PALLADIUM-CATALYSED COUPLING OF OXANORBORNENES AND TERTIARY PROPARGYLIC CARBOXYLATES: A SIMPLE ACCESS TO ALLENYLIDENEYCLOPROPANES

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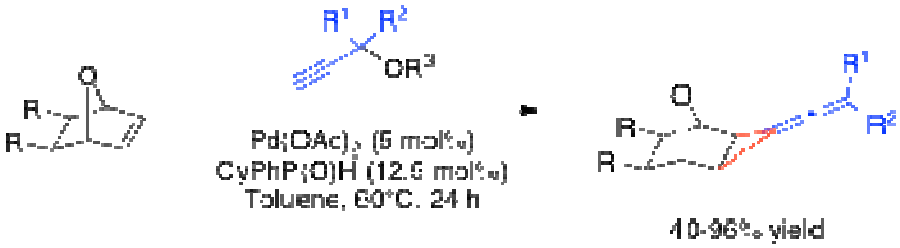
Secondary phosphine oxides (SPOs) and more particularly their P(III) form, phosphinous acids, have recently attracted much attention due to their ability to coordinate various transition metals giving them interesting catalytic properties.[1] Over the last decade, our group has been involved in the synthesis of SPOs as well as their application in coordination chemistry and in homogeneous catalysis.[2] For example, we have developed palladium-mediated tandem [2+1] cycloaddition/ring expansion sequence of norbornene derivatives with tertiary propargylic acetates giving rise to bicyclo[3.2.1]octadiene compounds.[3] In order to develop a methodology giving a straightforward access to functionalized seven-membered carbocycles, we intended to extend this catalytic transformation to oxanorbornene derivatives. Surprisingly, whereas the expected product was isolated in low yields for optimized reaction conditions, allenylidenecyclopropane cycloadducts were found to be the major or unique product. Herein, we will describe this new palladium-catalysed [2+1] cycloaddition that was investigated for various oxanorbornenes and tertiary propargylic carboxylates. A series of allenylidenecyclopropanes was obtained in good yields.



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PHOSPHORUS REAGENTS, A COLORFUL PALETTE FOR ORGANIC SYNTHESIS AND NEW BIOMOLECULES

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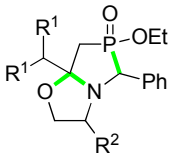
Modern organic synthesis requires the development of highly chemo- and stereoselective methods. Often mild and environmentally friendly, domino reactions are constituting a powerful tool shortening synthesis and substantially decreasing the wastes. In this context, starting from underused phosphinyl allenes, we have developed an effective synthesis of phosphorus heterocycles **1** through a hydrophosphination / isomerisation / Michael / Michael sequence. In another way, understanding the role played by trivalent nucleophilic phosphorus reagents allowed the formation of highly functionalized phosphine oxides through a four-component reaction or heterocycles by an organocatalyzed domino process.



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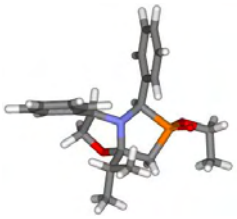
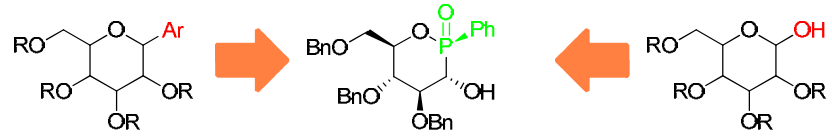


Besides the interest they may show in organic synthesis, organophosphorus compounds also exhibit special characteristics that make them unique in the field of biomolecules. Then, the phosphinolactone group can be regarded as a surrogate of lactol and could present an opportunity for the medicinal chemists to explore uncovered regions of chemical space. In addition to its isosteric relationship with the lactol group, phosphinolactone is an outstanding hydrogen bond acceptor and metal complexing agent which makes it an attractive structural motif for drug discovery.

Recently, we developed two different families of heterocycles which present the 1,2-oxaphosphinane **2** or the 1,4,2-oxazaphosphinane heterocyclic cores. The antiproliferative properties of oxaphosphinanes **2** were determined on C6 glial cells confirming their glycoside-like analogy.



C-Aryl-glycosides **3** Oxaphosphinane **2** Pyranosides **4**

The chemistry of the organophosphorus compounds may bring new developments either in organic synthesis or for dedicated applications.

Friday Afternoon

BOLTE Benoît

Gold(I) and Brønsted acid-catalyzed diastereocontrolled construction of variously substituted tetrahydropyrans

CHAMAS Zein el abidine

Synthesis of pentacyclic heterocycles through a regio- and diastereoselective cascade process

MAOUGAL Esma

Synthesis of analogue of AZT with triazole moiety

PESSET Bénédicte

TonB machinery: a new bacterial target for peptidic antibiotics

MONTEL Sonia

Synthesis of new fosmidomycin analogues as potential inhibitors of 1-deoxy-D-xylulose-5-phosphate reductoisomerase

PAGOAGA Bernard

Synthesis of new perylene derivates and applications in organic

AOUN Sameh

Synthesis of new therapeutic agents targeting the bone system

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Dr. COUDRET Christophe

Chemical dynamics, photochromism, nanosciences

GOLD(I) AND BRØNSTED ACID-CATALYZED DIASTEREOCONTROLLED CONSTRUCTION OF VARIOUSLY SUBSTITUTED TETRAHYDROPYRANS

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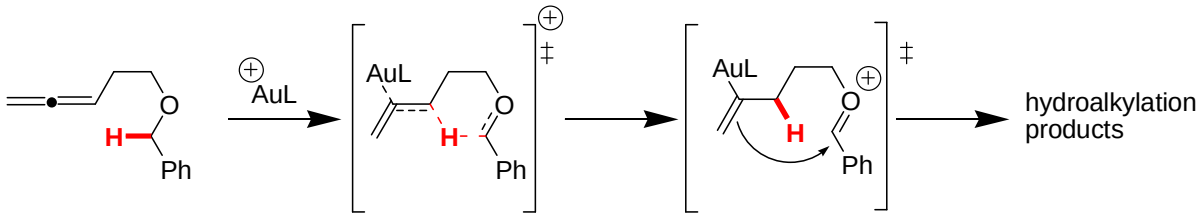
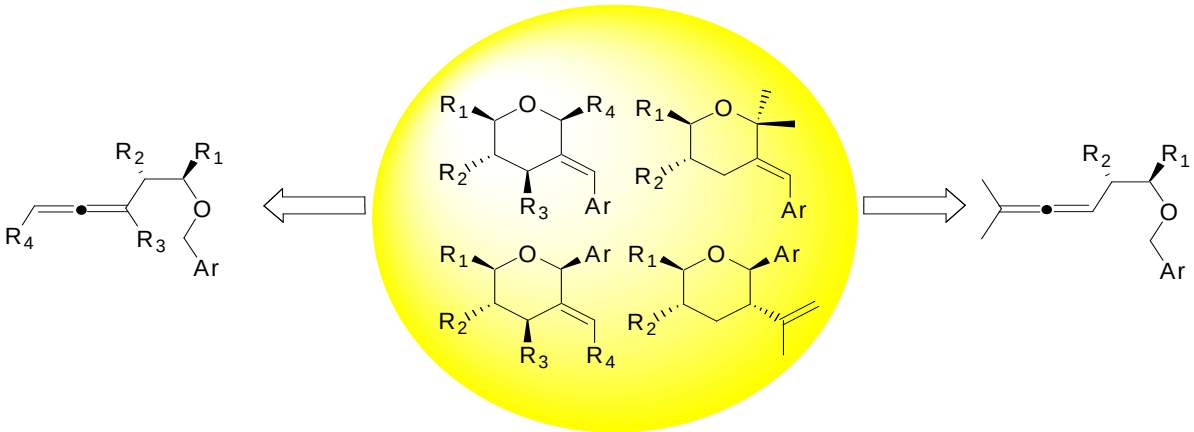
Allenes are valuable substrates or intermediates for the preparation of numerous motifs in organic synthesis. Gold (I) complexes have proved their ability to promote hydride shifts onto π-systems, and their efficiency to perform a reverse polarisation hydrofunctionalizations of allenes.
Benzyl ethers can be used as hydride donors to induce 1,5-hydride shifts. The resulting oxonium species can be trapped to furnish various cyclized products.



Depending on the degree of subsitution of the allene, the nature of the catalyst and the reaction time, an hydride transfer/cyclization sequence was observed, leading to variously substituted tetrahydropyrans. Several conditions have been developped in order to obtain the desired scaffold in excellent selectivities. The 6-membered ring transition state enables an excellent control of the newly-formed asymetric centers, which makes this transformation highly diastereoselective.



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SYNTHESIS OF PENTACYCLIC HETEROCYCLES THROUGH A REGIO-AND DIASTEREOSELECTIVE CASCADE PROCESS

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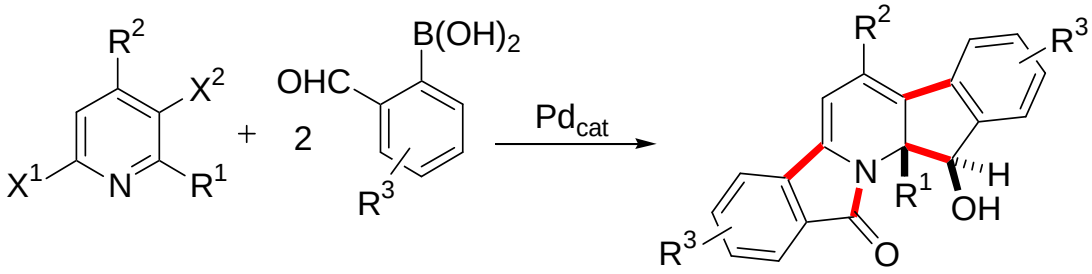
A new family of pentacyclic compounds incorporating a central 1,2-dihydropyridine core was obtained through a cascade reaction between 2-formylbenzene boronic acid and 2-halopyridines bearing additional reactive functions in 5-position. The cascade process was initiated by a palladium-catalyzed cross-coupling reaction and was followed by two successive nucleophilic cyclizations; the first cyclization performed on the pyridine nitrogen and the second occurred regioselectively on the adjacent carbon atom. Alternatively, when the reactive function in 5-position was an aldehyde, a cascade reaction starting from 2-chloro-5-bromopyridines and two equivalents of boronic acid was operating.



The crystal structures of two pentacyclic compounds were obtained and showed unambiguously the *trans* relationship between R and H. Overall, four new bonds and two stereocenters were produced during the cascade process and the observed regio- and diastereoselectivity were interpreted by Density Functional Theory calculations which evidenced the occurrence of an internal chelation in the transition state.

These new pentacyclic compounds show strong fluorescence emission which were quantitatively measured.

Z. Chamas, O. Dietz, E. Aubert, Y. Fort and V. Mamane *Org Biomol Chem* **8***(21):4815-8* (2010)

SYNTHESIS OF ANALOGUES OF AZT WITH TRIAZOLE MOIETY

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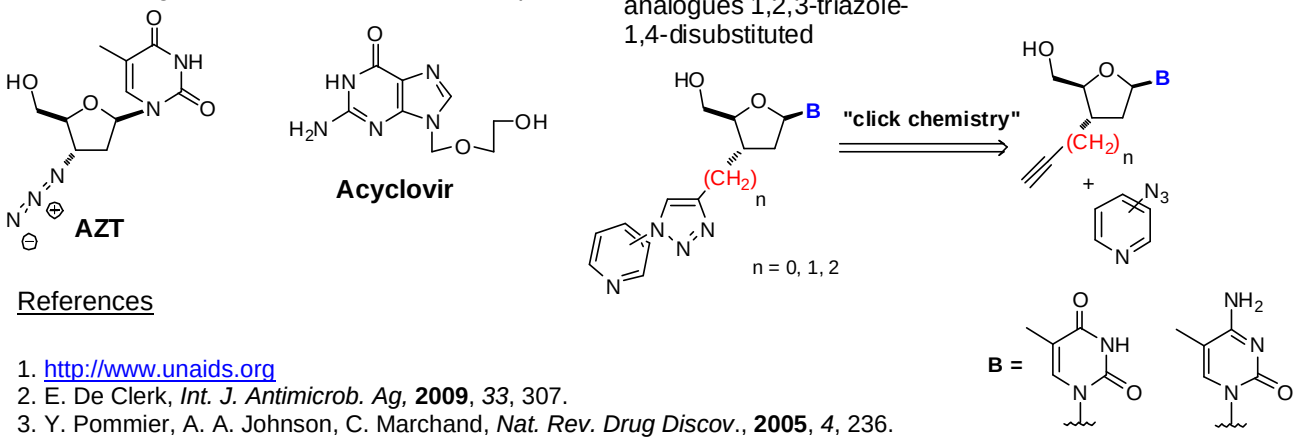
Currently**, AIDS** (Acquired Immune Deficiency Syndrome) is considered a pandemic, causing about 1.8 million deaths between 1981 (date on which the first case of AIDS was identified) and 2009.[1] AZT or 3'-azido-3'-deoxythymidine was the first antiretroviral approved for the treatment of AIDS [2]. This compound, due to the absence of the hydroxyl group in position 3', inhibits reverse transcriptase, and prevents the extension of the viral DNA chain and finally viral replication of the RNA.[3]

Herpes is a viral disease caused by both Herpes Simplex Virus type 1 (HSV-1) and type 2 (HSV-2). This pathogen, in its various forms, affects more than 90% of the population worldwide. Acyclovir or 9-[{2-hydroxyethoxy}-methyl]-guanosine (ACV) is an acyclic analogue of the natural nucleoside 2'-deoxyguanosine, considered as the first choice of treatment for herpes simplex virus types 1 and 2. ACV is also deprived of this 3'-OH function presents in the natural nucleosides, inhibits herpes DNA polymerase [4], and behaving as a "suicide inhibitor"

As part of the discovery of new molecules to complete the therapeutic arsenal directed to these two viral diseases, we are interested in the synthesis of a new family of antiretroviral of the AZT type with a 1,2,3-triazole-1,4-disubstituted moiety [5] (see Scheme below). These compounds were tested against HIV in collaboration with the group of Professor Raymond F. Schinazi (Emory University School of Medecine/Veterans Affair Medical Center, Atlanta, Georgia 30033, USA) and against herpes in collaboration with Professor Nathalie Bourgougnon (Université de Bretagne Sud, Laboratoire de Biotechnologie et Chimie Marines, Vannes).



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TonB MACHINERY: A NEW BACTERIAL TARGET FOR PEPTIDIC ANTIBIOTICS

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"It is absolutely crucial to identify new biological targets and develop new antimicrobial agents less prone to resistance. In this context, inhibitors of the TonB machinery are potential new antibacterial agents, without effects on the host."

Highly pathogenic Gram-negative bacteria such as *Yersinia pestis*, *Burkholderia mallei* and *Burkholderia pseudomallei* are listed as potential biological weapons on the battlefields or in the frame of bioterrorist attacks. Furthermore, antibiotic resistance is increasing dramatically: cases of resistance have now been reported for all antibacterial drugs, even those recently approved. Therefore, it is absolutely crucial to identify new biological targets and to develop new antimicrobial agents less prone to resistance. For this purpose, we focus our project on iron acquisition mechanisms in *Pseudomonas aeruginosa,* an opportunistic pathogen particularly resistant to most all the available antibiotics, and genetically close to the bacteria cited above.

In iron depleted medium, bacteria produce low molecular weight compounds able to chelate Fe(III), called siderophores.[1] Iron(III)-siderophore complexes are recognized by a specific outer membrane receptor before being actively transported into the periplasm. This translocation required the energy provided by a TonB-ExbB-ExbD protein complex embedded in the inner membrane, the TonB machinery. This energy transducer is involved as well in the uptake of others essential nutrients.[2] Moreover, sequence alignments of the periplasmic domain of TonB and various TonB-dependent transporters showed that the interaction domain is highly conserved. Last but not least, the bacterial proliferation is seriously affected when the TonB energy transducer is mutated.[3] Therefore inhibitors of this complex molecular machinery are potential antibacterial agents, without effects on the host since human beings do not express neither the target protein nor an homologous protein.

Our inhibition strategy consists in a direct disruption of the TonB protein / transporter interaction using molecules containing a peptidic moiety linked to a heterocyclic anchor, able to sequester TonB in the periplasm. The synthesis of our potential antibiotics will be presented, and their efficiency will be evaluated using various biological tests (MIC, iron uptake pathway inhibition, surface plasmon resonance, etc). In parallel we developed a siderophore-based vector able to address TonB inhibitors to a specific bacteria specie using the Trojan Horse prodrug strategy.[4]

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SYNTHESIS OF NEW FOSMIDOMYCIN ANALOGUES AS POTENTIAL INHIBITORS OF 1-DEOXY-D-XYLULOSE-5-PHOSPHATE REDUCTOISOMERASE

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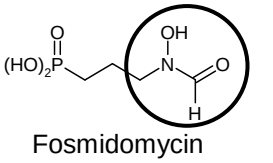
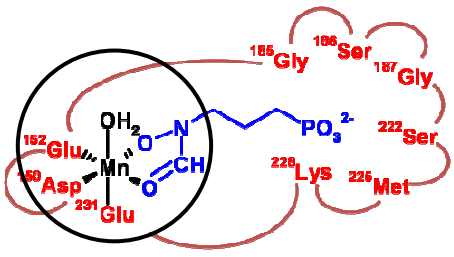
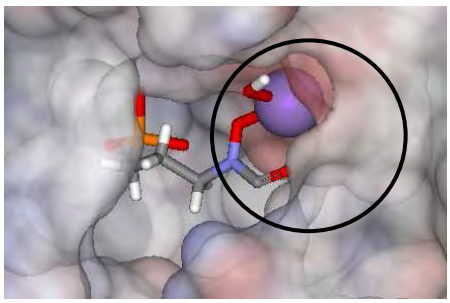
The non-mevalonate pathway is an alternative metabolic pathway leading to the formation of isopentenyl pyrophosphate. It is widely found in higher plants, protozoa or bacteria but interestingly has no equivalent in mammals.[1] Identifying a non-mevalonate pathway inhibitor would greatly contribute to the search for safer herbicides. The unique properties of 1-deoxy-*D*-xylulose 5-phosphate reductoisomerase (DXR) make it remarkable and a central target for drug design. The Fosmidomycin, a phosphonohydroxamic acid isolated from *Streptomyces lavendulae*, acts through the inhibition of DXR.[2]

Synthesis of Fosmidomycin analogues appears to be interesting in order to obtain new potential herbicides. We focused on the modifications of the complexing subunit which was sparsely studied to date. The X-Ray diffraction experiment of DXR co-cristallized with Fosmidomycin shows that in the enzyme site, the phosphonic group interacts with a highly specific and polar pocket. By contrast, the cation complexing unit involving the hydroxamic acid offers the possibilities of structural modifications or variations. Dedicated syntheses on this topic will be presented.



Fosmidomycin





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SYNTHESIS OF NEW PERYLENE DERIVATIVES AND APPLICATIONS IN ORGANIC

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Perylene bisimides are dyes studied for their spectroscopic properties and their ability to self-assemble. They can be used in protein labelling, single molecule spectroscopy, crystalline liquids, etc. They are also n-type semi-conductors and are focused in the field of organic electronics for one decade. Their air-stability, customization ability in several positions (bay, ortho, peri) and solubility in most organic solvents make them great candidates for integration in Organic-Field Effect Transistor[1].

In this goal we synthesized a large variety of perylene derivatives using the Suzuki-Miyaura coupling reaction on 1,6,7,12-tetrachloroperylene bisimides, 1,7-dibromoperylene bisimides and more uncommon 1,12-dichloroperylene bisimides (Fig. 1)[2]. These products have then been placed in devices (OFETs) so their mobility could have been determined.

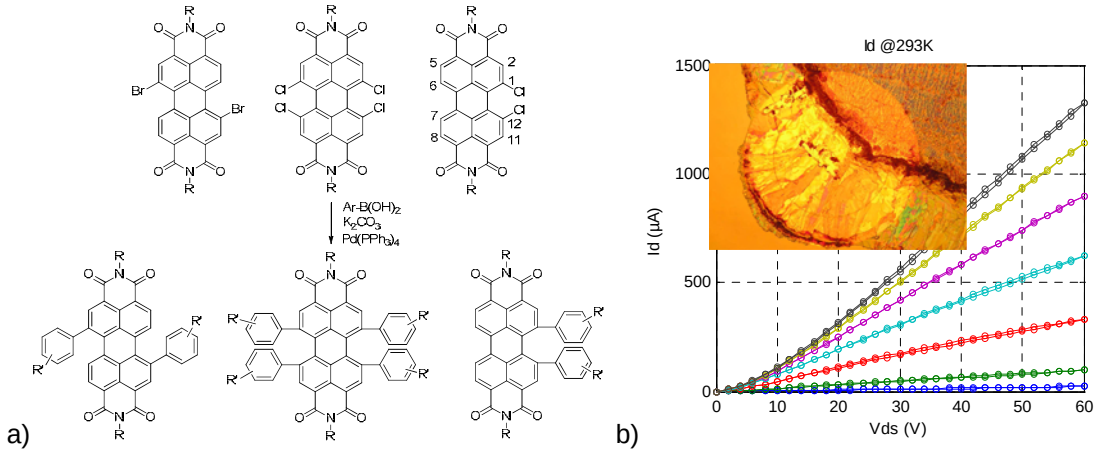

Fig. 1 : a) Suzuki-Miyaura coupling to obtain new perylene derivatives, b) Example of an electrical characterization measured on a commercial derivative and microscopic image of the crystals.

"Rylene diimides are a robust, versatile class of polycyclic aromatic electron-transport materials with excellent thermal and oxidative stability and high electron mobilities; they are, therefore, promising candidates for a variety of organic electronics applications"

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SYNTHESIS OF NEW THERAPEUTIC AGENTS TARGETING THE BONE SYSTEM

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The bone tissue is a connective tissue composed of a mineral part, made of calcium phosphate in the form of hydroxyapatite crystals (HA : $Ca_{10}(PO_4)_6(OH)_2$), and of an organic part containing an extracellular matrix and numerous specialized cells (osteoclasts, osteoblasts…) [1]. These different components are balanced and every malfunction leads to the appearance of specific pathologies such as osteoporosis which is a disease characterized by an important bone resorption.

Nowadays, different drugs are proposed to treat bone diseases such as zoledronic acid (or zoledronate, figure 1) which is the most efficient anti-resorptive commercialized agent. Its high activity on bone is particularly due to the presence of a Hydroxy-BisPhosphonic acid (HBP) function which strongly chelate hydroxyapatite crystals.

This efficient binding property of HBPs to the surface of bone makes them potential tools to target bone and deliver various drugs to it [2, 3]. Such a concept could be expressed by the design of "bifunctional molecules" where a HBP function is attached to a linker bearing a drug on the other end (figure 2). In the laboratory, we have recently developed this concept to potentially treat rheumatoid arthritis with attachment of NSAID (Non-Steroidal Anti-Inflammatory Drug) to the vector [4].

This communication will outline different synthetic routes to a family of "bifunctional molecules" with the application of a novel procedure to access to complex HBPs starting from carboxylic acid precursors [5, 6].

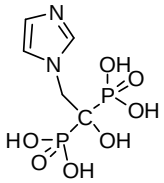

Figure 1

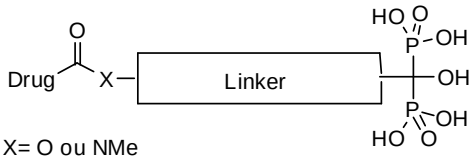

Figure 2

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« La vérité est parfaite pour les mathématiques, la chimie, la philosophie, mais pas pour la vie. »
Ernesto Sabato

CHEMICAL DYNAMICS, PHOTOCHROMISM, NANOSCIENCES

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Chemical Dynamics: triviality or the next frontier of chemistry?

Although obviously part of any chemical transformation, the time evolution of a chemical system is poorly considered especially in academic organic synthetic chemistry. Most of the text book studies rely on standard techniques such as quasi-stationary states or pseudo-first order conditions in order to smooth any weird behavior. However thanks to the progress of numerical treatments it now possible to treat row data without any approximation according to a hypothetical kinetic scheme (“hard-modeling”). This approach is also used in Chemometry: i.e. how to process complex spectroscopic data (typically a fluorescence decay).
The Chemical Dynamics will be exemplified in the lecture following two directions: the deliberate creation of chemical complexity using photochromic reactive dyes, and the spontaneous absolute asymmetric synthesis (Soai reaction) in which a product catalyses its own creation.

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