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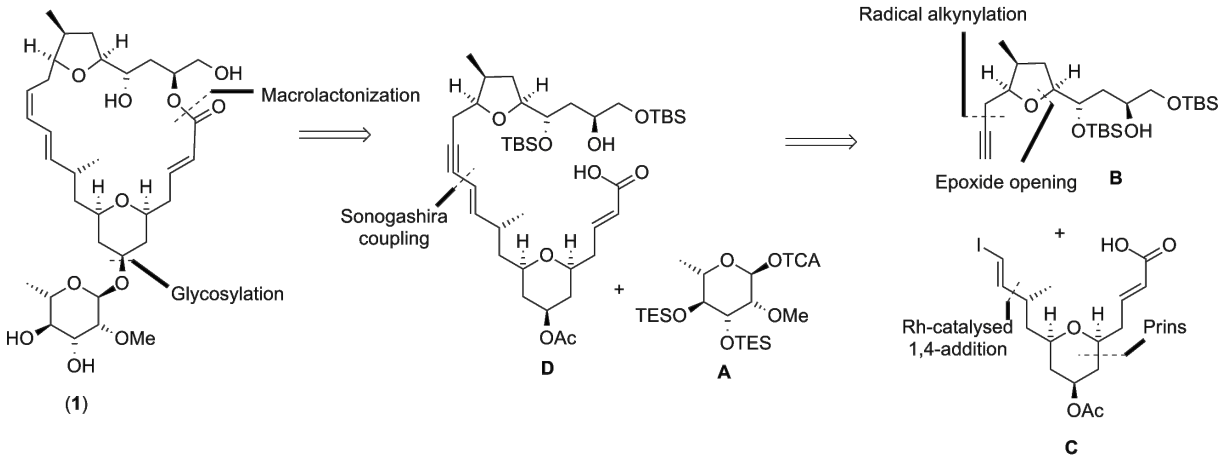
Swiss Chemical Society
Haus der Akademien
Postfach
3001 Bern
Switzerland
info@scg.ch
www.scg.ch

Total Synthesis of Mandelalide A

T. Brütsch[1], K.-H. Altmann[1]*

[1]ETH Zürich

Mandelalide A (**1**) is a glycosylated marine macrolide that was isolated in 2012 from a new *Lissoclinum* species and was found to exhibit significant cytotoxicity to human NCI-H460 lung cancer and to mouse Neuro-2A neuroblastoma cell lines. [1]



In the context of our general interest in biologically active natural products with potential anticancer activity, we embarked on the total synthesis of **1**, in order to provide material for additional biological testing and to establish a chemical basis for eventual SAR studies.

As depicted above, our synthesis of mandelalide A (**1**) [2] is based on building blocks **A**, **B** and **C** as advanced intermediates. Key steps are a macrolactonization of **D** followed by a *Z*-selective reduction of the alkyne moiety and a late stage glycosylation with glycoside donor **A**. Seco acid **D** was assembled by Sonogashira cross-coupling of fragments **B** and **C**. The synthesis **B** includes a rarely used radical alkynylation reaction and an epoxide opening strategy to form the tetrahydrofuran ring. The tetrahydropyran fragment **C** was obtained via a rhodium-catalyzed enantioselective 1,4-addition of TMS-acetylene to crotyl aldehyde and a Prins cyclization to obtain the tetrahydropyran moiety.

This contribution will discuss the details of the total synthesis of mandelalide A (**1)** and provide insight into its unexpected bioactivity profile.

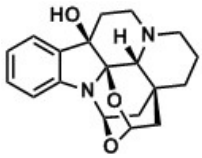
[1] McPhail et al., *J. Org. Chem*. **2012**, *77*, 6066.
[2] For the first total synthesis of **1** see: Xu *et al*., *Angew. Chem. Int. Ed.* **2014**, *53*, 6533.

First Enantioselective Total Synthesis of Terengganensine A

C. Piemontesi[1], R. De Matos[1], Q. Wang[1], J. Zhu[1]*

[1]EPF Lausanne

Terengganensine A, an eburnane indole alkaloid, was isolated in 1997 from *Kopsia terengganensis* by Païs and coworkers.[1] This heptacyclic compound contains an unusual cage-like structure involving the indole nitrogen with six stereogenic centers, one acetal and two aminal functions.



7 steps from commercially available materials
16% overall yield and 95:5 e.r.
Full control of the diastereoselectivity
Confirmation of the absolute configuration

We report herein the first enantioselective total synthesis of terengganensine A.[2] Starting from simple commercially available starting materials, we obtained terengganensine A in 7 steps and 16% overall yield with complete diastereocontrol and 95:5 enantiomeric ratio.

This synthesis also confirmed the absolute configuration of the natural product.

[1] Uzir, S.; Mustapha, A. M.; Hadi, A. H. A.; Awang, K.; Wiart, C.; Gallard, J.-F.; Païs, M. *Tetrahedron Lett.* **1997**, *38*, 1571-1574.
[2] Piemontesi, C.; De Matos, R.; Wang, Q.; Zhu, J. *manuscript in preparation.*

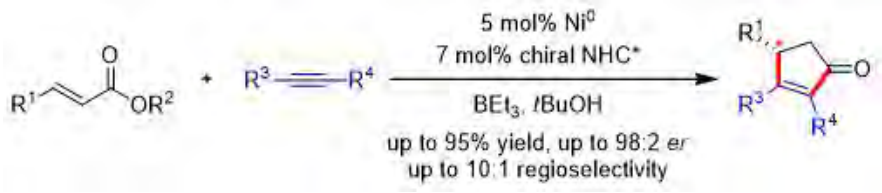
Synthesis of Cyclopentenones by an Asymmetric Nickel-Catalyzed [3+2] Reductive Cycloaddition of Enoates with Alkynes

J. Ahlin[1], N. Cramer[1]*

[1]EPF Lausanne

Amongst the members of the family of five membered carbocyclic rings, cyclopentenones are of utmost importance in chemistry. They are found in numerous natural and synthetic products and also are versatile building blocks in several routes towards the synthesis of complex molecules.[1] Thus, the generation of cyclopentenones in a straightforward manner from readily available substrates remains an important target.

Several transition metal-catalyzed reactions for accessing cyclopentenones have been reported over the past years,[1] including an intermolecular nickel-catalyzed [3+2]-cycloaddition of enoates with alkynes.[2,3] We report an asymmetric nickel-catalyzed [3+2] reductive cycloaddition of enoates with alkynes using a chiral bulky C_1-symmetric N-heterocyclic carbene ligand to provide an efficient highly yielding and enantioselective route to chiral cyclopentenones from simple, stable, and readily available acyclic π-systems.[4]



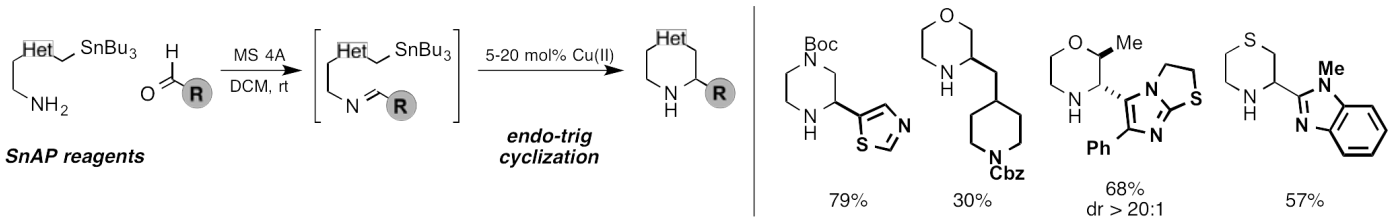
[1] S. E. Gibson, S. E. Lewis, N. Mainolfi, *J. Organomet. Chem.* **2004**, *689*, 3873–3890.
[2] M. Ohashi, T. Taniguchi, S. Ogoshi, *J. Am. Chem. Soc.* **2011**, *133*, 14900–14903.
[3] A. D. Jenkins, A. Herath, M. Song, J. Montgomery, *J. Am. Chem. Soc.* **2011**, *133*, 14460–14466.
[4] J. S. E. Ahlin, P. A. Donets, N. Cramer, *Angew. Chem. Int. Ed.* **2014**, *53*, 13229–13233.

Catalytic One-Step Synthesis of Unprotected Piperazines, Morpholines and Thiomorpholines using SnAP Reagents

M. U. Luescher[1], J. W. Bode[1]*

[1]ETH Zurich

Saturated N-heterocycles have long been considered as privileged elements for the preparation of bioactive small molecules. Increasing recognition of problems associated with heteroaromatic pharmacophores, such as poor solubility, bioavailability, or pharmacokinetics have further enhanced their importance in drug development. [1] Despite this, their synthesis often requires long, laborious synthetic routes to form these ring systems, including the need for protecting groups. To directly access a variety of fully saturated N-heterocycles in a single synthetic operation, we have recently introduced SnAP (Stannyl Amine Protocol) reagents, which convert aldehydes and ketones directly into morpholines, piperazines, diazepanes, thiomorpholines, spiro- and other N-heterocycles. [2–6]



The major limitation using the SnAP reagents is the need for stoichiometric copper reagents. We have now identified new ligands and conditions that render the reaction catalytic in copper and expanded the substrate scope to α-bis(substituted) SnAP reagents. These studies, including approaches towards an enantioselective process and insights into the unique reaction mechanism, will be discussed.

[1] N. A. Meanwell, *Chem. Res. Toxicol.* **2011**, *24*, 1420–1456.
[2] C.-V. T. Vo, G. Mikutis, J. W. Bode, *Angew. Chem. Int. Ed.* **2013**, *52*, 1705–1708.
[3] M. U. Luescher, C.-V. T. Vo, J. W. Bode, *Org. Lett.* **2014**, *16*, 1236–1239.
[4] C.-V. T. Vo, M. U. Luescher, J. W. Bode, *Nat. Chem.* **2014**, *6*, 310–314.
[5] W.-J. Siau, J. W. Bode, *J. Am. Chem. Soc.* **2015**, *136*, 17726–17729.
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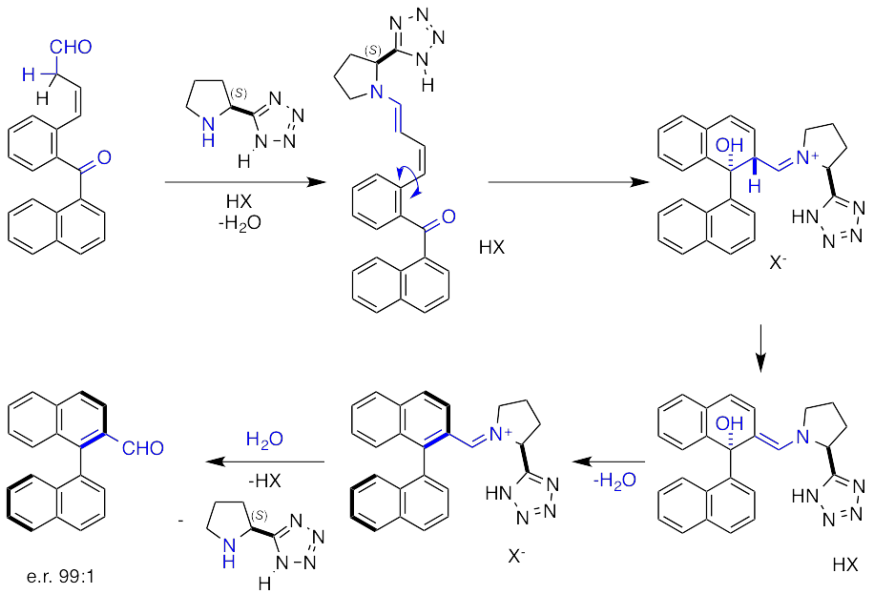
Organocatalytic Atroposelective Aldol Condensation

A. Link[1], C. Sparr[1]*

[1]University of Basel

Axially chiral compounds are important building blocks for various applications, e.g. in ligand design. Despite the importance of atropisomers such as binaphthyl derivatives, only few stereoselective methods are available for their synthesis.

A catalytic method was developed that converts ketoaldehyde precursors into tri-*ortho*-substituted biaryls upon treatment with a pyrrolidinyl-tetrazole catalyst. The stereochemical information is thereby efficiently transferred from the catalyst into the axial chirality of the product. Initially, an activated dienamine is formed to trigger a subsequent cyclization followed by a second α-deprotonation. This activates for a dehydration step leading to the formation of an aromatic ring providing sufficient driving force for the reaction. Hydrolysis to regenerate the catalyst gives the binaphthalene-carbaldehyde with an e.r. = 99 :1.



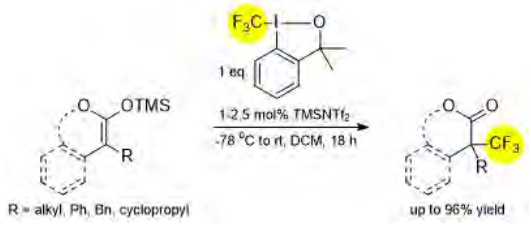
A. Link, C. Sparr, *Angew. Chem.* **2014**, *126*, 5562–5565; *Angew. Chem. Int. Ed.* **2014**, *53*, 5458–5461.

Lewis Acid Catalyzed Electrophilic Trifluoromethylation of Silyl Ketene Acetals: Access to Quaternary α-Trifluoromethylated Esters and Lactones

D. Katayev[1], V. Matoušek[1], R. Koller[1], A. Togni[1]*

[1]ETH Zurich

Herein we describe an efficient Lewis acid-catalyzed trifluoromethylation of silyl ketene acetals using a hypervalent iodine-CF_3-reagent as an electrophilic source of CF_3.[1-2] The mild reaction protocol provides direct access to quaternary α-trifluoromethylated esters and lactones in up to 96% isolated yield using only 1-2.5% of trimethylsilyl triflimide ($TMSNTf_2$) as catalyst. It is noteworthy that the reaction of cyclic ketene acetals is rather insensitive to steric bulk, with excellent yields for isopropyl, tert-butyl and cyclohexyl-substituted lactones. The 5-, 6- and 7-membered α-trifluoromethylated lactones were successfully prepared by this method. Cyclic α-aryl-substituted silyl ketene acetals reacted sluggishly, whereas α-heteroatom-substituted substrates gave complex reaction mixtures. Mechanistic studies led to conclude that free radical intermediates are unlikely in this transformation, although they cannot be ruled out with certainty.



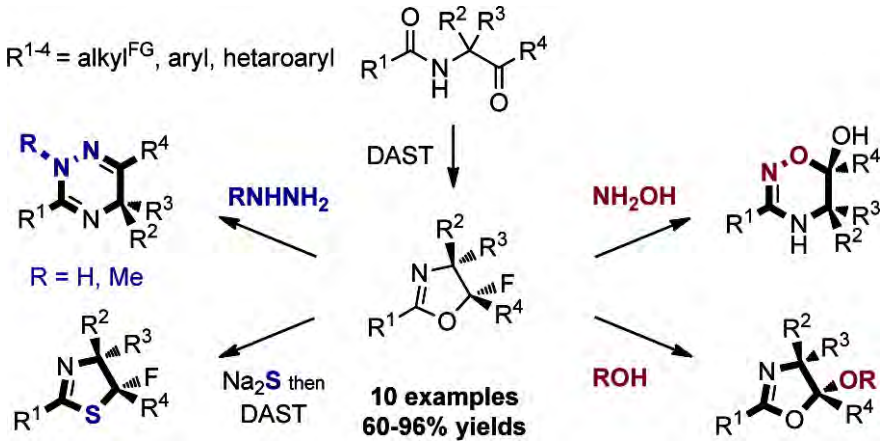
[1] J. Charpentier, N. Früh, A. Togni, *Chem. Rev.,* **2015**, *115*, 650-682.
[2] V. Matoušek, D. Katayev, R. Koller, A. Togni, **2015**, *manuscript in preparation.*

DAST-mediated Cyclization of a,a-Disubstituted-a-acylaminoketones: Efficient and Divergent Synthesis of Unprecedented Heterocycles

A. Bigot[1], O. Loiseleur[1]

[1]Syngenta Crop Protection AG

The ligand-based rational design of a new insecticidal ecdysone agonist will be presented.[1] Synthetic efforts towards this unprecedented fluorooxazoline scaffold further led to the discovery of a DAST-mediated cyclization of α,α-disubstituted -α-acylaminoketones. Mechanistic studies revealed that the resulting products can be ring-opened or selectively substituted by a range of nucleophiles to provide in high yields a diverse array of novel heterocyclic frameworks.[2]



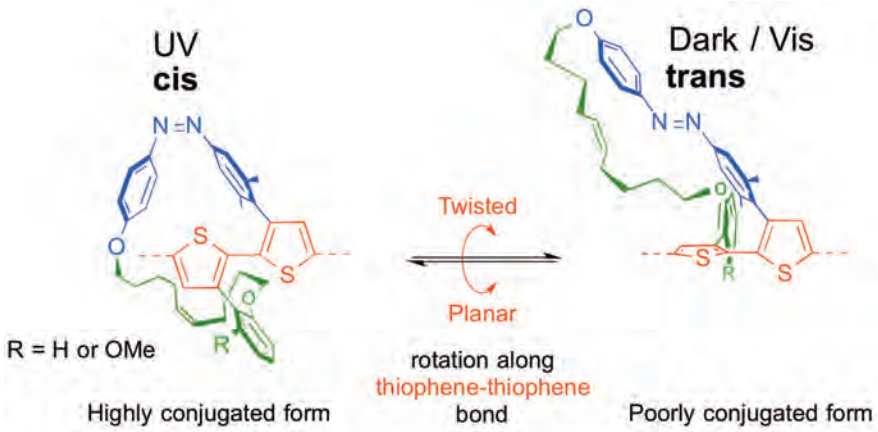
1. D. Emery, A. Bigot, O. Loiseleur, *unpublished results.*
2. Bigot, J. Blythe, C. Pandya, T. Wagner, O. Loiseleur, *Org.Lett.* **2011**, *13*, 192-195.

Design, synthesis and properties of 'Photochomic Torsional Switches' (PTS)

G. Sforazzini[1]

[1]École Polytechnique Fédérale de Lausanne (EPFL)

The geometrical arrangement of the p-orbitals in organic semiconductors plays a pivotal role for the optoelectronic properties of the resulting bulk materials.[1] Control over the π-bond geometry, e.g. the planarity, of an extended conjugated system offers the possibility to modulate the effective conjugation length of a π-system, thus, allowing for the tuning of optical and electronic properties.[1,2] A promising way to reversibly modulate the orientation of the p-orbitals in a conjugated strucrure is to incorporate photochromic segments onto the 'backbone' of the π-system. Attempts to use photochromic molecules as monomer units in a polymer chain have shown that the photo-reversibility efficiency decreases inversely with the enhancement of the π-conjugation.[3] In the present work we report on a novel molecular architecture, referred to as a 'photochromic torsional switch' (PTS), which can overcome the limits of todays photochromic dyes towards their incorporation into extended π-system. The aforementioned molecular structure consists of a polymerizable bithiophene unit able to mechanically change its π-system planarity in response to a photochromic isomerization of a laterally attached azobenzene unit. In the dark and upon exposure of visible light, the azobenzene moiety assumes its extended *trans* conformation, thus, forcing the bithiophene backbone to twist out of coplanarity (dihedral angle from 50° to 68°). By contrast, exposure to UV light results in isomerization of the azobenzene unit to the *cis* conformation, which allows the bithiophene fragment to assume a planar, π-conjugated conformation (dihedral angles from 150° to 168°). The PTS architectures, proposed in this work, represent a new generation of photochromic dyes that can allow for the preparation of 'conjugated photochromic polymers', and help to gain deeper understanding of the correlation between molecular conformation and optoelectronic properties of π-conjugated macromolecules.



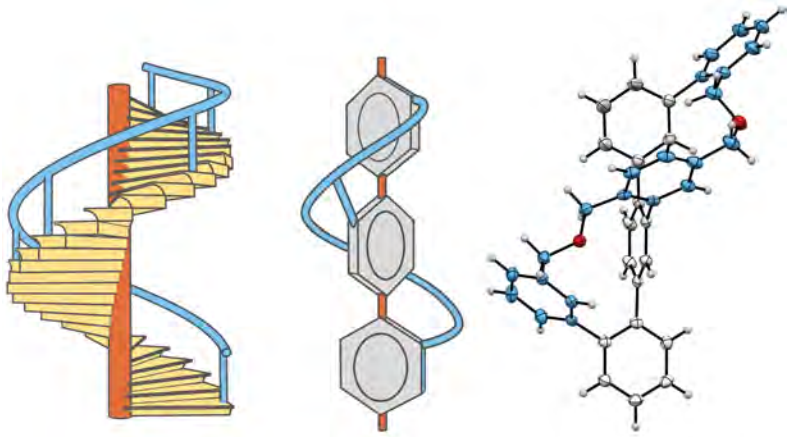
[1] S. Ko, E. T. Hoke, L. Pandey et al.,*J. Am. Chem. Soc.*,**2012**, 134, 5222-5232
[2] R. Rieger, D. Beckmann, A. Mavrinskiy et al.,*Chem. Mater.*,**2010**, 22, 5314-5218
[3] F. Stellacci, C. Bertarelli, F. Toscano et al.,*Adv. Mater.*,**1999**, 11, 292-295

Dancing Ladders - Inducing and Distorting Helical Chirality in Achiral Polycyclic Systems

M. Rickhaus[1], M. Mayor[1]*

[1]University of Basel

Recently we presented an exciting new concept in ***Angewandte Chemie***[1a,b] how to introduce helicity to a polyaromatic system by interlinking two oligomer strands of different lengths. To compensate for the dimensional mismatch, the longer oligomer wraps around the oligophenyl backbone. The obtained "Geländer"- (or bannister-) oligomers resemble helical staircases or pirouetting dance ribbons.



The new helical polyaromatics were fully characterized including X-ray diffraction analysis. Because the obtained helical structures lack a point of inversion they exist exclusively as one set of enantiomers. The isolation of the pure enantiomers enabled the chiroptical properties as well as the racemization process to be studied by circular dichroism spectroscopy. The targeted variation of the interlinking heteroatom allowed varying the degree of twist and studying the resulting heights on the racemization barriers.[2] By mismatching the oligomeric strands it was possible to distort the induced helicity and investigate the impact on the dynamics of the system.[2]

[1] a) M. Rickhaus, L. M. Bannwart, M. Neuburger, H. Gsellinger, K. Zimmermann, D.Häussinger, M. Mayor, *Angew. Chem. Int. Ed.*, **2014**, *53*, 52, 14587–14591; b) Synthetic details: M. Rickhaus, L. M. Bannwart, O. Unke, H. Gsellinger, D. Häussinger, M. Mayor, *Eur. J. Org. Chem.*, **2015**, *4*, 786-801.
[2] Manuscripts in preparation

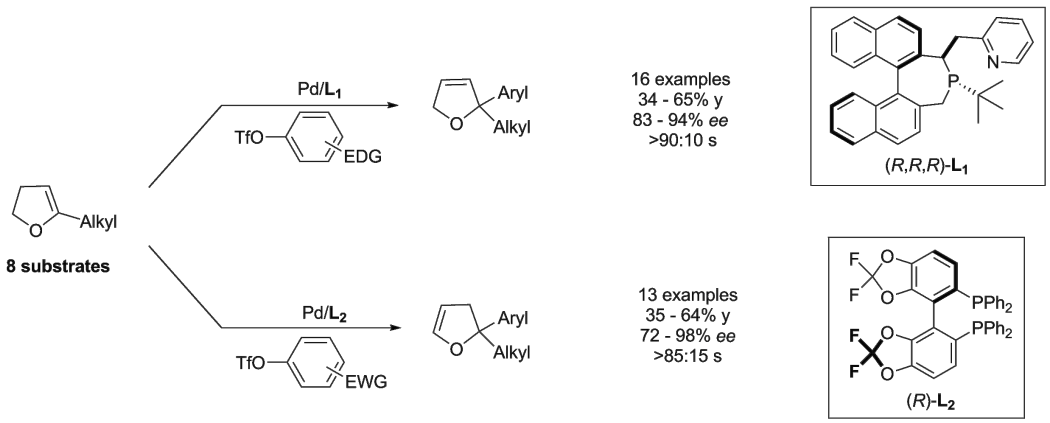
Access to dihydrofurans with a fully substituted C2 stereocenter by Pd-catalyzed intermolecular asymmetric Heck reaction

G. M. Borrajo-Calleja[1], V. Bizet[1], T. Bürgi[1], C. Mazet[1]*

[1]University of Geneva

During the past decades, the asymmetric *intramolecular* Heck reaction has been successfully applied in natural product synthesis to install tertiary and quaternary stereocenters.[1] Surprisingly, the *intermolecular* asymmetric version of the reaction has not reached the same level of development. To date, it has been used essentially as a benchmark reaction to validate the design of novel homotopic and heterotopic chiral bidentate ligands. Systematic studies with emphasis on expanding the scope of substrates are scarce.[2,3]

Herein we describe a highly selective methodology that gives access to chiral 2,3 and 2,5 dihydrofurans with a fully substituted C2 stereocenters. Under identical experimental conditions, with our homemade (P,N) ligand **L1** or the commercially available (P,P) ligand **L2**, 2,5 or 2,3-dihydrofurans can be obtained respectively with high enantioselectivity, regioselectivity and good yields.[4]



[1] a) A. B. Dounay, L. E. Overman *Chem. Rev*. **2003**, *103*, 2945; b) D. M. Cartney, P. J. Guiry *Chem. Soc. Rev*. **2011**, *40*, 5122.
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Domino Reaction to Functionalize Heterocycles: A Complementary Method to C-H Functionalization

Y. Li[1], J. Waser[1]*

[1]EPF Lausanne

Molecules containing heterocycles and alkynes play an important role in medicinal chemistry and material science.[1] A well-established method – the Sonogashira reaction- has dominated the introduction of alkynes onto heterocycles. Due to several shortcomings of the Sonogashira reaction as stoichiometric waste and poor stability or difficult accessibility of starting materials, a more direct and regioselective fashion is highly desirable.

Based on the previous work in our group,[2] a direct alkynylation method was first developed to access *C2* alkynylated furans and benzofurans with gold catalyst and benziodoxolone reagents.[3] In the case of benzofurans, $Zn(OTf)_2$ was discovered as a new and efficient reagent to activate alkynylated hypervalent iodine reagent.[3b]

Although C-H direct alkynylation provides a straightforward way to access these alkynylated heterocycles, there are still several shortcomings to consider. One of the most serious issues is that the functionalized positions are limited on heterocycles because of its inherent reactivity. It is therefore intriguing and challenging to find an alternative way to functionalize other positions. Domino reactions could provide a solution to this challenge, as the metal-carbon bond could be installed on positions different from the ones obtained by direct metallation pathway.

Starting from 2012, we spent intensive effort on developing a cyclization-alkynylation domino reaction to functionalize *C3* position on furan, with discovering that bistriflroromethyl benziodoxole reagent is an exceptionally efficient reagent for this process with gold (III) catalyst.[3a] Then we turned our interest to more challenging positions, *C5* and *C6* on benzene part of indoles. Platinum (II) catalyst showed superior reactivity towards carbon nucleophile cyclization –alkynylation process.[4]

Combining two approaches, a series of alkynylated hetercycles could be rapidly accessed. Further functionalization of these compounds could construct several unprecedented building blocks for organic material science. Developing new domino process with different metal catalysts and electrophilic reagents could be envisaged as the next step.

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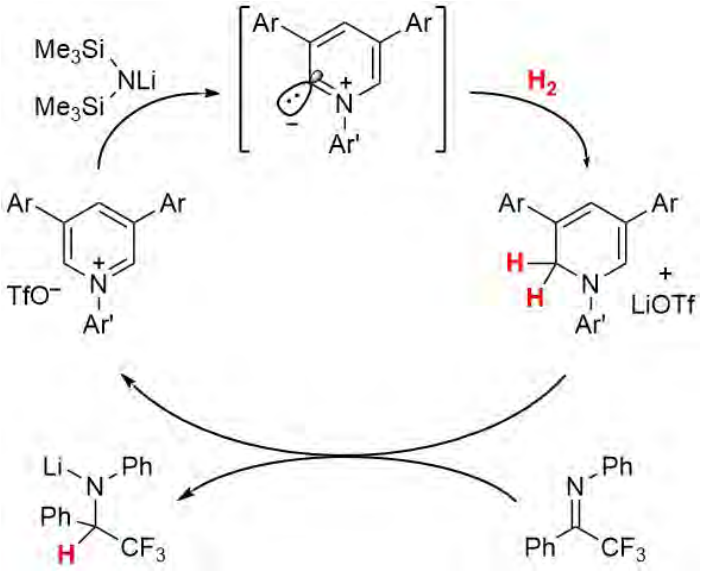
Pyridylidene-Mediated Dihydrogen Activation Coupled with Catalytic Imine Reduction

J. Auth[1], A. Pfaltz[1]*

[1]University of Basel

In recent years dihydrogen activation at non-metallic centers has found increasing attention. A breakthrough in the development of non-metallic catalytic systems for H_2 activation was achieved by Stephan and Erker, based on the concept of Frustrated Lewis Pairs (FLPs).[1] We developed a system in which dihydrogen is trapped by a pyridylidene intermediate that is generated from a pyridinium salt and base. This process has precedent in the H_2 addition to an aminocarbene reported by Bertrand and coworkers. [2] However, in contrast to the amine produced from an aminocarbene, dihydropyridine resulting from H_2 addition to a pyridylidene can act as reducing agent toward organic electrophiles. By coupling the hydrogen activation step with subsequent hydride transfer from the dihydropyridine to an imine, a catalytic process was established.



Scheme 1. Proposed catalytic cycle.

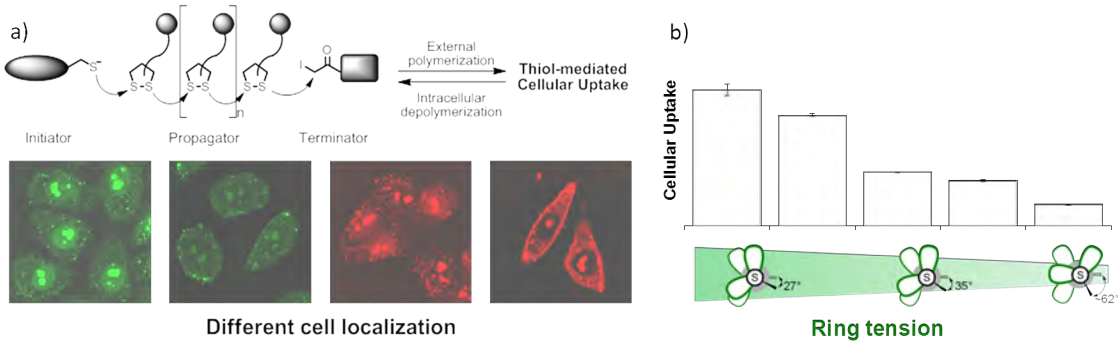
[1] Reviews: a) D. W. Stephan, G. Erker, *Angew. Chem. Int. Ed.* **2010**, *49*, 46; b) G. Erker, *Pure Appl. Chem.* **2012**, *84*, 2203; c) D. W. Stephan, *Org. Biomol. Chem.* **2012**, *10*, 5740; d) J. Paradies, *Angew. Chem. Int. Ed.* **2014**, *53*, 3552; e) L. Hounjet, D. W. Stephan, *Org. Process Res. Dev.* **2014**, *18*, 385.
[2] G. D. Frey, V. Lavallo, B. Donnadieu, W. W. Schoeller, G. Bertrand, *Science* **2007**, *316*, 439.
[3] K. Hata, Y. Segawa, K. Itami, *Chem. Commun.* **2012**, *48*, 6642.

Disclosing a Novel Way for Poly(disulfide)s to Enter Cells

G. Gasparini[1], D. Abegg[1], A. Adibekian[1], N. Sakai[1], S. Matile[1]*

[1]University of Geneva

We recently introduced a new methodology for the synthesis of cell-penetrating poly(disulfide)s, based on substrate-initiated polymerization (siCPDs).[1] Molecules of free choice are used as initiators for ring-opening disulfide-exchange polymerization, linking them covalently to the transporter. After cellular uptake, siCPDs are readily depolymerized by endogenous glutathione, releasing the substrate and providing optimal biocompatibility (Fig. 1a).[2] We also reported the major role played by molecular weight of the siCPDs on cellular localization: it influences the depolymerization kinetics and thus dictates the accumulation of the polymer in endosomes, cytosol or nuclei.[3]



We are now focused on the hypothesis that disulfide bonds not only impact cell localization, but also enhance transmembrane activity. To prove this proposed thiol-mediated uptake mechanism, we prepared a collection of fluorescent probes equipped with different thiols and disulfides. Experiments on living cells clearly indicate that the presence of disulfides, especially under ring tension, enhances the overall uptake (Fig. 1b), suggesting a new energy-dependent pathway that can be exploited to enter into cells.[4] Pull-down proteomics are ongoing in order to identify if and which proteins are involved in the process, towards the delivery of larger substrates.

[1] E.-K. Bang, G. Gasparini, G. Molinard, A. Roux, N. Sakai, S. Matile, *J. Am. Chem. Soc.* **2013**, *135*, 2088-2091.
[2] G. Gasparini, E.-K. Bang, G. Molinard, D. V. Tulumello, S. Ward, S. O. Kelley, N. Sakai, S. Matile, *J. Am. Chem. Soc.* **2014**, *136*, 6069-6074.
[3] N. Chuard, G. Gasparini, A. Roux, N. Sakai, S. Matile, *Org. Biomol. Chem.* **2015**, *13*, 64-68.
[4] G. Gasparini, G. Sargsyan, E.-K. Bang, N. Sakai, S. Matile, *Angew. Chem. Int. Ed.* **2015**, in press.

Halogen Bonding Supramolecular Capsules

O. Dumele[1], N. Trapp[1], F. Diederich[1]*

[1]Laboratorium für Organische Chemie, ETH Zürich, Vladimir-Prelog-Weg 3, 8093 Zurich, Switzerland

Molecular capsules based on solely the interaction of halogen bonding (XB) are presented along with their host–guest binding properties.[1] The first example of a well-defined four-point XB supramolecular system is realized by decorating resorcin[4]arene cavitands with polarized halogen atoms for the dimerization with tetra(4-pyridyl) resorcin[4]arene cavitands. NMR binding titrations for F, Cl, Br, and I cavitands as XB donor show association constants K_a up to 5370 L mol^{-1} (ΔG = –4.85 kcal mol^{-1}, for I) even in XB-competitive solvent such as benzene/acetone/MeOH 70:30:1, where comparable monodentate model systems show no association. XB binding is evidenced by 2D HOESY NMR, and the thermodynamic profile shows the largely enthalpic driven nature of the cooperative binding, as predicted earlier by our model system studies.[2]

With these results in hand, we provide detailed experimental data on all halogens (I, Cl, Br, and F) with respect to their XB binding properties. The presented capsular architecture shows the emerging impact of even weak XB interactions for their contribution in future supramolecular chemistry, especially in the context of modern ligand design for medicinal chemistry and crop science.[3]

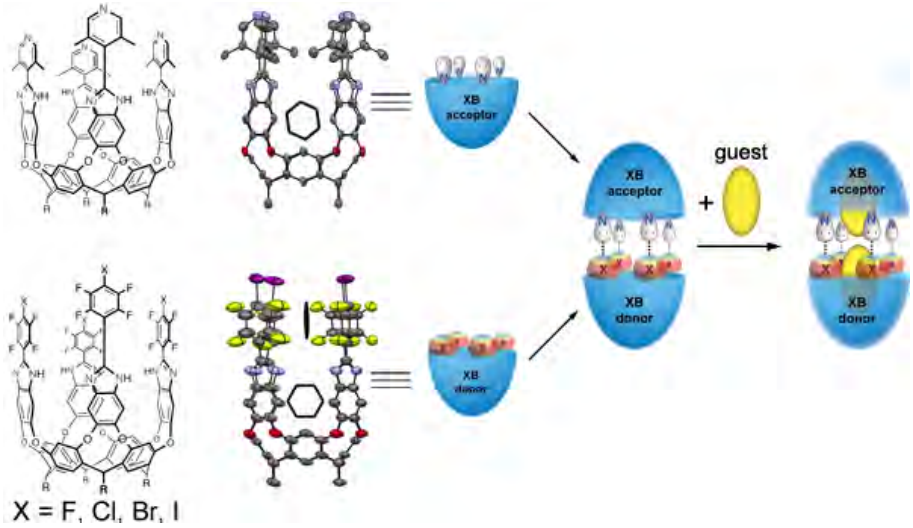


Figure 1. Resorcin[4]arene cavitands as XB acceptor (top) and XB donor hemispheres (bottom) for the assembly of XB molecular capsules. The X-ray structures show each cavitand (X = I) in its *vase* conformation with encapsulated benzene molecules (50% probability ellipsoids, 100 K). Upon mixing of the two components in solution, the formation of XB molecular capsules takes place.[1]

[1] O. Dumele, N. Trapp, F. Diederich, *Angew. Chem. Int. Ed.* **2015**, DOI: 10.1002/anie.201502960, Halogen Bonding Molecular Capsules.
[2] O. Dumele, D. Wu, N. Trapp, N. Goroff, F. Diederich, *Org. Lett.* **2014**, *18*, 4722–4725. Halogen Bonding of (Iodoethynyl)benzene Derivatives in Solution.
[3] E. Persch, O. Dumele, F. Diederich, *Angew. Chem. Int. Ed.* **2015**, 54, 3290–3327. Molecular Recognition in Chemical and Biological Systems.