REACTION DEVELOPMENT FOR THE TOTAL SYNTHESES OF THE TERPENOID NATURAL PRODUCTS (+)-PSIGUADIAL B, (+)-RUMPHELLAONE A, AND (–)-ISODOCARPIN

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To my family and friends

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ABSTRACT

The *de novo* synthesis of bioactive natural products provides an opportunity to learn more about the mechanism of bioactivity and to develop novel chemistry that is of interest to the synthetic community. Herein, we describe our strategy for the total synthesis of the trans-fused cyclobutane containing meroterpenoid (+)-psiguadial B. Key to this strategy was the development of a photochemical Wolff Rearrangement with asymmetric ketene aminolysis. A palladium-catalyzed C–H alkenylation is used to build structural complexity, and we use two different epimerization strategies to perform an enantiodivergent synthesis of (+)-psiguadial B.

This strategy was explored further and applied to the synthesis of chiral cyclobutanes through a 1,2-difunctionalization strategy, wherein a C–H arylation forges one carbon-carbon bond and a subsequent decarboxylative cross-coupling enables functionalization at the adjacent carbon. This strategy enabled the asymmetric total synthesis of (+)-rumphellaone A in 9 steps.

This report also highlights the work we have conducted in the development of a unified strategy for the enmein-type *ent*-kauranoid natural product, (–)-isodocarpin. We detail our investigation of a convergent cross-electrophile coupling as a means to build the core of (–)-isodocarpin. We also discuss our development of a 1,2-addition/semi-Pinacol rearrangement strategy for the preparation of all-carbon quaternary centers, which can be elaborated to enmein-type *ent*-kauranoid natural product scaffolds.

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LIST OF ABBREVIATIONS

$[\alpha]_D$	angle of optical rotation of plane-polarized light
Å	angstrom(s)
p-ABSA	*para*-acetamidobenzenesulfonyl azide
Ac	acetyl
acac	acetylacetonate
AIBN	azobisisobutyronitrile
alk	alkyl
aq	aqueous
AQN	anthraquinone-1,4-diyl diether
Ar	aryl group
atm	atmosphere(s)
BiOX	bi(oxazoline)
BINAP	2,2'-bis(diphenylphosphino)-1,1'-binaphthyl
BINOL	1,1'-bi-2,2'-naphthol
bipy	2,2'-bipyridine
Bn	benzyl
Boc	*tert*-butoxycarbonyl
bp	boiling point
br	broad
Bu	butyl
i-Bu	*iso*-butyl
n-Bu	butyl or *norm*-butyl

t-Bu	*tert*-butyl
BQ	1,4-benzoquinone
Bz	benzoyl
c	concentration of sample for measurement of optical rotation
^{13}C	carbon-13 isotope
/C	supported on activated carbon charcoal
°C	degrees Celcius
calc'd	calculated
CAM	cerium ammonium molybdate
CAN	ceric ammonium nitrate
cat.	catalyst
Cbz	benzyloxycarbonyl
CD	Cinchonidine
cf.	consult or compare to (Latin: *confer*)
cis	on the same side
cm^{-1}	wavenumber(s)
cod	1,5-cyclooctadiene
CN	Cinchonine
CoA	Coenzyme A
conc.	concentrated
conv.	conversion
Cp	cyclopentadienyl
CSA	camphor sulfonic acid

Cy	cyclohexyl
Δ	heat or difference
δ	chemical shift in ppm
d	doublet
d	deutero or dextrorotatory
D	deuterium
dba	dibenzylideneacetone
DBU	1,8-diazabicyclo[5.4.0]undec-7-ene
DCE	1,2-dichloroethane
DDQ	2,3-dichloro-5,6-dicyano-1,4-benzoquinone
de novo	starting from the beginning; anew
DIPEA	*N*,*N*-diisopropylethylamine
DHQ	dihydroquinine
DHQD	dihydroquinidine
DIBAL	diisobutylaluminum hydride
DMAP	4-(dimethylamino)pyridine
DME	1,2-dimethoxyethane
DMEDA	*N*,*N*'-dimethylethylenediamine
DMF	*N*,*N*-dimethylformamide
DMPU	1,3-dimethyl-3,4,5,6-tetrahydro-2(1H)-pyrimidinone
DMSO	dimethylsulfoxide
dppe	1,2-bis(diphenylphosphino)ethane
dppf	1,1'-bis(diphenylphosphino)ferrocene

dr	diastereomeric ratio
dtbpy	4,4'-di-*tert*-butyl-2,2'-bipyridine
ee	enantiomeric excess
E	methyl carboxylate (CO_2CH_3)
E^+	electrophile
E	*trans* (entgegen) olefin geometry
EDCI	*N*-(3-dimethylaminopropyl)-*N*'-ethylcarbodiimide hydrochloride
e.g.	for example (Latin: *exempli gratia*)
EI	electron impact
ent	enantiomer of
epi	epimeric
equiv	equivalent(s)
ESI	electrospray ionization
Et	ethyl
et al.	and others (Latin: *et alii*)
FAB	fast atom bombardment
FTIR	fourier transform infrared spectroscopy
g	gram(s)
Grubbs-II	Grubbs' catalyst™ 2nd generation
h	hour(s)
^1H	proton
[H]	reduction
HDA	hetero-Diels–Alder

HFIP	hexafluoroisopropanol
HG-II	Hoveyda–Grubbs' catalyst™ 2nd generation
HIV	human immunodeficiency virus
HMBC	heteronuclear multiple-bond correlation spectroscopy
HMDS	hexamethyldisilazide
HMPA	hexamethylphosphoramide
hv	irradiation with light
HPLC	high performance liquid chromatography
HRMS	high resolution mass spectrometry
Hz	hertz
IC_{50}	half maximal inhibitory concentration (50%)
i.e.	that is (Latin: *id est*)
iso	isomeric
in situ	in the reaction mixture
J	coupling constant in Hz
k	rate constant
kcal	kilocalorie(s)
kg	kilogram(s)
L	liter or neutral ligand
l	levorotatory
LA	Lewis acid
LC/MS	liquid chromatography–mass spectrometry
LDA	lithium diisopropylamide

LED	light-emitting diode
m	multiplet or meter(s)
M	molar or molecular ion
m	*meta*
μ	micro
m-CPBA	*meta*-chloroperbenzoic acid
Me	methyl
mg	milligram(s)
MHz	megahertz
MIC	minimum inhibitory concentration
min	minute(s)
mL	milliliter(s)
MM	mixed method
mol	mole(s)
MOM	methoxymethyl
Ms	methanesulfonyl (mesyl)
MS	molecular sieves
MTT	3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide
m/z	mass-to-charge ratio
NBS	*N*-bromosuccinimide
ND	not determined
NHC	*N*-heterocyclic carbene
nm	nanometer(s)

nM	nanomolar
NMO	*N*-methylmorpholine *N*-oxide
NMP	*N*-methyl-2-pyrrolidone
NMR	nuclear magnetic resonance
NOE	nuclear Overhauser effect
NOESY	nuclear Overhauser enhancement spectroscopy
NPh	naphthyl
Nu^-	nucleophile
o	*ortho*
o-QM	*ortho*-quinone methide
[O]	oxidation
P	peak
p	*para*
PCC	pyridinium chlorochromate
PDC	pyridinium dichromate
Ph	phenyl
PhMe	toluene
pH	hydrogen ion concentration in aqueous solution
PHAL	1,4-phthalazinediyl diether
PIFA	[bis(trifluoroacetoxy)iodo]benzene
PHOX	phosphinooxazoline
Pin	pinacol
Piv	pivaloyl

pK_a	acid dissociation constant
pm	picometer(s)
PMB	*para*-methoxybenzyl
ppm	parts per million
PPTS	pyridinium *para*-toluenesulfonate
Pr	propyl
i-Pr	isopropyl
n-Pr	propyl or *norm*-propyl
psi	pounds per square inch
py	pyridine
PyBOX	pyridine-bis(oxazoline)
PyOx	pyridine-oxazoline
PYR	2,5-diphenyl-4,6-pyrimidinediyl diether
q	quartet
QD	Quinidine
QN	Quinine
QuinOx	quinoline-oxazoline
quant.	quantitative
R	generic (alkyl) group
R_L	large group
R	rectus
RCM	ring-closing metathesis
recry.	recrystallization

ref	reference
R_f	retention factor
rgt.	reagent
rt	room temperature
s	singlet or seconds
S	sinister
sat.	saturated
SET	single-electron transfer
SFC	supercritical fluid chromatography
t	triplet
TBAF	tetra-*n*-butylammonium fluoride
TBAI	tetra-*n*-butylammonium iodide
TBME	*tert*-butyl methyl ether
TBS	*tert*-butyldimethylsilyl
TC	thiophene-2-carboxylate
temp	temperature
terpy	2,2′:6′,2″-terpyridine
Tf	trifluoromethanesulfonyl
TFA	trifluoroacetic acid
THF	tetrahydrofuran
TIPS	triisopropylsilyl
TLC	thin layer chromatography
TMEDA	*N*,*N*,*N*’,*N*’-tetramethylethylenediamine

TMS	trimethylsilyl
TOF	time-of-flight
tol	tolyl
TPAP	tetrapropylammonium perruthenate
trans	on the opposite side
Ts	*para*-toluenesulfonyl (tosyl)
UV	ultraviolet
vide infra	see below
w/v	weight per volume
X	anionic ligand or halide
xs	excess
Z	*cis* (zusammen) olefin geometry