9ème Symposium Francophone de Synthèse Totale

23-24 Mai 2019

Nantes

Chimie Et Interdisciplinarité Synthèse, Analyse, Modélisation
CEISAM
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"The total synthesis of complex natural products remains the most difficult, daunting, and challenging endeavor in organic chemistry. It is also the most humbling, exhilarating, and formative entreprise in our science."

(István E. Markó, Science 2001, *294*, 1842)

Le Symposium Francophone de Synthèse Totale est une occasion pour nous de conter nos aventures en synthèse totale, partager nos joies, nos désillusions, nos échecs et surtout de continuer à apprendre grâce à cette discipline.

Jules Verne, né à Nantes, était un auteur qui, dans un domaine tout autre, a su lui aussi conter d'extraordinaires voyages. Alors que nous nous apprêtons à célébrer les 150 ans de parution de « 20 000 lieues sous les mers » nous vous proposons pour cette escale à Nantes d'effectuer un voyage de 20 000 lieues en synthèse totale.

Le comité d'organisation manceau-nantais du 9ème Symposium Francophone de Synthèse Totale est heureux de vous accueillir à Nantes sur le campus de la Faculté des Sciences et des Techniques de l'Université de Nantes.

Nous tenons à remercier nos partenaires pour leur soutien financier, les conférencier·ère·s et les différent·e·s orateur·rice·s qui ont accepté de nous présenter une partie de leurs travaux.

Nous vous souhaitons un agréable séjour parmi nous ainsi que des échanges fructueux.

Bonne aventure en Synthèse Totale !

Le comité d'organisation

Pr. Sylvain COLLET, Laboratoire CEISAM, Université de Nantes
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Programme

Jeudi 23 mai 2019

13h00-13h50	**Accueil des participants**
13h50	**Mot d'ouverture**
14h00-15h00	**CP1: Pr. Janick Ardisson** Synthesis and Structure of Biologically Active Natural Products. Development of Methods
15h00-15h30	**CO1: Dr. Philippe Peixoto** Bio-inspired total synthesis of Securinega alkaloids
15h30-16h00	**CO2: Pr. Vincent Dalla** Towards Sustainability and Molecular Diversity by Design of Domino and Multicatalytic Strategies in N-Acyliminium Ion Chemistry
16h00-17h00	**Pause-Café - Posters**
17h00-17h30	**CO3: Pr. Véronique Bellosta** Toward the total synthesis of bioactive polyhydroxylated polyenic macrolactones
17h30-18h00	**CO4: Dr. Aurélien De la Torre** Journeys in total synthesis
18h00	**Clôture de la 1ère journée**

Programme

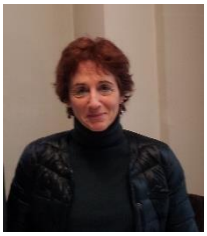
Vendredi 24 mai 2019

8h30-9h30	**CP2: Pr. Jonathan Burton** Oxonium Ions, Rearrangements and Natural Products
9h30-10h00	**CO5: Dr. Laurence Miesch** Ynamides and Enamides for the total synthesis of Natural Products
10h00-10h30	**CO6: Dr. Andrei Corbu** Chromenes in total synthesis: Metachromin T and Tuberatolide B Case Studies
10h30-11h00	**Pause-Café - Posters et exposants**
11h00-11h30	**CO7: Dr. Sandrine Py** Carbohydrates-derived nitrones as synthetic tools for the discovery of novel classes of iminosugars
11h30-12h00	**CO8: Dr. Zacharias Amara** Photo-oxidations with Supported Photocatalysts & Applications to Natural Products Synthesis
12h00-12h30	**CO9: Dr. Xavier Bugaut** Towards Bioactive and/or Natural Atropisomers by Central-to-Axial Conversion of Chirality
12h30-14h00	**Buffet - Posters et exposants**
14h00-15h00	**CP3: Pr. Antonio Echavarren** Gold-catalyzed chiral folding of enynes
15h00	**Clôture du Symposium et remise du prix poster**

Résumés des conférences plénières

CP1

Synthesis and Structure of Biologically Active Natural Products. Development of Methods

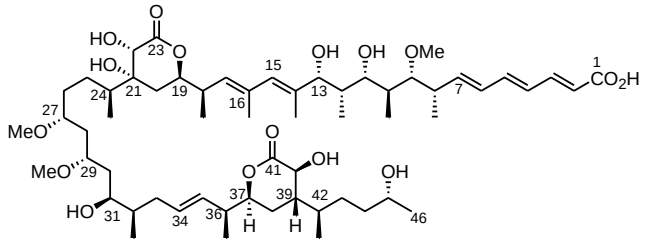


Janick Ardisson

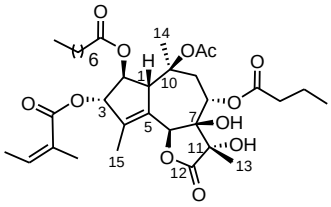
CNRS UMR 8038 CiTCoM, Cibles Thérapeutiques et Conception de Médicaments,
Produits Naturels, Analyse et Synthèse
Faculté de Pharmacie de Paris.
Université Paris Descartes

The team has a particular interest in the multi-stage synthesis of natural products with biological (or therapeutic) interests. More precisely, main research projects involve the total synthesis of molecules possessing complex structures: polyketides, terpenes, alkaloids. Although antitumorals (Thapsigargin, Hemicalide) are mainly targeted, an antidiabetic agent (FR225654) and recently a neurotrophic (Vibsatin A) have also attracted our attention. These syntheses are carried out through the development of original methods in organometallic chemistry (Pd, Rh, Au, …).

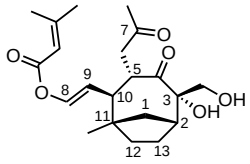
The determination of the relative configurations and synthesis of *Hemicalide*, a cytotoxic polyketide of marine origin, studies towards the synthesis of the guaianolide *Thapsigargin*, a nM SERCA inhibitor, and towards the synthesis of *Vibsatin A*, a diterpenoid with prominent neurite outgrowth activity, will be presented.



Hemicalide



Thapsigargin



Vibsatin A

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4. Sanogo, Y.; Ben Othman, R.; Dhambri, S.; Selkti, M.; Jeuken, A.; Prunet, J.; Férézou, J.-P.; Ardisson, J.; Lannou, M.-I.; Sorin, G. Ti(II) and Rh(I) Complexes as Reagents towards Thapsigargin Core. *J. Org. Chem.* ***2019****, 84, 5821–5830.*

CP2

Oxonium Ions, Rearrangements and Natural Products

Jonathan W. Burton

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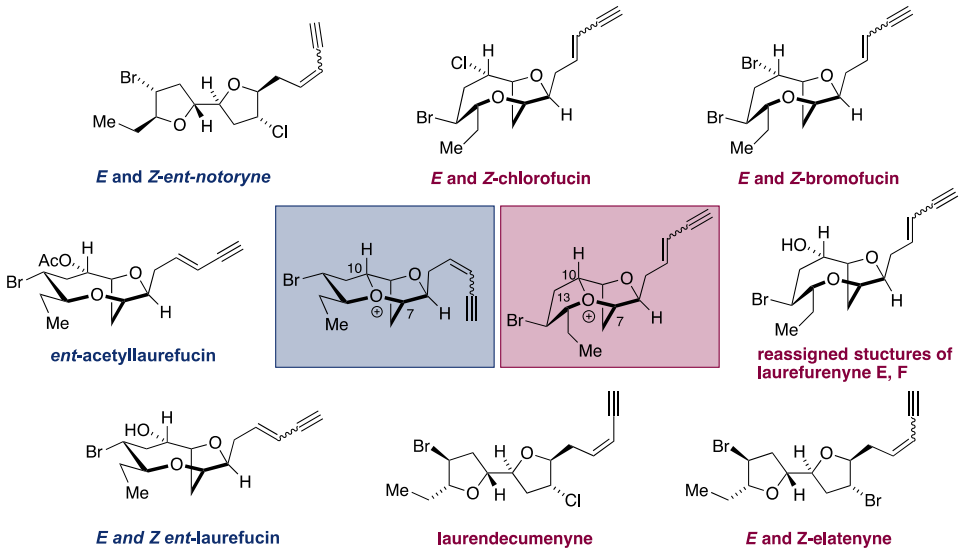
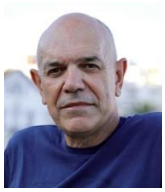
Abstract :

Oxonium ions, molecules with a trivalent oxygen atom that carries a formal positive charge, have a rich history in chemistry. The simplest and most prevalent is the hydronium ion – H_3O^+. Trialkyl oxonium ions, e.g. Meerwein's salts (Me_3O^+ BF_4^- and Et_3O^+ BF_4^-) are less common, and are among the most potent alkylating agents encountered in the laboratory. Although the hydronium ion is ubiquitous in chemistry and biology, trialkyl oxonium ions are rarities in both areas. Interestingly, a number of halogenated natural products isolated from *Laurencia* species, are proposed to arise biosynthetically *via* complex trialkyl oxonium ion intermediates (red and grey boxes). Good circumstantial evidence for the intermediacy of such oxonium ions has been provided through biomimetic total syntheses of a number of *Laurencia* natural products. This lecture will describe our work on the synthesis and characterisation of four of these biosynthetically relevant oxonium ions (red and grey boxes) which represent the most structurally and stereochemically complex oxonium ions characterised to date. The utility of these oxonium ions in the total synthesis of more than ten halogenated natural products from *Laurencia* species will also be described.



CP3

GOLD-CATALYZED CHIRAL FOLDING OF ENYNES



Antonio M. Echavarren

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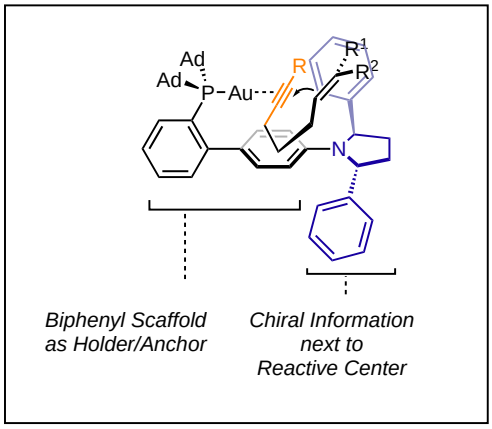
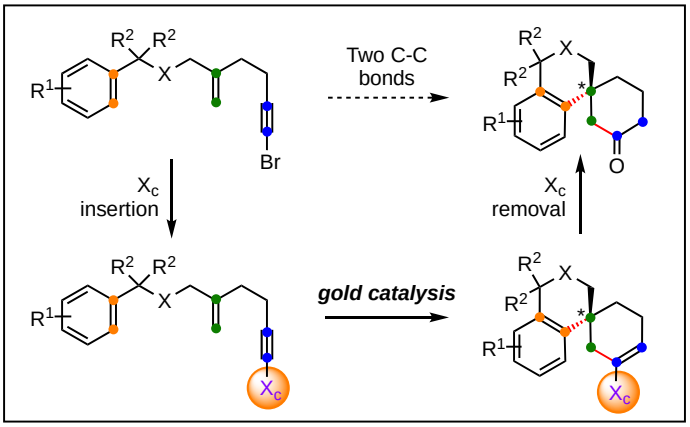
Our group has developed cyclizations and cascade reactions based on the selective activation of alkynes with cationic gold(I) complexes[1] for the construction of complex polycyclic molecules such as englerin A, schisanwilsonene, and other sesquiterpenoids.[2] We have also reported the first enantioselective cycloaddition between alkynes and alkenes that leads to chiral cyclobutenes in a general manner.[3] As a step forward towards mimicking the action of polyene cyclases, which catalyze a wide variety of transformations for the formation of complex carbocyclic skeletons, we have recently developed new series of C_2-chiral gold(I) catalysts for the enantioselective folding of enynes.





Recent results on the application of these new chiral catalysts for the synthesis of natural products as well on the development of alternative strategies based on the use of chiral auxiliaries for the construction of polycyclic skeletons will be presented.

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Résumés des communications orales

CO1

Bio-inspired total synthesis of Securinega alkaloids

Philippe A. Peixoto

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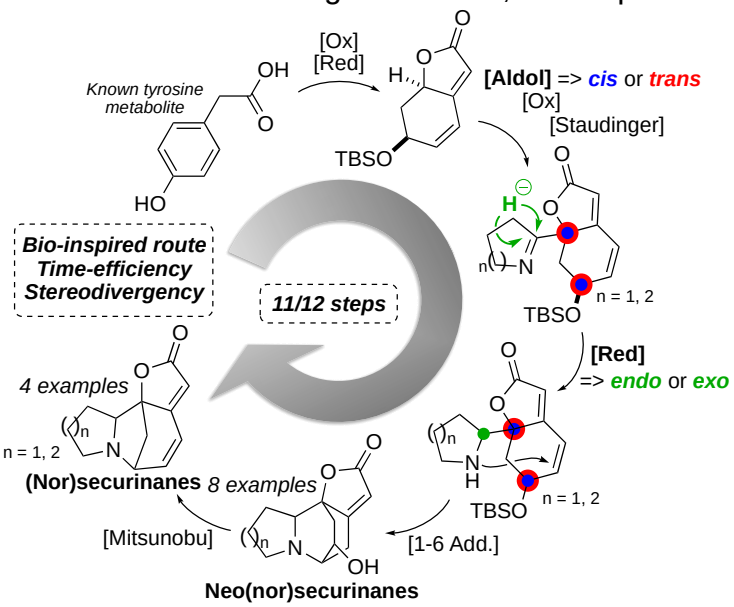
Abstract :

The so-called Securinega alkaloids constitute a class of tetracyclic biologically active specialized metabolites isolated principally from subtropical plants belonging to the Phyllanthaceae family.[1] While mixed dimers, trimers and even tetramers were recently isolated, all monomeric structures exhibit similar tetracyclic backbones, namely neo(nor)securinane and (nor)securinane.

Most interestingly, almost all possible diastereomers were found to be naturally occurring, and often demonstrated significant pharmacological properties.[1] To date, most Securinega alkaloids syntheses have been achieved, but a genuine doubt still exists in regard to the absolute configuration of certain monomeric members.[1,2]

Following a strategy based on alternative hypotheses for their biosynthesis, an easy and time-efficient divergent synthesis enabled access to twelve of those alkaloids featuring (neo)(nor)securinane skeletons. This work,[3] and its implication in the structural reassigment of two monomeric Securinega members, will be presented.



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CO2

Towards Sustainability and Molecular Diversity by Design of Domino and Multicatalytic Strategies in *N*-Acyliminium Ion Chemistry

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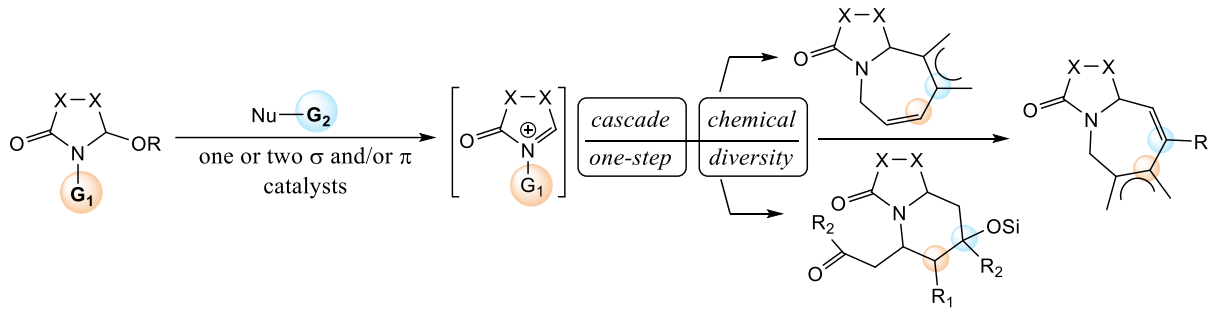
Abstract :

Over the last decade my research group developed some efficient catalytic methodologies in the area of *N*-acyliminium ion chemistry. Those methods are largely contingent to the use of Brønsted and Lewis superacidic catalysts of the triflate and triflimidate family to activate and enable effective alkylations of cyclic N,O-acetals as *N*-acyliminium precursors. Driven by the current societal stakes to develop sustainable chemistry with the respect of atom- and step economy principles as the guidelines, recently we particularly endeavored to integrate our catalytic *N*-acyliminium processes into more complex, sequential events. This talk will illustrate our efforts in designing cascade and multicatalytic transformations that provide a wide array of relatively sophisticated polyanellated structures from trivial cyclic N,O-acetals with general good efficiency.[1]



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CO3

Toward the total synthesis of bioactive polyhydroxylated polyenic macrolactones

Véronique Bellosta

Chimie Moléculaire, Macromoléculaire, Matériaux, ESPCI Paris, CNRS, PSL University, 10 rue Vauquelin, 75005 Paris

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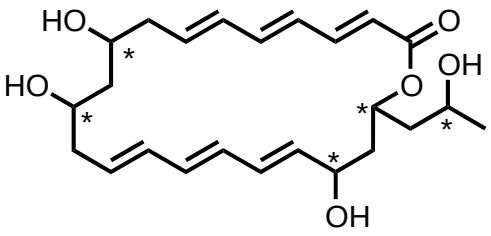
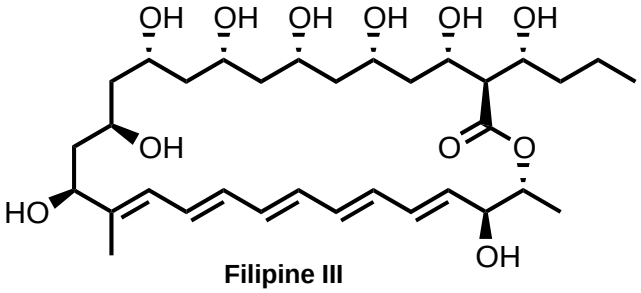
Abstract :

Polyenes encompass a wide range of biologically relevant natural products from a wide variety of sources, including marine organisms, fungi or moulds. In the context of our interest in polyenes, two bioactive macrolactones, wortmannilactone C[1] and filipin III,[2-4] were selected as synthetic targets. Our efforts toward the total synthesis of these natural products[5,6] will be presented as well as the development of stereoselective methods to access 1,3,5,7-tetraols and trienol subunits.



Wortmannilactone C



Filipine III

References :

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CO4

JOURNEYS IN TOTAL SYNTHESIS

Aurélien DE LA TORRE, with the groups of Profs. Maulide, Marek and Kouklovsky

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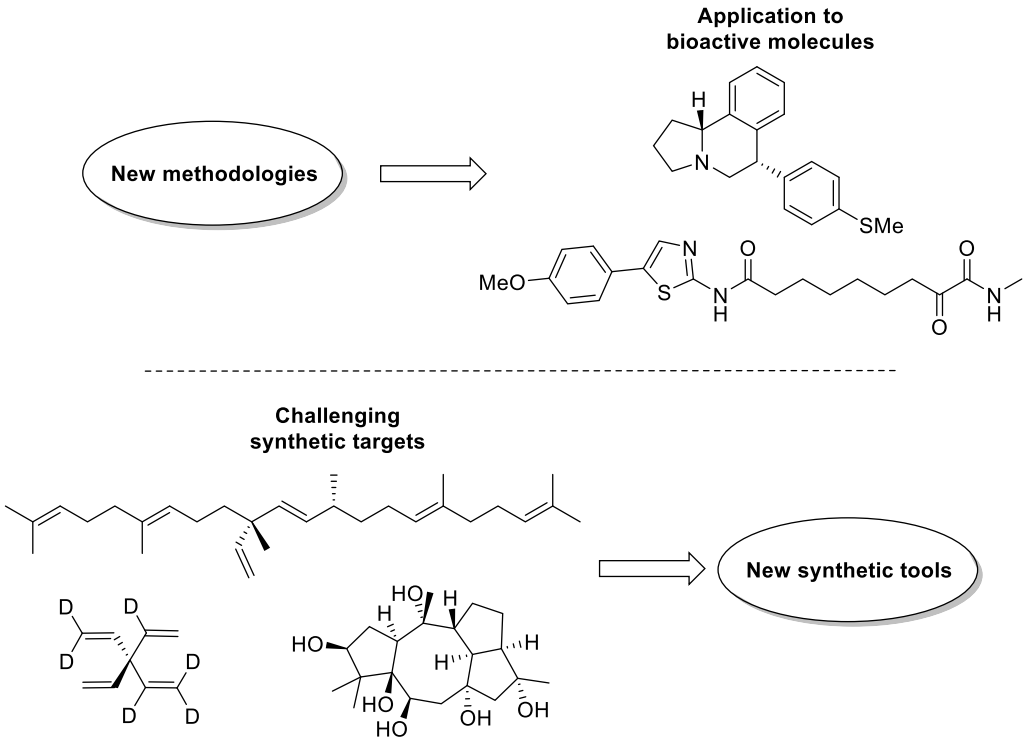
Synthetic methodologies and total synthesis are two complementary aspects of organic chemistry.[1] Natural product total syntheses are often designed to use and highlight the synthetic interest of a new methodology. In these cases, the target is chosen after having developed the methodology, and is chosen to fit to it. However, total synthesis can sometimes inspire the development of new methodologies, as part of a novel synthetic approach.

In this presentation, we will show examples of methodologies which were applied to the syntheses of bioactive molecules,[2] as well as total syntheses that required unlocking a synthetic tool to allow an efficient strategy.[3]



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3) M. Cormier, A. de la Torre, I. Marek, *Angew. Chem. Int. Ed.* **2018**, *57*, 13237-13241.

CO5

Ynamides and Enamides for the total synthesis of Natural Products

Laurence Miesch,

Equipe Synthèse Organique et Phytochimie, Institut De Chimie CNRS-UdS UMR 7177; 4, Rue Blaise Pascal CS 90032, 67081 Strasbourg, France

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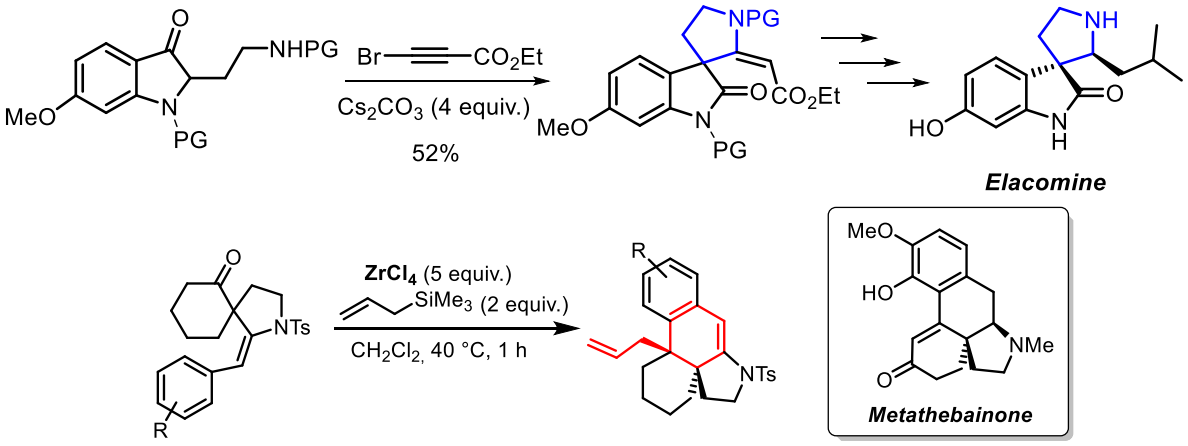
Abstract :

Azaspiro scaffolds are increasingly being used in drug discovery because of their three-dimentionality. A large group of bioactive, natural diterpenoids possesses a quaternary carbon incorporated whitin a spirocyclic system that is part of a more complex ring system. In particular, spiroindoles such as Aspidosperma alkaloids like elacomine or jerantinine E are important targets due to their valuable pharmacological profiles. Because of their unique polarization of the triple bond, ynamides became ideal building blocks for the synthesis of nitrogen-containing molecules.Transition metal-catalyzed 7-*exo-dig* cyclization of silyl enol ether-ynesulfonamides afforded only bridged bicyclic keto-ynamides in a short time at room temperature.[1] Thus, we focused on the spirocyclization from keto-ynamides and found that spontaneous spirocyclization of keto-sulfonamides via ynamides through one-pot process was possible.[2] The obtained azaspiro compounds are building blocks for indole alkaloids. Hence, we investigated the total synthesis of elacomine. Our research interest in the chemistry of ketoynamides led us to exclusively *E*-configurated spiro-tertiary enesulfonamides.[3] The exploration of the activation of the carbonyl in the presence of a Lewis acid and allylsilane, provided a spiro-tetracyclic fused ring system[4] that may serve as an analogue of the analgesic metathebainone of the morphinan family.



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CO6

Chromenes in total synthesis: Metachromin T and Tuberatolide B Case Studies

Andrei Corbu [a], Kelly Maurent [a], Stellios Arsenyadis [b]

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[b] *Queen Mary University of London – School of Biological and Chemical Sciences – Mile End Road, London E1 4NS, United Kingdom*
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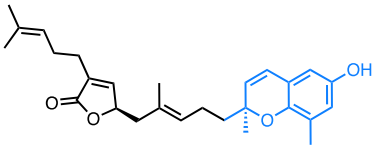
Abstract :

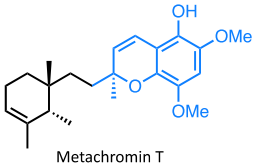
A large class of terpenoid natural products, namely the meroterpenes, is defined by a polyphenolic moiety coming from a polyketide biosynthetic pathway. Their total synthesis is important, not only for the challenge, but also for their biological activity. Our first target, metachromin T showed cytotoxicity against L1210 murine leukemia and KB human epidermoid carcinoma cells *in vitro.*[1] The other synthetic target, tuberatolide B is an Farnesoid X receptor antagonist, a rare propriety of small molecules, that can open new therapeutic uses.[2]

The challenges of a convergent strategy towards the tuberatolides and the metachromins will be presented.[3] Also, a focal point will be the chirality of the benzopyrane-chromene part and the methodological development around it.

A newer vision about retrosynthetic strategies involving enzyme catalysed transformations will also be applied in the synthetic campaign towards chromene containing natural products.[4]



Tuberatolide B



Metachromin T

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CO7

CARBOHYDRATE-DERIVED NITRONES AS SYNTHETIC TOOLS FOR THE DISCOVERY OF NOVEL CLASSES OF IMINOSUGARS

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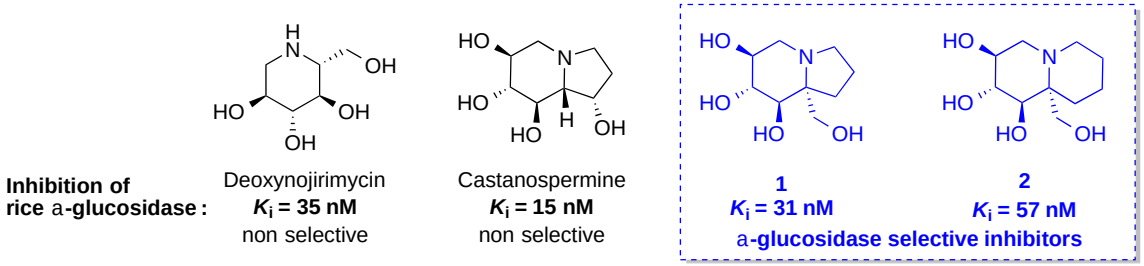
Abstract :

Due to their stability *in vivo* and their activity as glycosidase inhibitors or activators, iminosugars are among the most promising drug candidates for the treatment of diseases such as diabetes, viral infections and lysosomal storage disorders.[1] Recently, our group has identified a series of iminosugars (i.e. compounds **1** and **2**), exhibiting a quaternary center in α-position of their nitrogen atom, which proved to be excellent inhibitors (nanomolar K_i) of α-glucosidases with unprecedented selectivity.[2]



In this communication, the synthesis of novel iminosugars from carbohydrate-derived nitrones will be presented. The variety of synthetic methods that can be applied to nitrones to prepare bioactive molecules will be emphasized by successful syntheses of original iminosugar scaffolds.[3] Investigation of the biological activities of these new molecules will also be presented.

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CO8

Photo-oxidations with Supported Photocatalysts & Applications to Natural Products Synthesis

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Abstract :

Photo-oxidations are important reactions occurring in Nature. They rely on the generation of singlet oxygen, which is produced by sensitization of molecular oxygen with light and a photocatalyst. Singlet oxygen is a highly reactive species that can induce structural changes in biomolecules, or interfere with secondary metabolic pathways to produce important natural products such as artemisinin, a major antimalarial drug.[1] However, despite the large number of studies dedicated to photo-oxidations and singlet oxygen reactivity, these transformations remain underutilized in organic synthesis. One of the reasons for this is the high sensitivity of singlet oxygen to its local environment, which makes it difficult to react in a productive manner.[2]

We recently developed a heterogeneous photocatalytic system that offers a potential solution to this problem and efficiently applies to natural products synthesis.[3] Our approach focuses on the adsorption of a photocatalyst ($[Ru(bpy)_3]^{2+}$) on an inorganic material (SiO_2) to produce a stable heterogeneous system. This strategy introduces new physical and chemical parameters to control photochemical reactions with singlet oxygen. The catalytic performances of such systems are compared using dedicated batch and flow photo-reactors resulting in an optimization at the micro- and macro-scale levels. The combination of these factors might contribute to the development of cost-competitive photo-oxidation processes with wider applications to natural products synthesis.

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CO9

Towards Bioactive and/or Natural Atropisomers by Central-to-Axial Conversion of Chirality

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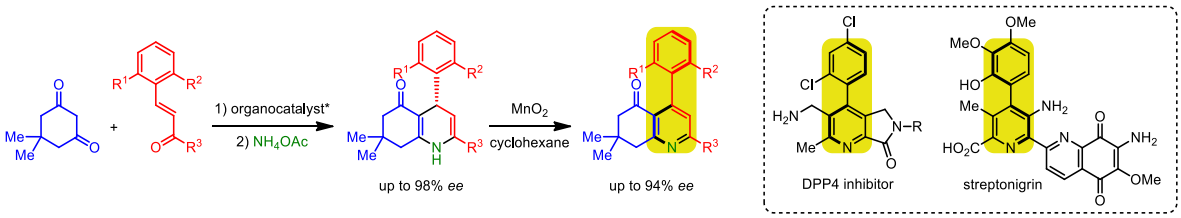
Abstract:

Atropisomers are structural motifs that arise because of the restricted rotation around a single bond. They possess interesting properties, especially a defined spatial organization, that have made them very popular in catalyst design.[1] Atropisomeric structures are also present in diverse natural products[2] and gain significance in the design of bioactive compounds.[3] The enantioselective preparation of atropisomers represents a dauting synthetic challenge.[4] Indeed, in addition to the necessity of favoring the formation of one enantiomer over the other, the kinetic stability of all axially chiral synthetic intermediates has to be considered to avoid their enantiomerization.

Recently, we have developed a strategy combining enantioselective organocatalysis with central-to-axial conversion of chirality to prepare enantioenriched 4-arylpyridine atropisomers.[5] We now wish to disclose our on-going efforts in the application of this strategy to the synthesis of more complex targets such as DPP4 inhibitors and the natural product streptonigrin.



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Résumés des posters

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P10	Ali Soulieman	Synthesis of new gamma-lactams carrying *gem*-difluoroalkyl side chains.
P11	Laurent Evanno	Total synthesis and isolation of xerocomic acid dimers from *Scleroderma citrinum.*
P12	Victor Turpin	Exploration of mechanisms into the indole monoterpene alkaloids biosynthesis from geissoschizine.
P13	Jean-Pierre Hurvois	Total Synthesis of Tylophorine by alkylation of an α-aminonitrile.
P14	Manon Dupuis	New Synthetic Methods for the Preparation of Saturated Heterocycles
P15	Julien Braire	Allylboration of isatins catalysed by BINOL-derivatives. A tool for synthesis of natural products.
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P17	Meng Liu	Total synthesis of peloruside A analogues.
P18	Abdelrahman Hamdi	Aza-Aromatic Building-Blocks for Multi- Step Synthesis: Practical Access to Vinyloxy and Allyl (*iso*)-Quinolines.
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P20	Vincent Coeffard	Light, Oxygen and Photosensitizer: an Efficient Cocktail for the Synthesis of Functionalized Furans

Stereodivergent approach in the protected glycals synthesis of L-vancosamine, L-saccharosamine, L-daunosamine and L-ristosamine involving a ring- closing metathesis step

Pierre-Antoine Nocquet,[1] Aurélie Macé,[1] Frédéric Legros,[2] Jacques Lebreton,[3] Gilles Dujardin,[2] Sylvain Collet,[3] Arnaud Martel,[2] Bertrand Carboni[1] and François Carreaux[1]

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Abstract :

Several classes of medicinally useful molecules with antibiotic and anti-cancer activities contain 3-amino-2-deoxy sugars.[1] For instance, *N*,*N*-dimethyl L-vancosamine is an essential component of pluramycin antibiotics such as kidamycin and pluramycin A (**Figure 1**). The development of new asymmetric synthetic sequences is still of high interest to access functional and stereochemical diversity. Considering that the glycal scaffolds are versatile building blocks in the field of natural products synthesis,[2] we reported a new access to several chiral 3-amino glycals as precursors for glycosylated natural products from (-)-methyl-L-lactate as common starting material.[3] The stereodivergent strategy is based on the implementation of a ring-closing metathesis of vinyl ethers as key step (**Figure 2**).

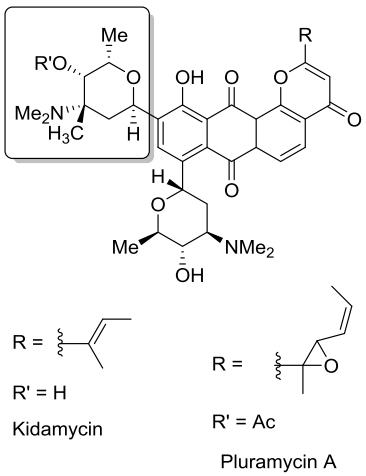

Figure 1

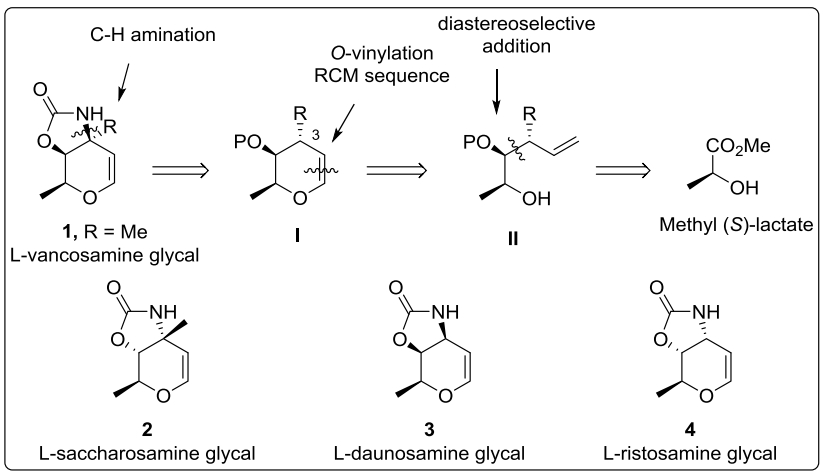

Figure 2

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Catalytic and stereodivergent access to complex 1,3-aminoalcohols

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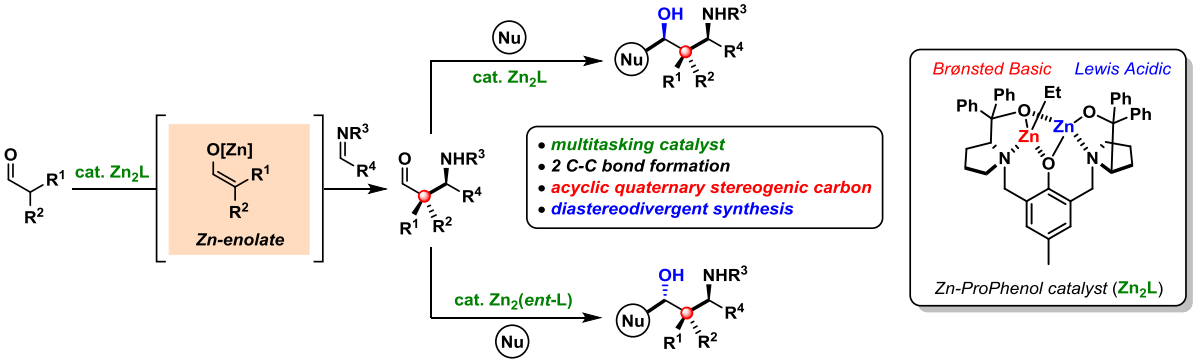
Abstract :

Herein, we report the use of branched aldehydes as versatile linchpins for various Zn-ProPhenol catalysed C-C bond-forming reactions to efficiently construct enantioenriched 1,3-aminoalcohols bearing an acyclic quaternary stereogenic center.[1] Importantly, this approach can be diastereodivergent by simply selecting the proper catalyst combination. Overall, this catalytic method directly transforms simple and readily available aldehydes into highly functionalized compounds and provides streamlined access to complex 1,3-aminoalcohols relevant to the synthesis of biologically important molecules.



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Organocatalyzed asymmetric halolactonization of acrylate-type benzoic acids. Total synthesis of (–)-herbaric acid.

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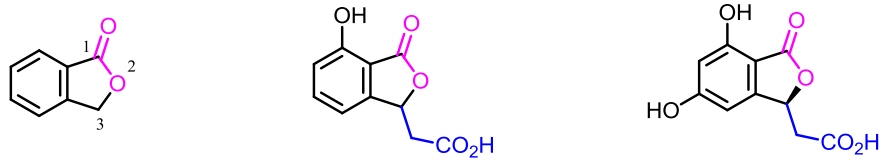
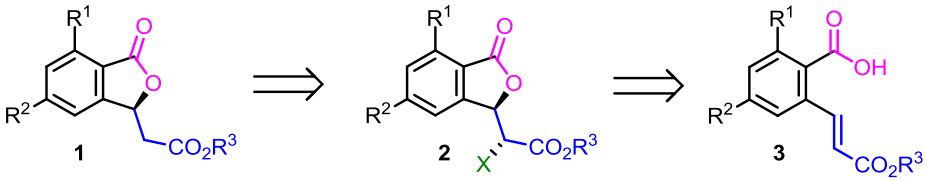
Résumé :

Isobenzofuranones bearing a carboxymethyl group at the 3 position of the lactone ring system such as isoochracinic acid or the fungal metabolite (–)-herbaric acid constitute a minor group of natural products which exhibits biological activities. The structural originality of these chiral functionalized isobenzofuranones makes them challenging synthetic targets, and the development of stereoselective methodologies for their synthesis constitutes an area of current interest.[1]



The regioselective (5-exo-tet) organocatalyzed asymmetric halolactonization of benzoic acid **3** bearing a chiral acrylate group at the *ortho* position of the benzene ring furnished a wide array of halo-lactones **2** with good diastereoselectivity.[2] Finally, photoinduced radical dehalogenation of compounds **2** furnished the targeted enantioenriched isobenzofuranones **1**. This methodology was then successfully applied to the total synthesis of (-)-herbaric acid.



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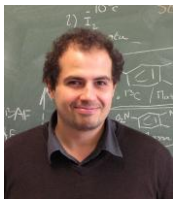
Total Synthesis of the Amphidinolide F



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Laboratoire de pharmacognosie, BioCIS, Faculté de Pharmacie, Université Paris-Saclay/CNRS, Chatenay-Malabry, 92290

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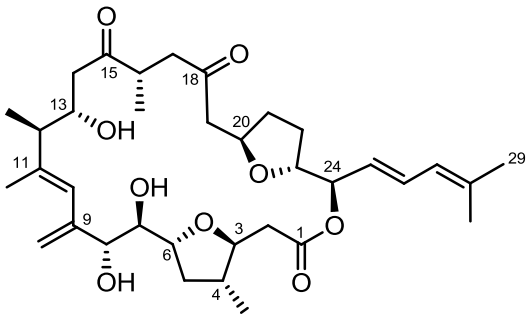
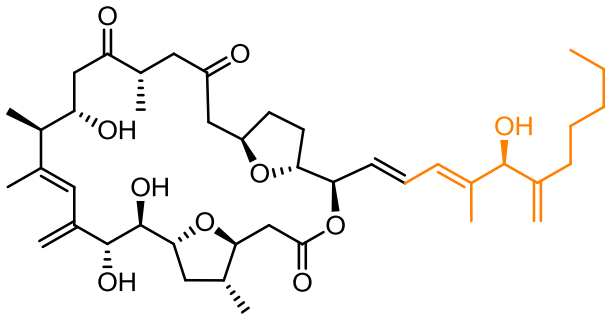
Abstract :

Nature is an outstanding source of inspiration for organic chemists. In particular oceans are a pool of original and biologicaly active natural products. Despite their complex structure, their high level of activity makes them very valuable, illustrated by some recent examples of marketed anti-cancer drugs which possess a marine natural product origin (trabectedin, epothilones, halichondrin B). Accordingly, amphidinolides[1] are a promising source of potent anti-cancer agents.

Among of all its congeners, Amphidinolide C and related brother Amphidinolide F retained specially our attention by their structures containing two tetrahydrofurans in a macrolactone core. Due to the difficult availability from their natural source, total synthesis of amphidinolides C and F are necessary to explore further the biological activities of these compounds. After some synthetic studies towards these two natural products,[2,3] we finally attained the total synthesis of amphidinolide F recently, through 23 steps considering the longest linear steps and 43 total steps.[4] The details of this major accomplishment will be discussed in the presented poster.



Amphidinolide F



Amphidinolide C

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Access to tricyclic fused 1,4-Benzodiazepin-5-ones from pipecolic derivatives.

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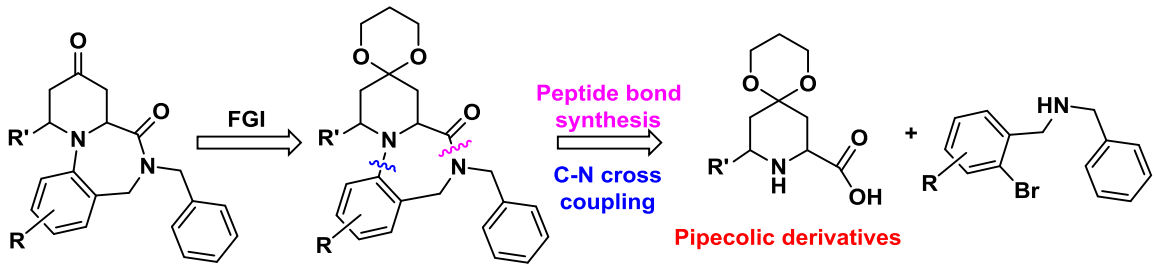
Abstract :

The 1,4-benzodiazepin-one scaffold is still regarded as a "privileged structure"[1] for drug discovery and development. Though, as a consequence, this skeleton is used as core of several pharmaceutically important families. It can be divided into three different sub-families, namely 1,4-diazepin-2-one, 1,4-diazepin-3-one and 1,4-diazepin-5-one. The literature showed that 1,4-diazepin-2-one and 1,4-diazepin-5-one types are well described but only few reports are devoted to 1,4-diazepin-3-one type compounds which has been used mainly as β-turn peptidomimetics. Moreover, fused polycyclic benzodiazepinones which have shown inhancement in their biological activities have promoted interest in such heterocyclic structures and consequently new structural analogs with new applications have appeared in the literature [2] We have recently reported chiral routes for the synthesis of 2,6-disubstituted piperidin-4-one[3] and its application to the synthesis of natural products. So, we can extend the methodology to chiral compounds which can be medicinally relevant heterocycles.



Inspired by these concepts, we will present a new series of fused heterocyclic 1,4-benzodiazepin-3-one in which the heterocyclic ring is a substituted piperidine based on a new strategy starting from pipecolic acid derivatives and substituted bromo di-benzylamines.

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"

BIOINSPIRED OXIDATIVE CYCLIZATION OF THE GEISSOSCHIZINE SKELETON FOR THE TOTAL SYNTHESIS OF EXCELSINIDINE AND MAVACURAN ALKALOIDS

Maxime Jarret,[1] Victor Turpin,[2] Aurélien Tap,[1] Cyrille Kouklovsky,[1] Erwan Poupon,[2] Laurent Evanno,[2] Guillaume Vincent[1]

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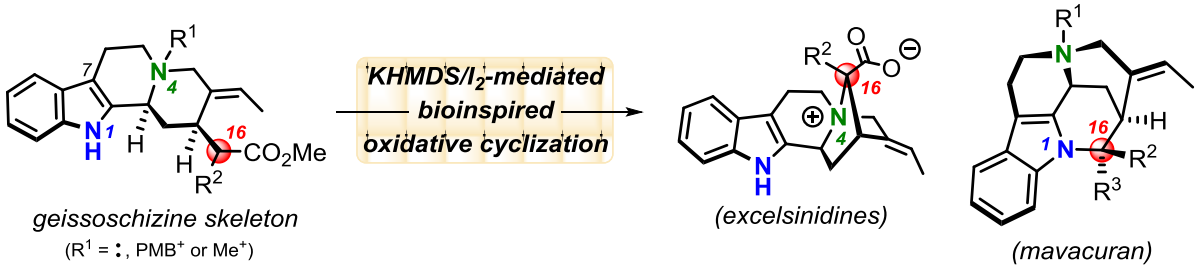
Abstract :

Geissochizine is considered as a common biosynthetic precursor of several families of monoterpene indole alkaloids *via* divergent oxidative cyclizations between the C16 carbon and the N1 nitrogen for mavacurans, the N4 nitrogen for excelsinidines or the C7 carbon for akuammilans. Inspired by this biosynthetic hypothesis, we sought to study the intrinsic selectivity of an intramolecular oxidative coupling directly from the complete geissoschizine skeleton.

Application of slightly modified Ma's oxidative coupling conditions (KHMDS/I_2) directly to synthetic geissoschizine led to a selective formation of the C16-N4 bond of the excelsinidines core and, consequently, to the completion of the first total synthesis of (−)-17-nor-excelsinidine.[1] To sequester the superior nucleophilicity of the N4 aliphatic nitrogen in comparison to the N1 indolic nitrogen and, moreover, to lock the *cis* conformation of the geissoschizine framework required to insure proximity between the indole nucleus and the C16 carbon, we decided to quaternarize the N4 nitrogen. To our delight, this strategy allowed us to observe the formation of the desired mavacuran skeleton *via* the long-expected formation of the N1-C16 bond and, therefore, to achieve the total synthesis of (+)-16-*epi*-pleiocarpamine, (+)-16-hydroxymethyl-pleiocarpamine and (+)-taberdivarine H as well as 16-formyl-pleiocarpamine, a postulated biosynthetic intermediate.[2]



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"

Synthesis and Functionalization of 3,5-Disubstituted 1,2-Dioxolanes: Toward the Total Synthesis of Mycangimycin

Alexis PINET, Bruno FIGADERE and Laurent FERRIE

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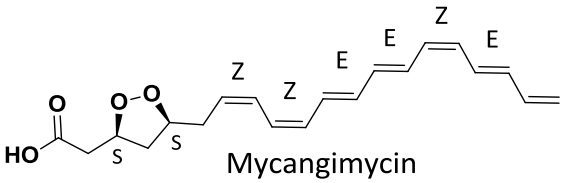
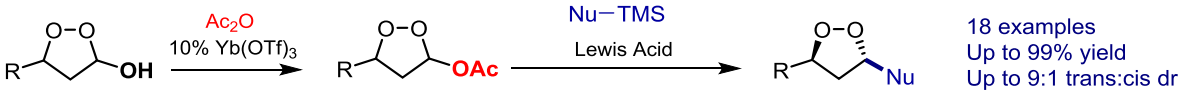
Abstract :



Among isolated and described natural products, endoperoxides species represent a small class of molecules. Assuredly, the most famous one is Artemisinin (Youyou Tu, 2015 Nobel Prize) which exhibits high therapeutic efficiency against paludism. This discovery allowed then endoperoxides to take its first steps as a therapeutic agent.

Mycangimycin have been isolated from *Streptomyces* by Clardy's team in 2009,[1],[2] revealing an uncommon mutualism and a very good antimalarial activity. This molecule is made of a long polyenic chain with seven conjugated double bonds and a rare 3,5-disubstituted 1,2-dioxolane ring, indeed, most of common endoperoxides (dioxolanes or dioxanes) exhibit tri- or tetra-substitution at their peroxide moiety.

Despite their potential in therapeutics as pharmacophores or as full-fledged molecules, the synthesis of endoperoxides have been scarcely studied. This is particularly the case for 3,5-disubstituted 1,2-dioxolanes, whose the synthesis and the mono-functionalization are hard to implement with actual methods.



R = alkyl, aryl, benzyl, Protected alcohols

Our strategy implies acetylation of a peroxyhemiacetal (from cyclopropanol ring expansion). The acetate is a good leaving group and allows in presence of a Lewis acid the generation of a peroxyoxocarbenium ion that can be attacked by various nucleophiles. This work constitutes the first steps for the total synthesis of mycangimycin[3], and/or therapeutically active synthetic endoperoxides.

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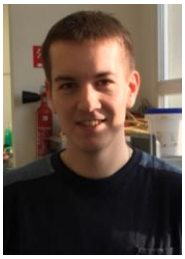
Total synthesis by DNA-templated photocatalysis



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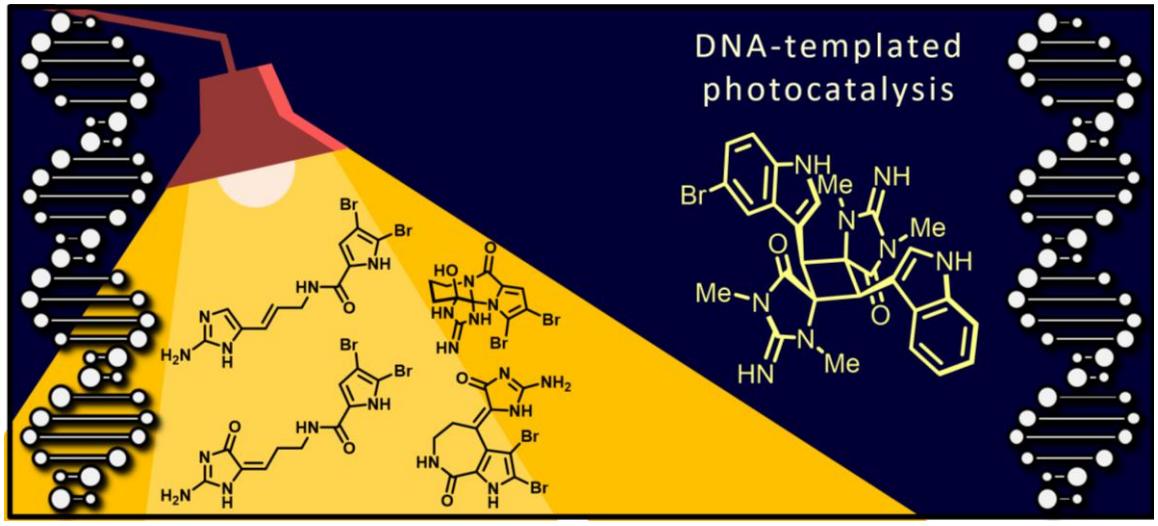
Light-induced reactions are widely occurring in biosynthetic processes. However, photochemical reactions remain underused mainly because of the formation of highly reactive intermediates.

Total synthesis of dictazole B, firstly achieved by a [2+2] bioinspired photo-cycloaddition under solvent-free conditions,[1)] was recently improved by using a DNA-templated photocatalytic process.[2)] The compartmentalization of monomeric aplysinopsins in DNA promotes self-organisation and ultimately the desired cycloaddition. This new procedure improved yields and made scale-up possible.

This first total synthesis combining DNA and photochemistry opens large developing prospects. The current studies aiming at applying this methodology to other substrates will be presented.



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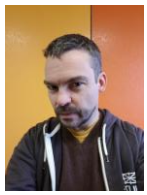
"UPSIDE DOWN" ACROLEIN IN A CHEMICAL INVESTIGATION OF MANZAMINE ALKALOIDS BIOSYNTHETIC PATHWAY



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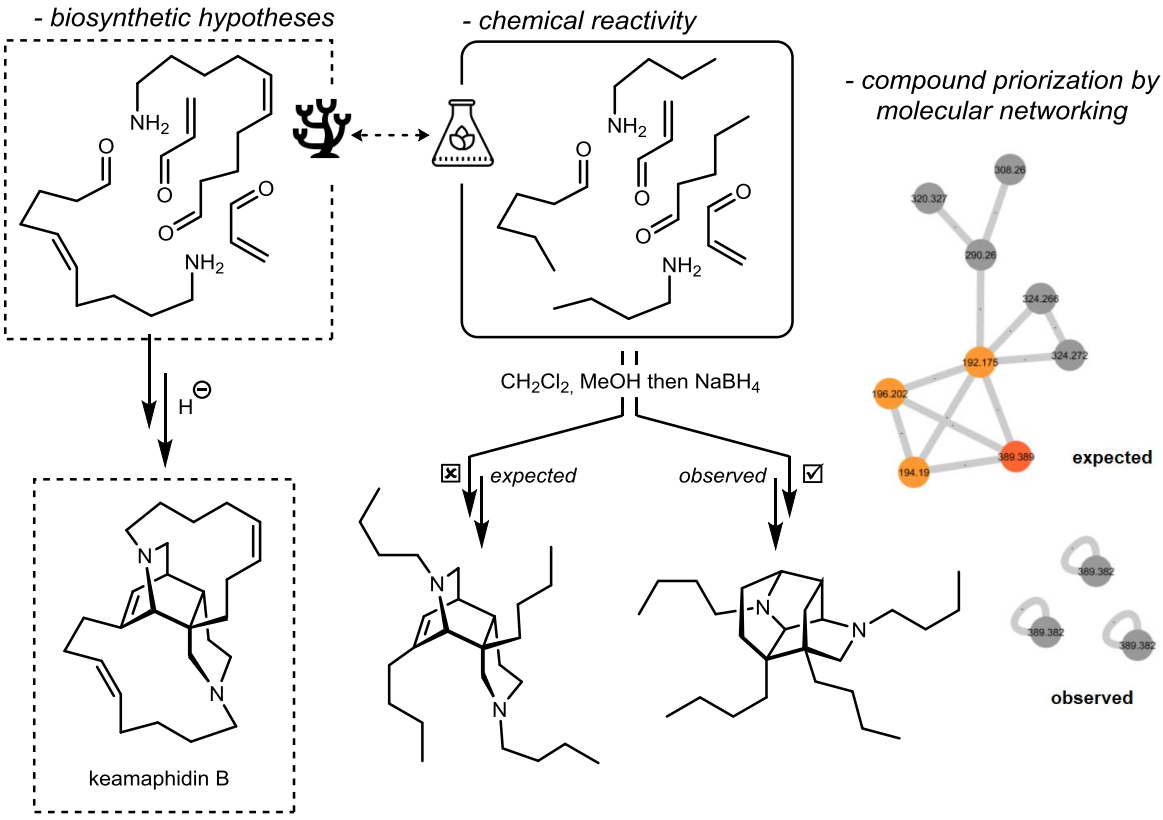
Manzamine-type alkaloids are one of the most fascinating class of marine alkaloids isolated along the years from different genera of sponges (*Haliclona*, *Xestospongia* etc.).[1] Different biosynthetic hypotheses by Baldwin and coll. and Marazano and coll. have been proposed to explain the rise of complexity from simple units derived from fatty acid metabolism.[2] Especially, small reactive units such as acrolein or malonaldehyde have been put forward.

We wish to confront biosynthetic hypotheses with chemical reactivity by studying complex cascade reactions in promiscuous biomimetic models as exemplified, on the figure, by keramaphidin B. The discovery (and biosynthetic consequences) of a caged alkaloid-like structure will be fully detailed in this communication. The help of bio-informatic tools (such as molecular networking) was crucial in this study.



1) B. Delpech, *The Saraine Alkaloids*, chap. 4 *in* The Alkaloids, vol 73, **2014**, Elsevier Inc.
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Synthesis of new gamma-lactams carrying *gem*-difluoroalkyl side chains

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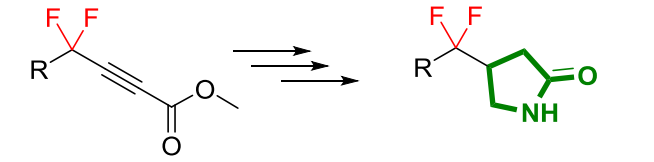
Abstract :

The γ-lactam ring, also known as γ-butyrolactam, is part of the core structure of a large number of natural and non-natural compounds covering a broad spectrum of biological activities[1]. On the other hand, it is well established that fluorine chemistry has a very strong impact nowadays in bioorganic and medicinal chemistry.[2] Therefore our research program focus on the synthesis of new gamma lactams with *gem*- difluorinated side chains. Our synthetic strategy takes advantage of the easily accessible and versatile *gem*-difluoro propargylic derivatives.[3] The synthesis of our target molecules will be presented in details in this poster.



Gem difluorinated propargylic esters

Gem difluorinated γ - lactams analogues

References :

1) For a recent review, see: Caruano, J.; Muccioli, G.; Robiette, R. *Org. & Biomol. Chem.* **2016**, *14*, 10134-10156 and references cited therein.
2) For a recent review, see: Meanwell, N. A. *J. Med. Chem.* **2018**, *61*, 5822-5880 and references cited therein.
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Total synthesis and isolation of xerocomic acid dimers from *Scleroderma citrinum*

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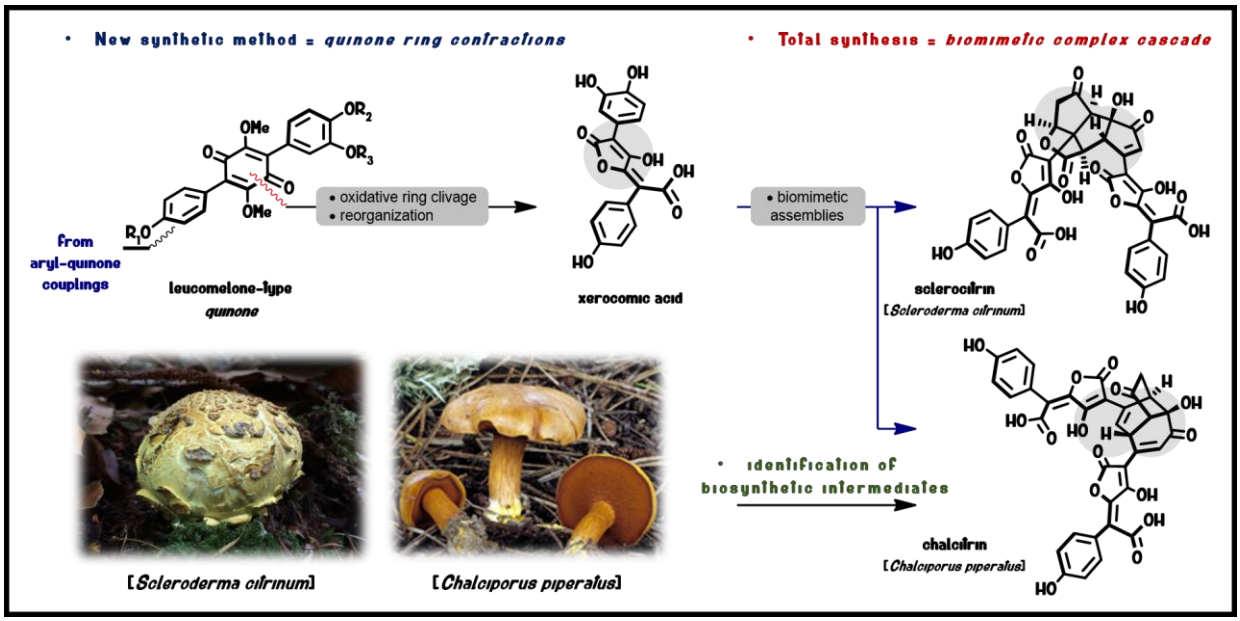
Fungus phyla produce a large variety of pigments dominated by biosynthetic related diphenyl-quinones and tetronic acids. Among others, two unusual and highly complex dimers, sclerocitrin and chalcitrin were isolated from two European mushrooms:[1] the common earthball and the peppery bolete. The two molecules originate from the dimerization of xerocomic acid and biosynthetic hypothesis has been emitted based on a complex cascade of skeletal reorganization. The project of total synthesis toward sclerocitrin and chalcitrin is based on a full biomimetic strategy *via* an oxidative dimerization of xerocomic acid. In addition, isolation of dimer structures from *Scleroderma citrinum* is ongoing to identify new complex structures and intermediates to afford a final validation of the biosynthetic hypothesis.



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Exploration of mechanisms into the indole monoterpene alkaloids biosynthesis from geissoschizine.

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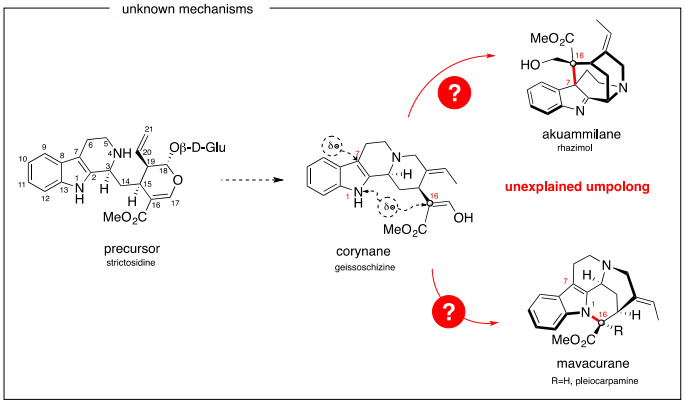
Monoterpene indole alkaloids are a family of alkaloids all derived from a single precursor: strictosidine[1,2] Thousands of compounds belonging to this family have been isolated and characterized since the 1950s. Although the biosynthetic pathways linking these highly complex carbon skeletons appear to be established, many of these mechanisms are not known to date.

At a very early stage of the biosynthesis of these alkaloids, geissoschizine is formed from strictosidine. Geissoschizine is considered to be a precursor of akuammilane and mavacurane alkaloid types *via* oxidative couplings whose exact nature is still not elucidated.

We will present our results on the cyclization that can undergo geissoschizine under oxidative conditions. These results will be supported by an *in silico* (DFT) approach to elucidate the early stages of the monoterpene indole alkaloids biosynthesis.



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Total Synthesis of Tylophorine by alkylation of an α-aminonitrile

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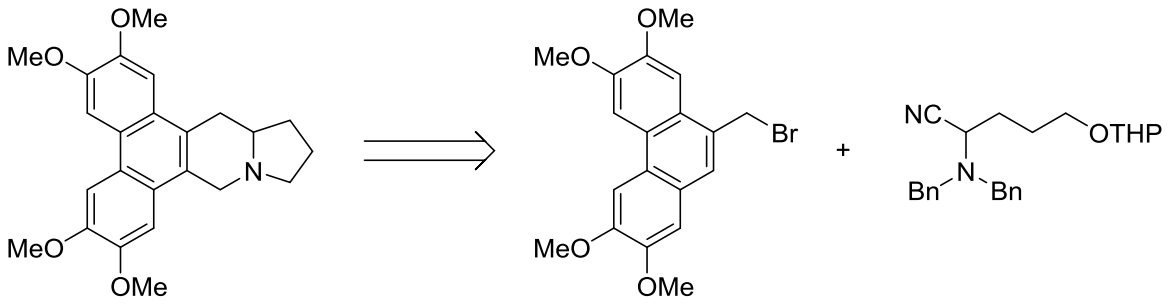
Abstract :

The phenanthroindolizidines alkaloids are a small family of compounds which have been isolated by Rathnagiriswaran *et al*.[1] from a vine *Tylophora indica* native from subtropical India, Asia and Australia. The leaves of this plant have been utilized in the folklore medicine to treat asthma, as well as bronchitis and dysentery in India. The isolation of the major alkaloid, tylophorine, incited chemist to elaborate different synthetic routes aimed at the total synthesis of the title alkaloid or structurally related analogues.[2] These approaches are based on various methodologies such as: Friedel-Crafts condensation, Stevens or Overman rearrangements, Sonogashira type coupling. In this contribution we which to present a new convergent approach based on the condensation of bromide 1 and α-aminonitrile **2** to construct the C14-C13a bond or the future alkaloid. The synthesis of the phenanthrene moiety as well as the elaboration of α-aminonitrile **2**, will be discussed during the poster presentation



rac-Tylophorine **1** **2**

Scheme 1. Retrosynthetic analysis of *rac*-Tylophorine from α-aminonitrile 2.

References :

1) Rathnagiriswaran, A.N; Ventakachalam, K.; *Indian J. Med. Res.*, **1935**, *22*, 433. 2) For a general review, see: Chemler, S. R. *Curr. Bioact. Compd*. **2009**, *5*, 2.

New Synthetic Methods for the Preparation of Saturated Heterocycles

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Abstract :

Five- and six-membered ring saturated heterocycles are key fragments that can be found in numerous compounds of natural sources, presenting potent biological activities. For instance tetrahydrofurans (THFs) and tetrahydropyrans (THPs) are recurrent motifs present in the skeleton of very complex compounds such as those depicted in Figure 1. The complexity of these structures is highlighted by the presence of substituents at the different possible positions of the ring *and* by the relative configuration of the stereogenic centres. Several efficient synthetic methods have been reported for the preparation of tetrahydrofurans[1,2] and tetrahydropyrans.[3,4] However, none of these reported methods are really general when both the substitution pattern and the relative configuration are taken into account and, as a result, the **development of new synthetic strategies to access this important class of compounds** from readily available starting materials is still **highly relevant**.

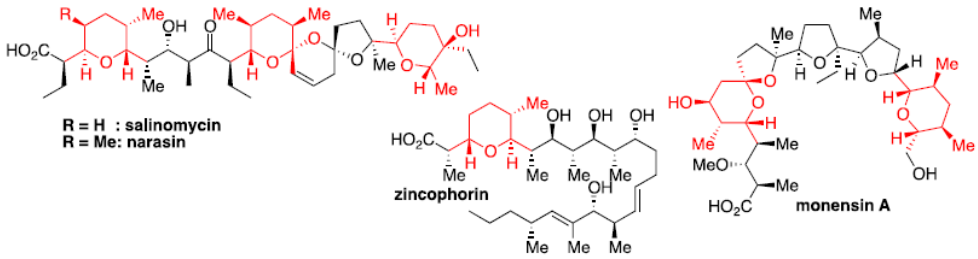

Figure 1 : Selected examples of naturally-occuring poly-oxygenated molecules THP and THP rings

The project presented here aims at developing novel and efficient synthetic methodologies to provide access to polysubstituted-THP with good control of the stereocentres. This methodology will be illustrated with several substrates previously synthetized. Our recent results in this field will be discussed.

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Allylboration of isatins catalysed by BINOL-derivatives. A tool for synthesis of natural products

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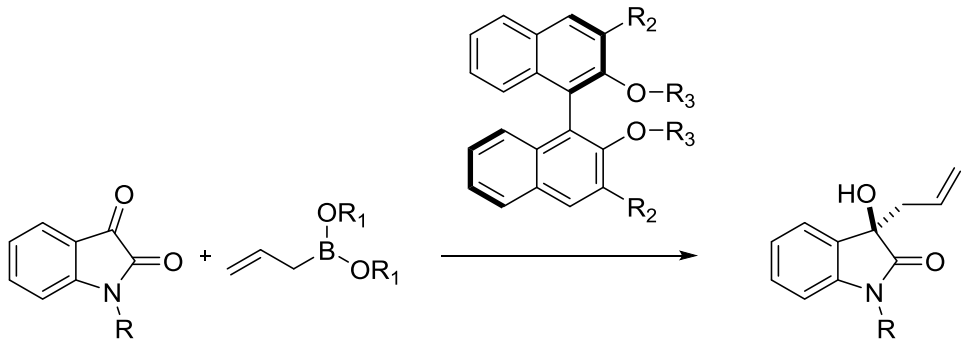
Abstract :

The 3-Substituted-3-hydroxyoxindoles core is present in a lot of natural products, drugs and bioactive compounds. Especially, 3-allyl- 3-hydroxyoxindole is an interesting building block for the synthesis of natural products such as Madindoline, CPC-1, Convulotamydine[1]. Because of these potential applications, the catalytic building of its quaternary chiral center is currently a target in organic synthesis. In the past few years, some examples of enantioselective allylation of isatins have been described mainly by using metal in the process. The allylboration reaction is an efficient tool to avoid metal by using organocatalyst. Actually, there is only one example of enantioselective allylboration of isatin by using chiral amino-phenol[2] but no process using commercial organocatalysts such as BINOL-derivatives. In this contribution, we report our investigation and recent advancement about the allylboration of isatins catalyzed by a chiral BINOL-derived catalysis.



Scheme: Enantioselective formation of 3-allyl-hydroxyoxondole

References :

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Enantioselective total synthesis of *Haliclona* alkaloids and fluvirucinin B_1

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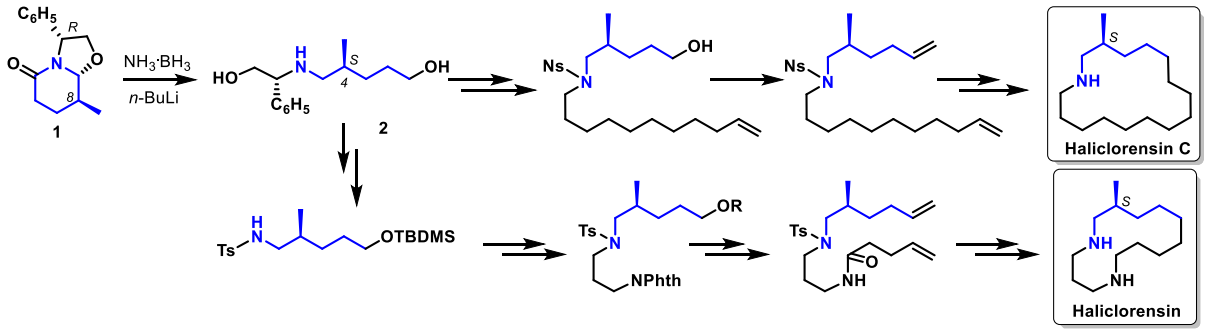
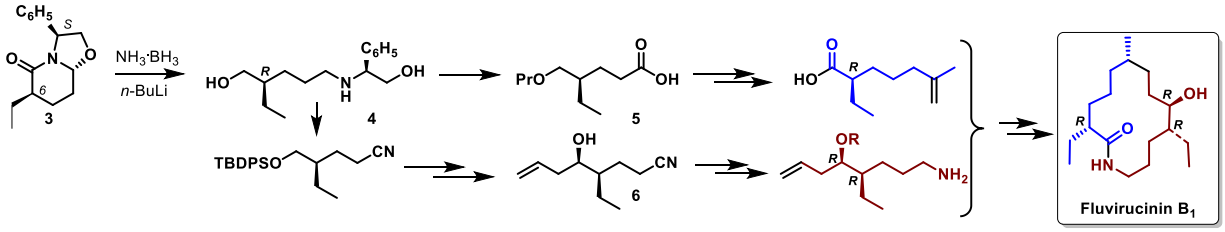
Abstract :
$LiNH_2BH_3$-promoted reductive opening of 8-substituted (*R*)-phenylglycinol-derived lactam **1** leads to enantiopure 4-substituted-5-aminopentanol **2**, which is used as the starting building block in the synthesis of the *Haliclona* alkaloids haliclorensin C and haliclorensin[2]. The synthesis involves the conversion of **2** into appropriate long-chain secondary amino derivatives bearing two terminal alkene functionalities, and a final ring-closing metathesis reaction.



The synthetic usefulness of substituted 5-aminopentanols as building blocks is also illustrated by the convergent synthesis of fluvirucinin B_1[3]. Key steps of the synthesis are the reductive $LiNH_2BH_3$-promoted double ring opening of 6-substituted (*S*)-phenylglycinol-derived lactam **3**, an organocopper coupling, a stereoselective allylation, a ring-closing metathesis reaction, and a stereoselective hydrogenation.



References and notes

1) Present address: Laboratoire de Recherche CEISAM, 2 Chemin de la Houssinière 44300 Nantes.
2) a) Amat, M.; Guignard, G.; Llor, N.; Bosch, J. *J. Org. Chem*. **2014**, *79*, 2792-2802. b) Guignard, G.; Llor, N.; Urbina, A.; Bosch, J.; Amat, M. *Eur. J. Org. Chem*. **2016**, 693-703.
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TOTAL SYNTHESIS OF PELORUSIDE A ANALOGUES

Meng Liu,[a] Ismael Rabouel,[a] Anne-Caroline Chany,[a] Frédéric Legros,[a] Héloua Haroun,[a] Monique Mathé-Allainmat,[b] Jacques Lebreton,[b] David Marchand,[b] Jérôme Graton,[b] Jean-Yves Le Questel,[b] Pascal Gosselin,[a] Gilles Dujardin,[a] Catherine Gaulon-Nourry[a]

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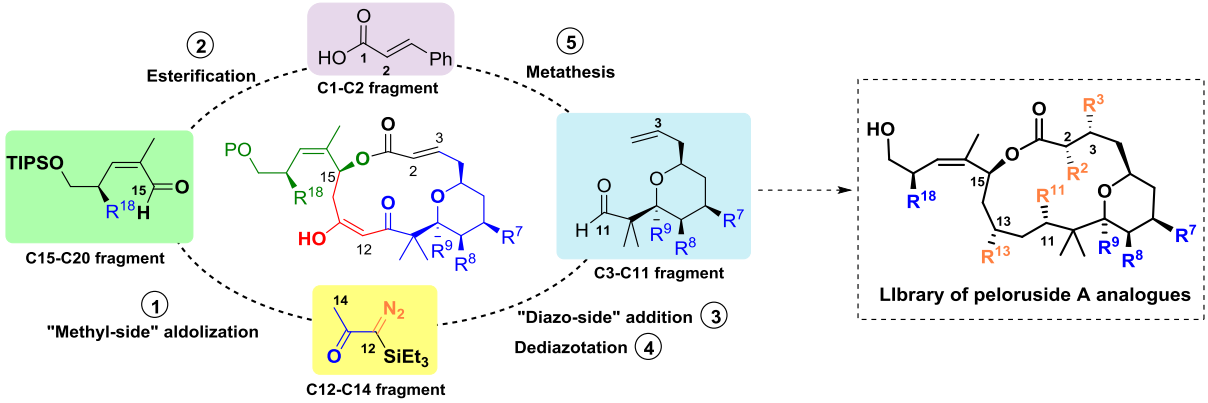
Abstract :

Sixteen-membered macrolide peloruside A is a potent microtubule stabilizing agent, extracted in 2000 from the New Zealand marine sponge *Mycale hentscheli*.[1] We recently achieved a convergent and rapid synthesis of original C2,C3-unsaturated, C11,C13-keto-enol macrocycles with peloruside A skeleton.[1] The four-fragment strategy implemented features two aldol-type couplings with the central C12-C14 building-block TES-diazoacetone and a late-stage ring-closing metathesis. These original unsaturated macrocycles constitute synthetic platforms to access peloruside A analogues, as both the C11,C13 keto-enol moiety and the C2,C3 double bond constitute an attractive gateway to diversity on the macrolactone skeleton. Modulation of substituents R^7, R^8 and R^9 is currently investigated through the synthesis of original C3-C11 fragments, while docking studies are underway to help the rational design of new analogues.



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Aza-Aromatic Building-Blocks for Multi-Step Synthesis: Practical Access to Vinyloxy and Allyl (*iso*)-Quinolines

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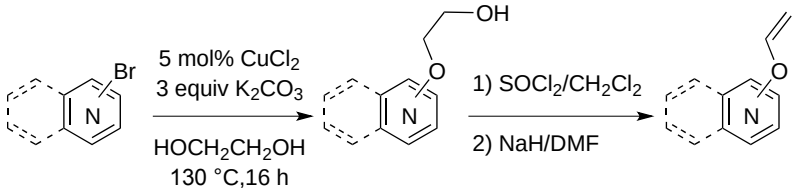
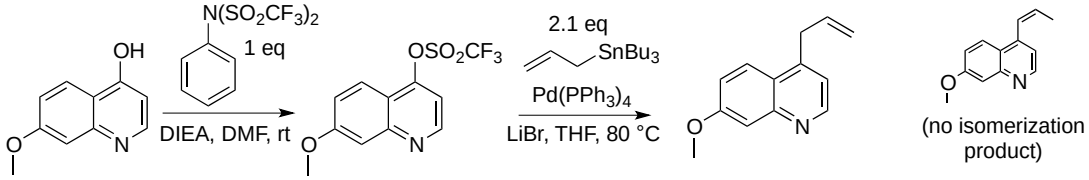
Abstract :

Vinyloxy and allyl quinolines and isoquinolines are interesting intermediates in a wide variety of reactions (e.g. cyclopropanation, cycloaddition[1] and metathesis processes) as well as in the production of new biologically active molecules.[2] However, general procedures still lack for their practical preparation.

Indeed, while the preparation of phenol-derived vinyl ethers has been extensively studied, the preparation of aza-aryl vinyl ethers has been far less developed. For instance, the only method available for the synthesis of 4-vinyloxy pyridines or quinolines is the *O*-vinylation using the Reppe chemistry, a method which requires severe conditions (strong base, high pressure of acetylene and temperature) and specific apparatus.[3] We succeeded to prepare such vinyl ethers *via* a more simple three-step sequence from the corresponding N-heteroaryl bromides.[4]



Concerning the allylation of quinolines at unreported positions, we carried out Stille cross-coupling conditions allowing access to allylquinolines from the corresponding triflates, without any sort of isomerization.



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TOWARDS THE TOTAL SYNTHESIS OF CHAXALACTINS

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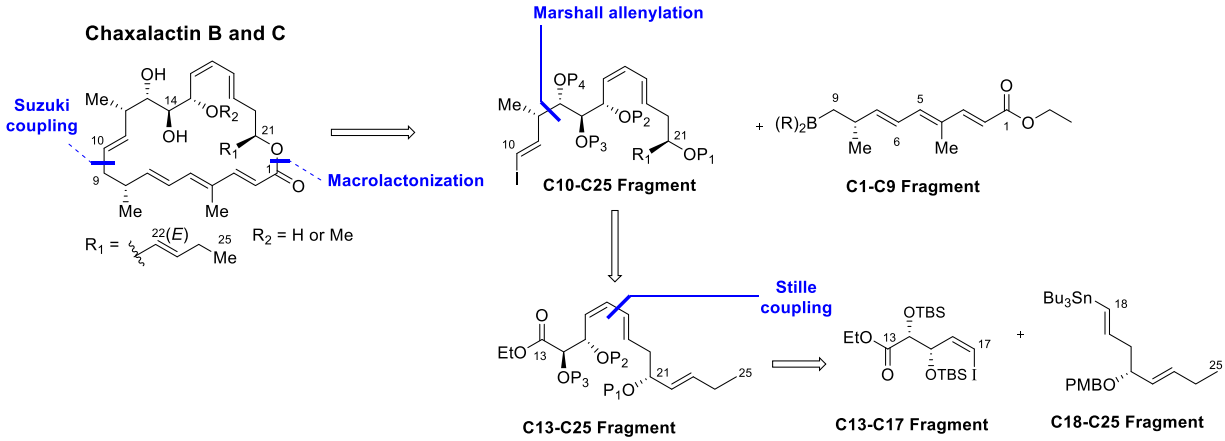
Abstract :

Chaxalactins A, B and C are 22-membered macrolactones isolated in 2011 from a strain called *Streptomyces sp. C34*, collected in hyper-arid Atacama Desert (North of Chili).[1] The complex structure of these molecules coupled with their interesting antibiotic and potential antitumoral activities make this family of molecules synthetically challenging important targets. Despite of their interest, no total synthesis of these compounds has been reported so far.
The aim of this project is to synthesise for the first time chaxalactins A, B and C and their analogues. Chaxalactins could be obtained by a Suzuki coupling between the C1-C9 and C10-C25 fragments. The C10-C25 fragment could be prepared from the C13-C25 fragment using a key step of Marshall allenylation to introduce and control the C12, C13 stereocenters (Scheme 1). The C13-C25 fragment could in turn be obtained by a Stille coupling between the C13-C17 and C18-C25 fragments.



Scheme 1: Retrosynthetic analysis of chaxalactins B and C

The synthesis of the C1-C9 fragment and of the C10-C25 fragment will be presented in this poster.

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P20

Light, Oxygen and Photosensitizer: an Efficient Cocktail for the Synthesis of Functionalized Furans

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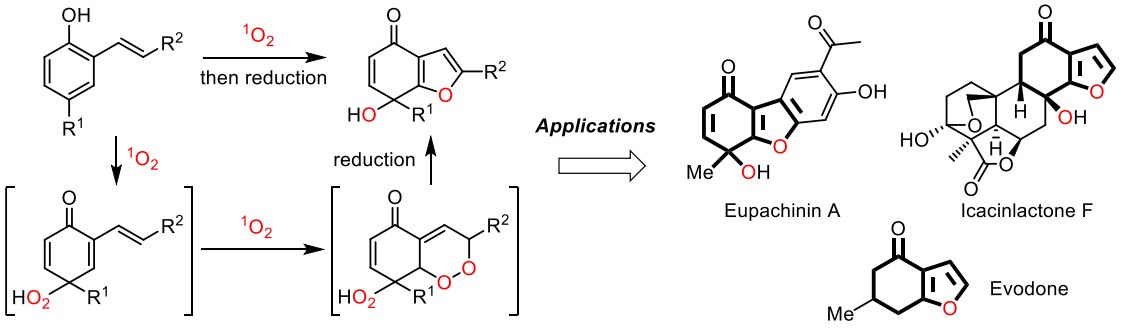
Abstract :

Photosensitized oxygenation is a convenient process to introduce oxygen atoms into organic architectures. This strategy requires the combination of light, oxygen and a photosensitizer to produce highly reactive oxygen species (ROS) such as singlet oxygen ($^1O_2$).[1] Singlet oxygen is an excited state of molecular oxygen and its high reactivity towards electron-rich substrates has been harnessed in various synthetic transformations.[2] Over the past decades, there has been a growing interest in domino reactions which can produce an important increase of molecular complexity. As part of a program focusing on the one-pot preparation of polycyclic architectures,[3] this communication will present our recent research on the synthesis of functionalized fused furans via a BODIPY-catalyzed domino photooxygenation.[4]



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UNIVERSITÉ DE NANTES

Le Mans Université

9ème Symposium Francophone de Synthèse Totale

Nantes, 23 et 24 mai 2019

Société Chimique de France

Programme

Jeudi 23 mai 2019

13h00-13h50	**Accueil des participants**
13h50	**Mot d'ouverture**
14h00-15h00	**CP1: Pr. Janick Ardisson**
15h00 - 15h30	**CO1: Dr. Philippe Peixoto**
15h30-16h00	**CO2: Pr. Vincent Dalla**
16h00-17h00	**Pause-Café - Posters**
17h00-17h30	**CO3: Pr. Véronique Bellosta**
17h30-18h00	**CO4: Dr. Aurélien De la Torre**
18h00	**Clôture de la 1ère journée**

Vendredi 24 mai 2019

8h30-9h30	**CP2: Pr. Jonathan Burton**
9h30-10h00	**CO5: Dr. Laurence Miesch**
10h00-10h30	**CO6: Dr. Andrei Corbu**
10h30-11h00	**Pause-Café - Posters et exposants**
11h00-11h30	**CO7: Dr. Sandrine Py**
11h30-12h00	**CO8: Dr. Zacharias Amara**
12h00-12h30	**CO9: Dr. Xavier Bugaut**
12h30-14h00	**Buffet - Posters et exposants**
14h00-15h00	**CP3: Pr. Antonio Echavarren**
15h00	**Clôture du Symposium et remise du prix poster**