃	54
Table 2.4 Crystal data and structure refinement for (+)-hygrophorone <i>ent</i> - 6	95

CHAPTER 3**Page No.**

Table 3.1	Comparison of ^1H and ^{13}C NMR spectral data of natural (–)-Callyspongidic acid 1 with synthetic (±)-Callyspongidic acid 1 in CD_3OD	108
Table 3.2	Comparison of ^1H and ^{13}C NMR spectral data of natural (–)-Callyspongidic acid 2 with synthetic (±)-Callyspongidic acid 2 in CD_3OD	109
Table 3.3	Comparison of ^1H and ^{13}C NMR spectral data of natural (–)-Callyspongidic acid 3 with synthetic (±)-Callyspongidic acid 3 in CD_3OD	110

GENERAL EXPERIMENTAL CONSIDERATIONS

All solvents employed were purified by standard procedures. Anhydrous solvents were dried over sodium wire (THF, diethyl ether, benzene) or molecular sieves (CH_2Cl_2 , CHCl_3 , DMF). Nitrogen or Argon gas used for creating an inert atmosphere was freed from oxygen prior to entry into the reaction vessel.

Commercially sourced TLC plates were used, and the spots were visualized by exposure to iodine or by dipping in KMnO_4 . Column chromatography was carried out on silica gel (230–400 mesh) using hexane and ethyl acetate mixtures as eluent unless otherwise mentioned.

Optical rotations were recorded on an Autopol V (Rudolph Research Flanders, NJ) instrument. All the rotations were measured at 589 nm (sodium D-line).

All melting points reported in this thesis are uncorrected and were taken on an electric melting point apparatus (Ambassador, India).

IR spectra were taken within the range $4000\text{--}600\text{ cm}^{-1}$ either as KBr pellets or neat on a Nicolet (Madison, USA) FT-IR spectrophotometer (Model Protégé 460).

^1H -NMR spectra were recorded on 300 MHz or 400 MHz, or 500 MHz Bruker Spectrospin DPX FT-NMR instruments. The solvents employed were CDCl_3 or CD_3OD with Me_4Si as the internal standard. The multiplicities are denoted as s-singlet, brs-broad singlet, d-doublet, brm-broad multiplet, t-triplet, q-quartet, dt-doublet triplet and m-multiplet. ^{13}C -NMR spectra were recorded at 75 MHz or 100 MHz, or 125 MHz instruments. The chemical shifts are reported in δ values (parts per million, ppm) relative to the internal standard Me_4Si .

High-resolution mass spectra were recorded with a Q-TOF Bruker instrument, using electrospray ionization (ESI) as the ionization method.

X-ray crystallography

Suitable crystal of compounds was obtained by slow evaporation of their saturated solutions in chloroform-methanol solvent mixture or water. BRUKER AXS SMART-APEX diffractometer equipped with CCD area detector ($K\alpha=0.71073\text{\AA}$, monochromator: graphite). Frames were collected at $T=298\text{K}$ by ω , ϕ and 2θ -rotation with full quadrant data collection strategy (four domains each with 600 frames) at 10s per frame with SMART. The measured intensities were reduced to F^2 and corrected for absorption with SAINT. Structure solution and refinement were carried out with the SHELXTL package by direct methods. Non-hydrogen atoms were refined anisotropically. All hydrogen atoms were included in idealized positions, and a riding model was used for the refinement. Images were created with the program Diamond.

LIST OF ABBREVIATIONS

Ac	:	acetyl
Ac ₂ O	:	acetic anhydride
Anhyd.	:	anhydrous
Ar	:	aryl
aq	:	aqueous
Bn	:	benzyl
brs	:	broad singlet
C	:	concentration
calcd	:	calculated
cat.	:	catalytic
cm	:	centimeter
DCM	:	dichloromethane
dd	:	doublet of a doublet
ddd	:	doublet of a doublet of a doublet
ddt	:	doublet of a doublet of a triplet
dt	:	doublet of a triplet
DIPA	:	<i>N, N</i> -diisopropylamine
DIPEA	:	<i>N, N</i> -diisopropylethylamine
DMF	:	<i>N, N</i> -dimethylformamide
DMAP	:	4-(<i>N, N</i> -dimethylamino)pyridine
2,2-DMP	:	2,2-dimethoxy propane
DMP	:	Dess-Martin periodinane
DMSO	:	dimethylsulfoxide
<i>dr</i>	:	diastereomeric ratio
dt	:	doublet of a triplet
<i>ent</i>	:	enantiomer
eq.	:	equivalents
Et	:	ethyl
Fig.	:	figure
g	:	gram(s)
h	:	hour(s)

HRMS	:	high resolution mass spectrum
Hz	:	hertz
IC ₅₀	:	half maximal inhibitory concentration
<i>i</i> Pr	:	isopropyl
IR	:	infrared
liq	:	liquid
Lit.	:	literature
LiHMDS	:	lithium hexamethyldisilazide
m	:	multiplet
<i>m</i> -CPBA	:	<i>meta</i> -chloroperbenzoic acid
Me	:	methyl
mg	:	milligram(s)
MHz	:	megahertz
min	:	minute(s)
mL	:	milliliter(s)
mmol	:	millimole
MOM	:	methoxymethyl
M.p.	:	melting point
MS	:	Molecular sieves
NMR	:	Nuclear Magnetic Resonance
<i>p</i>	:	para
Ph	:	phenyl
ppm	:	parts per million
PPTS	:	pyridinium para-toluenesulphonate
<i>p</i> -TSA	:	<i>para</i> -toluenesulfonic acid
py	:	pyridine
pent.	:	pentet
q	:	quartet
rt	:	room temperature
s	:	singlet
sat.	:	saturated
<i>t</i> Bu	:	<i>tertiary</i> -Butyl
t	:	triplet

td	:	triplet of a doublet
TBAF	:	tetra- <i>n</i> -butylammonium fluoride
TBS	:	<i>tertiary</i> -butyldimethylsilyl
TBDMSCl	:	<i>tertiary</i> -butyldimethylsilyl chloride
TBDMSOTf	:	<i>tertiary</i> -butyldimethylsilyl trifluoromethanesulfonate
tert	:	<i>tertiary</i>
TFA	:	trifluoroacetic acid
THF	:	tetrahydrofuran
TLC	:	thin layer chromatography
UV	:	ultraviolet