

# Geneva chemistry & biochemistry days 2017

TH 19 January 2017, 09:00–17:00

FR 20 January 2017, 09:00–12:00

Sciences II – auditoire A100 – quai Ernest-Ansermet 30 – 1205 Genève

**No registration required**

**Prof. Peter J. Hore**  
University of Oxford

**Prof. Andreas Mayer**  
Université de Lausanne

**Prof. Jonathan R. Nitschke**  
University of Cambridge

**Prof. Peter H. Seeberger**  
Max-Planck-Institut für Kolloid- und Grenzflächenforschung Potsdam

Junior speakers: • Daniel Abegg • Karine Baudet • Gustavo Borrajo-Calleja • Miguel Coll Crespi • Maria Teresa Delgado Perez • Bogdan Dereka • Maria Labrador Beltran • Timothée Lathion • Giuseppe Licari • François Miros • Takuya Machida • Mohsen Moazzami Gudarzi • Marko Pavlovic • Daniel Sethio • Lukas Wettmann • Dajing Yuan • Bei Zhang •



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FACULTÉ DES SCIENCES  
**SECTION DE CHIMIE ET BIOCHIMIE**



**UNIVERSITÉ  
DE GENÈVE**



## FOREWORD

The *Section de chimie et biochimie*, University of Geneva, has the pleasure to announce the 7<sup>th</sup> edition of its “**Geneva Chemistry & Biochemistry Days**”.

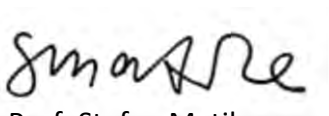
The vocation of the event is to give our students who are close to finishing their PhD studies the opportunity to present their research as short talks to an audience from academia and industry, and the steering committee is glad to welcome you in this context.

Four distinguished lecturers further enrich the programme. They have been invited by our four departments, and they will illustrate the extent and quality of top-level fundamental research in chemistry and biochemistry today.

Our BSc and MSc students are welcome to smell the very flavour of the research held in our School and abroad, and to learn a bit more about how to present results to a scientific audience.

It is hoped that the event will catalyse fruitful discussions between young and advanced researchers, and give our students an opportunity to get ready for their professional careers, yet offering our guests an overview of the quality of the fundamental research performed in our School.

Looking forward to meeting you at this event, we hope that you will enjoy the lectures and interactions!



Prof. Stefan Matile

*Président de la Section de chimie et biochimie*

## Steering and organising committee

Prof. Claude Piguet	claudio.piguet@unige.ch <i>Département de chimie minérale et analytique</i>
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## PROGRAMME – THURSDAY, 19 JANUARY

Chairmen: **Prof. Claude Piguet + Dr Fabien Cougnon**

09:00-09:05	<b>Prof. Stefan Matile</b>	Welcome message Introduction
09:05-09:50	<b>Prof. Jonathan R. Nitschke</b> University of Cambridge	Transformative cages and luminous chains: Functional systems through subcomponents self-assembly
09:50-10:10	<b>Giuseppe Licari</b>	DNA probes at liquid interfaces
10:10-10:30	<b>Maria Labrador Beltran</b>	Modular and enantiospecific synthesis of cationic [6]helicenes and selective orthogonal functionalizations
10:30-10:45	Coffee break Main hall of Sciences III	
10:45-11:05	<b>Timothée Lathion</b>	Iron(II) spin crossover in mono and dinuclear <i>d-f</i> complexes
11:05-11:25	<b>Lukas Wettmann</b>	Stochastic switching of Min proteins in short <i>Escherichia coli</i> cells
11:25-11:45	<b>Maria Teresa Delgado Perez</b>	Structural investigation of the HS to LS relaxation dynamics in spin crossover compounds
11:45-12:05	<b>Daniel Abegg</b>	Strained cyclic disulfides enable cellular uptake by reacting with the transferrin receptor
12:05-13:40	Lunch (senior speakers + junior lecturers) Restaurant-pizzeria Sole Mio, boulevard Carl-Vogt	

Chairmen: **Prof. Eric Vauthey + Dr Céline Besnard**

13:40-14:00	<b>Karine Baudet</b>	How the chemical potential of the solvent acts in intermolecular association processes
14:00-14:20	<b>Bogdan Dereka</b>	Excited-state symmetry breaking: Toward asymmetrical photochemistry
14:20-14:40	<b>Gustavo Borrajo-Calleja</b>	Palladium-catalyzed enantioselective intermolecular carboetherification and carboamination of dihydrofurans
14:40-15:00	<b>Miguel Coll Crespi</b>	Ionophore based emulsions as heterogeneous pH buffer
15:00-15:15	Coffee break Main hall of Sciences III	
15:15-15:35	<b>Bei Zhang</b>	Ag doped Au <sub>38</sub> (SR) <sub>24</sub> nanocluster: Surface flexibility and silver migration
15:35-15:55	<b>François Mirois</b>	The anion- $\pi$ interaction: A tuneable non-covalent interaction for catalysis
15:55-16:15	<b>Mohsen Moazzami Gudarzi</b>	Depletion and double layer forces in the presence of like-charged polyelectrolytes
16:15-17:00	<b>Prof. Peter J. Hore</b> University of Oxford	A chemical magnetic compass for avian navigation
17:00-	<i>Verre de l'amitié</i> Main hall of Sciences III	

## PROGRAMME – FRIDAY, 20 JANUARY

Chairmen: **Prof. Alexander Adibekian + Prof. Marcos González Gaitán + Dr Bernhard Lang**

09:00-09:45	<b>Prof. Peter H. Seeberger</b> Max-Planck-Institut Potsdam	Automated glycan assembly as basis for vaccine development and material science
09:45-10:05	<b>Dajing Yuan</b>	Voltammetric thin layer ionophore based membranes for ion activity detection: Two sensing modes
10:05-10:25	<b>Takuya Machida</b>	Phosphatase activity screening based on constrained peptide-peptide nucleic acid (PNA) conjugates
10:25-10:40	Coffee break Main hall of Sciences III	
10:40-11:00	<b>Daniel Sethio</b>	Influence of van der Waals dispersion correction on the DFT description of alkaline-earth fluorohalides
11:00-11:20	<b>Marko Pavlovic</b>	Stabilization of enzyme-clay nanocomposite in dispersion by heparin adsorption
11:20-12:05	<b>Prof. Andreas Mayer</b> Université de Lausanne	Roles of an inorganic intracellular polymer in metal acquisition, phosphate and energy homeostasis
12:05-12:10	<b>Prof. Eric Vauthey</b>	Award of the best oral presentation
12:10-12:15	<b>Prof. Stefan Matile</b>	Concluding remarks

## A chemical magnetic compass for avian navigation

Peter J. HORE

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Migratory birds travel spectacular distances each year, navigating and orienting by a variety of means, most of which are poorly understood. Among them is a remarkable ability to perceive the intensity and direction of the Earth's magnetic field.



Biologically credible mechanisms for sensing such weak fields are scarce and in recent years just two proposals have emerged as frontrunners.

One, essentially classical, involves clusters of iron-containing particles.

The other relies on magnetically sensitive chemistry. The latter began to attract interest following a proposal 16 years ago that photochemically formed flavin-tryptophan radical pairs in cryptochrome proteins in the retina could be responsible.

The quantum spin dynamics of such transient reaction intermediates is thought to lead to changes in the yield of a signalling state of the protein even though the interaction with the geomagnetic field is six orders of magnitude smaller than the thermal energy,  $k_B T$ .

In this talk, I will outline the basis of the radical pair mechanism, present some of the experimental and theoretical evidence for the cryptochrome hypothesis and comment on the extent to which cryptochromes are fit-for-purpose as magnetoreceptors.



## Roles of an inorganic intracellular polymer in metal acquisition, phosphate and energy homeostasis

## Andreas MAYER

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Acidocalcisomes are nearly orphan organelles that occur in all kingdoms of life. Their broad distribution and their invariant composition suggest that they perform fundamental functions for cell metabolism, which are, however, poorly explored. One hallmark is their high content of polyphosphate, an inorganic polymer of up to several hundreds of phosphate units linked by phosphoric anhydride bonds.

Recent elucidation of enzymes and regulatory domains involved in the synthesis and turnover of acidocalcisomal polyphosphate in eukaryotes<sup>1,2</sup> has allowed the identification of novel pathways for metal acquisition, energy<sup>3,4</sup> and phosphate homeostasis<sup>5</sup>, which will be presented.

1. Hothorn M., Neumann H, Lenherr E.D., Wehner M., Rybin V., Hassa P.O., Uttenweiler A. Reinhardt M., Schmidt A., Seiler J., Ladurner A.G., Herrmann C., Scheffzek K., Mayer A. *Science* **2009**, 324, 513-.
2. Gerasimaite R., Mayer A. *Biochem. Soc. Trans.* **2016**, 44, 234-.
3. Sziogyarto Z., Garedew A., Azevedo C., Saiardi A. *Science* **2011**, 334, 802-.
4. Desfougères Y., Gerasimaitė R., Jessen H.J., Mayer A. *J. Biol. Chem.* **2016**, 291, 22262-.
5. Wild R., Gerasimaitė R., Jung J.-Y., Truffault V., Pavlovic I., Schmidt A., Saiardi A., Jessen H.J., Poirier Y., Hothorn M. *Maver A. Science* **2016**, 352, 986-.

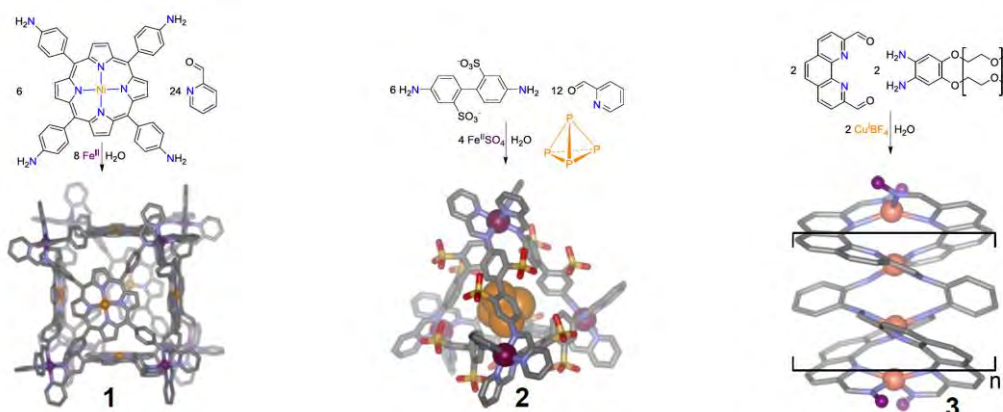
## Transformative cages and luminous chains: Functional systems through subcomponent self-assembly

Jonathan R. NITSCHKE

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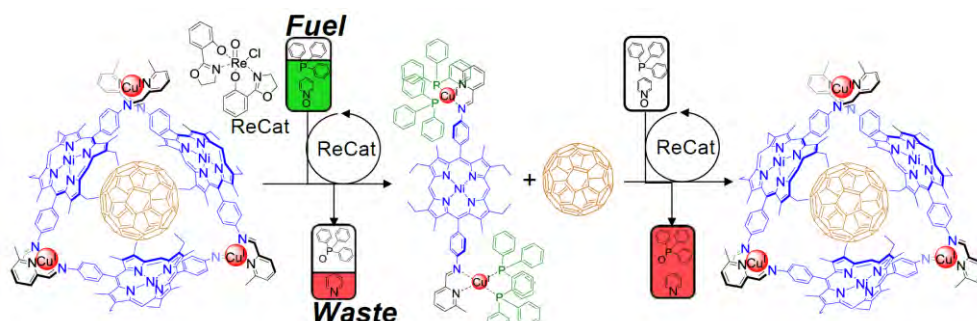
The materials that we depend on rely upon ever-increasing structural complexity for their function. The use of chemical self-assembly as a synthetic technique can simplify materials preparation by shifting intellectual effort away from designing molecules, and towards the design of *chemical systems* that are capable of self-assembling in such a way as to express desired materials properties and functions. Below are shown the subcomponent precursors and structures of three of products that can form functional constituents of these systems (Figure 1).



**Figure 1.**

$\text{Fe}^{\text{II}}_8$  cubic cage **1**<sup>1</sup>,  $\text{Fe}^{\text{II}}_4$  tetrahedral cage **2**<sup>2</sup>, and electroluminescent  $\text{Cu}^{\text{I}}_n$  double-helical polymer **3**<sup>3</sup>

Current challenges involve inducing multiple structures to form in parallel<sup>4</sup>, such that they may act in concert to achieve a catalytic goal<sup>5</sup>, our techniques allow entry into the emerging field of *systems chemistry*<sup>6</sup>. Functional systems that we have recently developed include a fuel-controlled self-assembly process (Figure 2)<sup>7</sup> and a triphasic sorting system, wherein three guests are selectively encapsulated within three cages, each in turn soluble in only one of three mutually-immiscible phases (water and two different ionic liquids)<sup>8</sup>.



**Figure 2.**

A system wherein duration of release of a guest ( $\text{C}_{60}$ ) can be programmed by the amount of fuel ( $\text{PPh}_3$ ) present

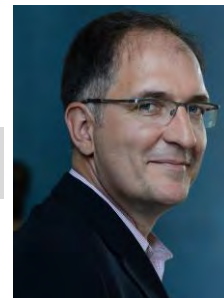
1. Meng W., Breiner B., Rissanen K., Thoburn J.D., Clegg J.K., Nitschke J.R. *Angew. Chem. Int. Ed.* **2011**, 50, 3479-.
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## Automated glycan assembly as basis for vaccine development and material science

**Peter H. SEEBERGER**

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 Freie Universität Berlin; Berlin, Germany  
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Pure glycans are key to enable biochemical, biophysical and immunological studies aimed at understanding the role of carbohydrates. Described is the development of a fully integrated platform for automated glycan assembly (AGA) based on solid-phase oligosaccharide synthesis<sup>1</sup> and carbohydrate arrays to address biological problems.

Particular emphasis in this lecture will be placed on the new automated synthesis platform<sup>2</sup> that has been commercialized<sup>3,4</sup>. Access to defined polysaccharides as long as 50-mers enables now biological as well as materials science investigations<sup>5,6</sup>. These synthetic polysaccharides can be combined much like “molecular LEGO” to create even larger oligosaccharide assemblies. Quality control of synthetic glycans can now be guaranteed using ion mobility mass spectrometry to very low levels<sup>7</sup>.

Carbohydrate arrays are used as diagnostics and in support of vaccine programs that are based on conjugates with synthetic oligosaccharides to screen blood sera<sup>8</sup>. Case studies of specific vaccines will provide an appreciation for the approach that is now advancing candidates toward clinical testing<sup>9-11</sup>. Fully synthetic vaccine candidates exploit iNKT cells to induce a robust and protective immune response<sup>12</sup>.

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3. [www.glycouniverse.de](http://www.glycouniverse.de)
4. Seeberger P.H. *Acc. Chem. Res.* **2015**, 48, 1450-.
5. Hahm H.S., Hurevich, Seeberger P.H. *Nature Comm.* **2016**, 7, in press.
6. Kottari N., Schuhmacher F., Seeberger P.H. *Manuscript in preparation*.
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12. Cavallari M., Stallforth P., Kalinichenko A., Rathwell D., Gronewold T.M.A., Adibekian A., Mori L., Landmann R., Seeberger P.H., De Libero G. *Nature Chem. Bio.* **2014**, 10, 950-.

## Strained cyclic disulfides enable cellular uptake by reacting with the transferrin receptor

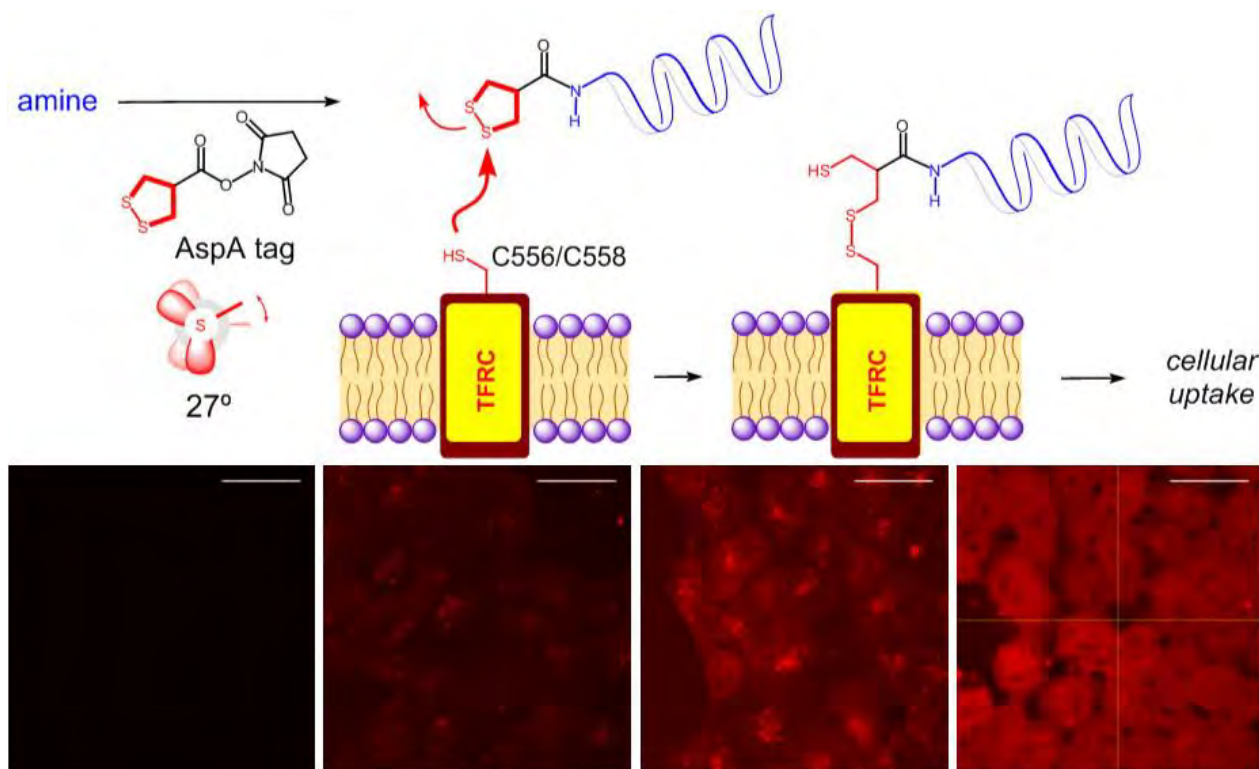
Daniel ABEGG

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In this study, we demonstrate that appendage of a single asparagusic acid residue (AspA tag) is sufficient to ensure efficient cellular uptake and intracellular distribution of fully unprotected peptides. We then apply this new delivery method to induce apoptotic response in cancer cells using long (up to 20mer) pro-apoptotic BH3 domain peptides.

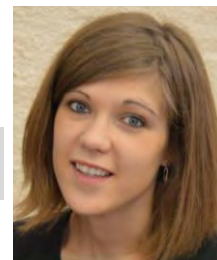
In order to elucidate the molecular mechanism of the cellular uptake, we perform chemical proteomics experiments and identify the direct molecular targets of the asparagusic acid tag. Our results show direct covalent bond formation between the AspA tag and cysteines 556 and C558 on the surface of the transferrin receptor (TFRC) resulting in subsequent endocytic uptake of the cargo. We believe that the small size, low cellular toxicity and the efficient transferrin receptor-mediated uptake render the AspA tag highly attractive for various life science applications.



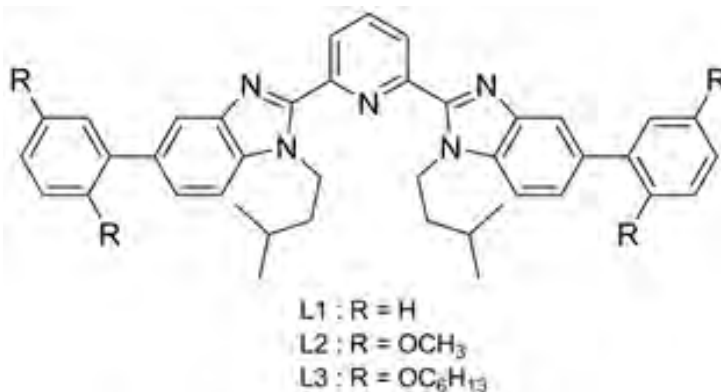
## How the chemical potential of the solvent acts in intermolecular association processes

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Thermodynamic laws consider solvent as having a constant activity when binding equilibria occur in diluted solutions. However the neglect of solvent molecules involved in the solvation reactions may be detrimental to the comprehension of intermolecular association processes. In this context, Castellano and Eggers<sup>1</sup> developed a novel approach, in which solvent molecules were introduced as chemical partners. The explicit consideration of solvent contribution within the frame of the changes in chemical potential results in significant deviations of the law of mass action. Stability constants, which characterize the formation of coordination complexes, are therefore significantly affected. Bearing all these elements in mind, we established theoretical and experimental methods, which aim at separately evaluating solvent effects and “pure” binding affinity accompanying the reaction of tridentate ligands (**L**<sub>1</sub>, **L**<sub>2</sub> and **L**<sub>3</sub>) with saturated trivalent lanthanide carriers [Ln(hfac)3(diglyme)]. Changes in solvent polarity, in ligand lipophilicities and in metallic sizes are investigated for decrypting robust trends in binding selectivity<sup>2</sup>.



Chemical structures of the tridentate ligands **L**<sub>1</sub>, **L**<sub>2</sub>, **L**<sub>3</sub>

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## Palladium-catalyzed enantioselective intermolecular carboetherification and carboamination of dihydrofurans

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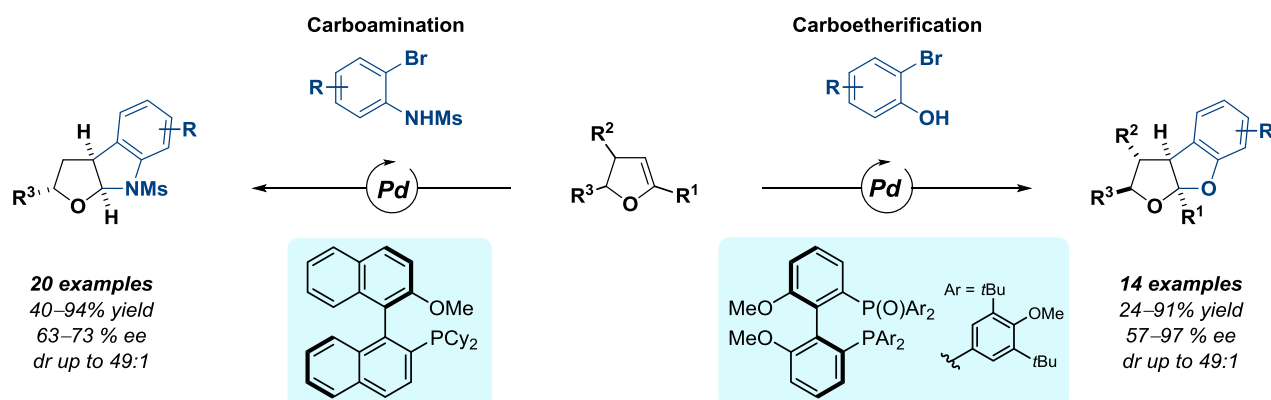


In recent years, efforts have been focused on the development of new methodologies for carbon-heteroatom bond formation<sup>1</sup>, owing to the ubiquity of aryl C-N and C-O bonds in agrochemicals, pharmaceuticals and natural products.

Among these methodologies, the Pd-catalyzed carboetherification of alkenes has emerged as a promising strategy. Despite remarkable advances in the field, most reported examples proceed via intramolecular reactions and their enantioselective variants are still scarce<sup>2,3</sup>.

Herein we describe a novel intermolecular carboetherification that gives direct access to fused tetrahydrofurobenzofurans; a scaffold that can be found in numerous biologically active compounds and which is typically accessible via long and unpractical synthetic routes<sup>4</sup>. Under optimized conditions and using readily available starting materials, the final cross-coupling products are obtained in high yields, enantio- and diastereoselectivity<sup>5</sup>.

Capitalizing on this synthetic strategy, an analogous carboamination methodology has been developed. The final tetrahydrofuroindolines are obtained in practical yields and high diastereoselectivity using substituted dihydrofurans and mesyl-protected anilines<sup>6</sup>.

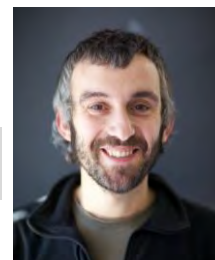


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## Ionophore based emulsions as heterogeneous pH buffer

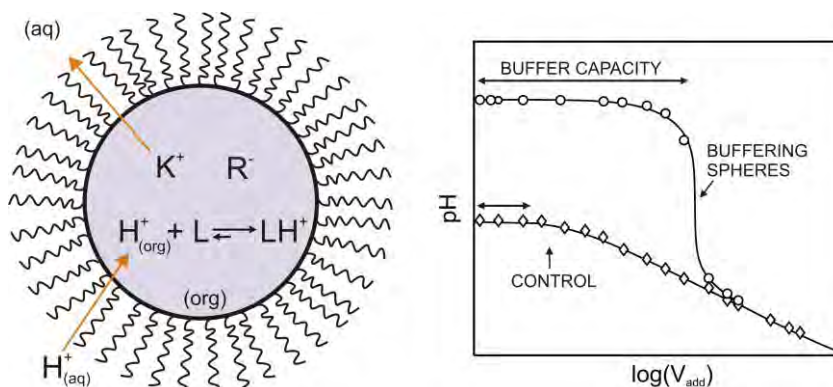
Miguel COLL CRESPI

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A heterogeneous pH buffer system based on a colloidal dispersion for buffering solutions and electrode/solution interfaces is reported here. The dispersion is composed of a selective hydrogen ion receptor, a lipophilic cation exchanger, plasticizer and triblock copolymer. In alignment with this concept, Xie *et al.* introduced ion-selective nanodroplets prepared by solvent displacement (THF) followed by self-assembly of bis(2-ethylhexyl)sebacate (DOS) and pluronic F127 in aqueous solution<sup>1</sup>.

The exchange of hydrogen ions with alkali metal ions between the solution and the high surface area emulsion allows this system to compensate local changes in hydrogen ion concentration by ion-exchange, resulting in a release or uptake of hydrogen ions from the spheres. Each individual sphere works on the basis of reversible ion-exchange chemistry with rapid equilibration time. As a result, such spheres exhibit pH buffer properties that can be predicted and set by adjusting the chemical composition and the initial conditions of the experiment.



The incorporation of these spheres into a hydrogel membrane was explored. Agarose gels with entrapped pH buffer emulsions are shown by potentiometry to exhibit negligible permselective properties above an ionic strength of 1 mM, suggesting that such pH buffers do not give rise to substantial ion-exchange properties of the gel material. In a first attempt to control the pH in the vicinity of an electrode surface by this approach, the emulsion was entrapped in an agarose gel in intimate contact with a pH electrode surface, demonstrating the ability to buffer such gel films. The emulsion-based system may be attractive for exploring a range of dynamic systems that exhibit proton evolution or consumption and where ion-exchange properties of the buffer material are undesired. Furthermore, the compatibility with hydrogels matrices makes them a potentially attractive platform for a range of different science applications where the pH at an interface or in gel layers needs to be precisely controlled.

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## Structural investigation of the HS to LS relaxation dynamics in spin crossover compounds

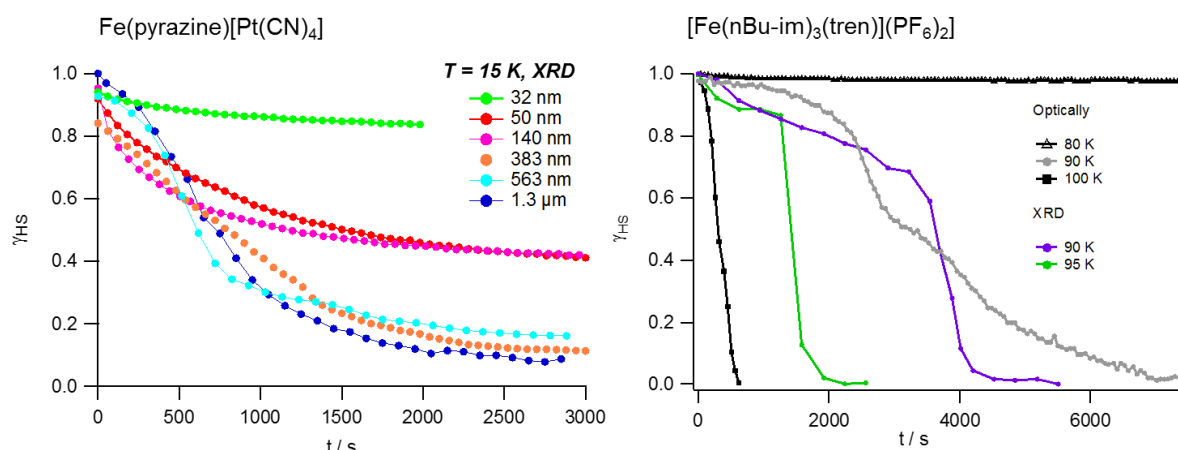
Maria Teresa DELGADO PEREZ

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Spin-crossover compounds are very topical, because of their possible applications in electronic and optical devices<sup>1</sup>. Typically,  $d^4$ - $d^7$  transition metal ions can be converted from the low-spin (LS) to the high-spin (HS) state and vice versa with different external stimuli such as temperature, pressure, light or guest adsorption. At cryogenic temperatures, a photo-induced conversion from the LS stable state to the HS metastable state can be induced through the Light-Induced Excited Spin State Trapping (LIESST) effect<sup>2</sup>.

In our studies Synchrotron X-Ray powder diffraction has been used to follow the structural changes of different compounds after LIESST. In the case of the Hofmann clathrate  $[\text{Fe}(\text{pz})\text{Pt}(\text{CN})_4]$  a complete study on the relaxation behaviour has been performed for six different particle sizes and a strong dependence of the relaxation mechanism with the particle size has been found<sup>3</sup>. In the case of the  $[\text{Fe}(\text{n-Bu-im})_3(\text{tren})](\text{PF}_6)_2$ , for which two different spin crossover behaviours have been observed in the thermal transition depending on the sweeping rate of the temperature<sup>4</sup>, spectroscopic studies show a plateau during the relaxation after LIESST when approximately half of the centres have relaxed back to the LS state indicating a specific structural feature at this composition. Synchrotron X-Ray powder diffraction reveals the occurrence of an order/disorder transition.



HS  $\rightarrow$  LS relaxation after LIESST at 10 K followed at different temperatures for the spin crossover complexes studied.

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## Excited-state symmetry breaking: Toward asymmetrical photochemistry

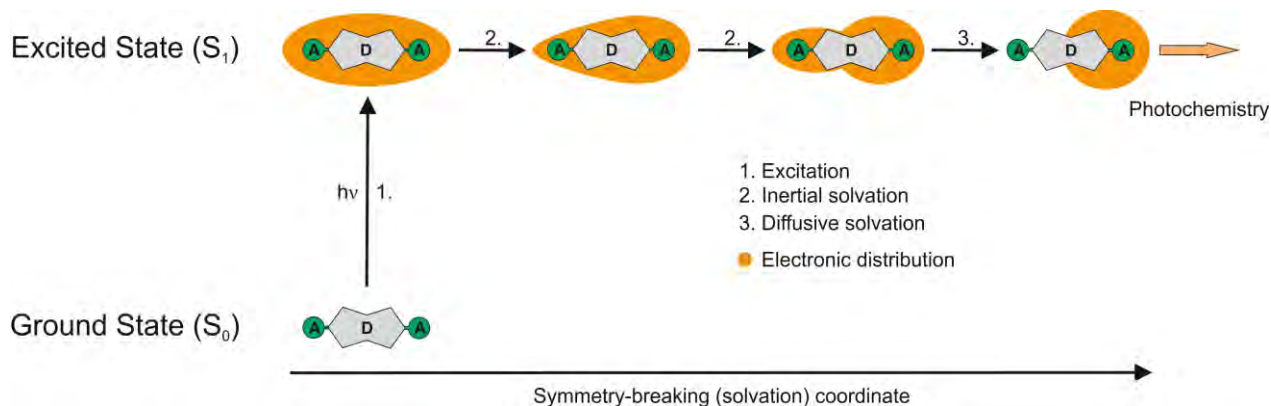
Bogdan DEREKA

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Symmetry breaking has been invoked to explain an anomalous behaviour of many quadrupolar molecules in the electronic excited state. However, for a long time it remained a purely theoretical concept as its real-time experimental observation was missing. Recently we have shown that ultrafast transient infrared (TRIR) unveils clear spectroscopic signatures of symmetry breaking and is capable of following it step by step with femtosecond time resolution<sup>1</sup>.

The optically populated excited state of these molecules is symmetric and quadrupolar and remains as such in non-polar environments where the solvent field is low. In polar media, initial solvent asymmetry around the chromophore and solvent fluctuations drive the molecule toward a symmetry-broken state where the amount of electronic excitation on the two branches is different. This state can further evolve into a purely dipolar excited state where the whole excitation is localized on one arm. Solvent field and dynamics play a paramount role in driving this process and determining its timescales.



This behaviour is a general feature of a vast majority of quadrupolar molecules. The main practical utility of this process is the ability to modulate the nature of the excited state and to fine-tune the amount of electronic excitation on specific molecular moieties within a large molecular architecture. It can be used to tune the molecular interactions, which can determine the properties of functional systems. This is exemplified by the formation of asymmetric photoproducts that results from an initial symmetry breaking. One example involves hydrogen bonding as the simplest intermolecular interaction leading to the formation of an asymmetric stoichiometric solvent-solute complex where a strong H-bond donating solvent molecule binds to a single arm of the two seemingly identical molecular branches<sup>2</sup>. The nature of such an excited complex and the mechanism of its fast deactivation are revealed by applying a novel methodological approach in transient infrared spectroscopy.

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## Modular and enantiospecific synthesis of cationic [6]helicenes and selective orthogonal functionalizations

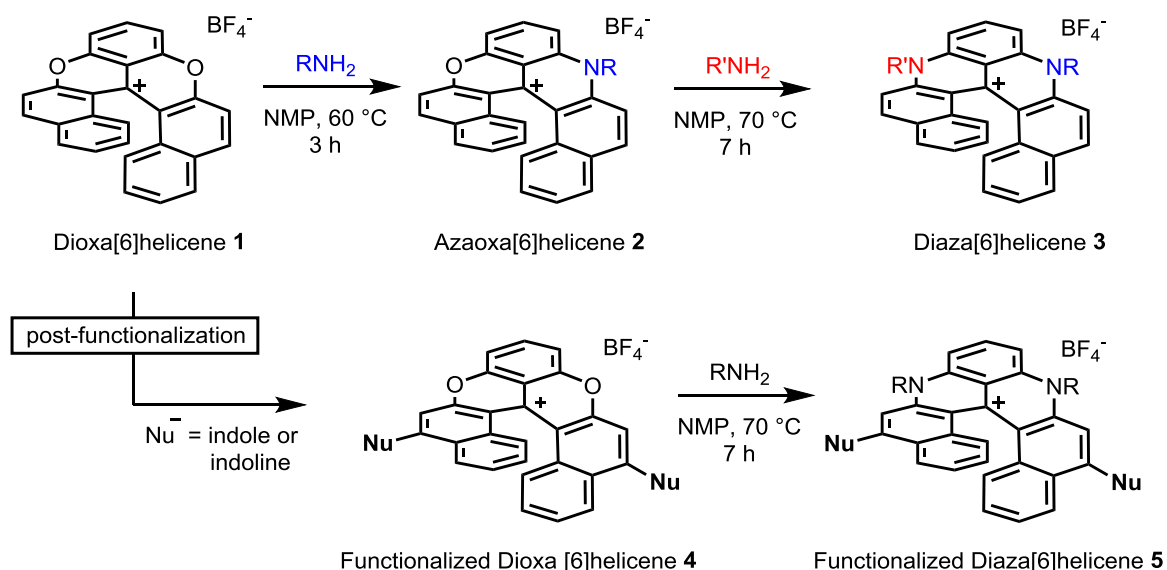
Maria Geraldine LABRADOR BELTRAN

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Helicenes are ortho-condensed polyaromatic compounds that are used broadly from biology to physics<sup>1</sup>. Recently, we have reported a new class of cationic dioxa **1**, azaoxa **2** and diaza **3** [6]helicenes. These stable carbocations are accessible from a common precursor at high temperatures (>140 °C) and can be selectively and orthogonally post-functionalized at will<sup>2,3</sup>.

Herein, dioxa **1** is promoted as precursor in [6]helicene synthesis and the transformation from **1** to **2** and then to **3** is reported at 60 – 70 °C only. Furthermore, using enantioenriched substrates resolved by CSP-HPLC<sup>4</sup>, it is shown that **2** is obtained from **1** in almost racemic form while the transformation from **2** to **3** is essentially enantiospecific (*es* = 94 %).



Moreover, post-functionalization of dioxa **1** can be achieved readily with indoles and indolines as nucleophilic partners. The resulting chromophores **4** are ultimately converted to the diaza analogues **5** using the above detailed strategy. The influence of the peripheral auxochrome substituents is evidenced by a dramatic impact on the optical properties of these cationic fluorophores<sup>5</sup>.

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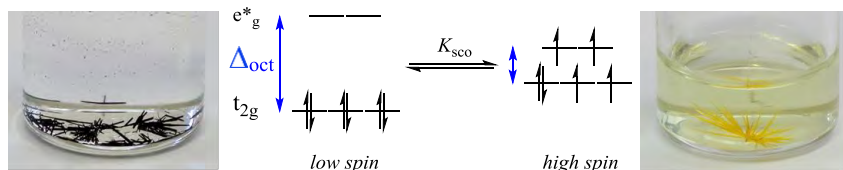
## Iron(II) spin crossover in mono and dinuclear *d-f* complexes

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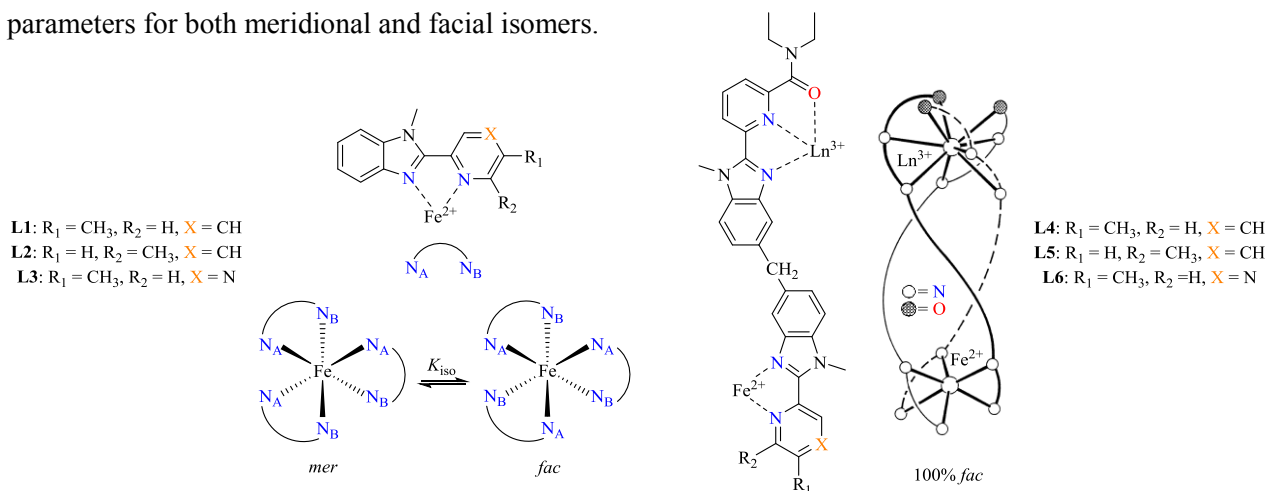


Since their discovery, iron(II) spin crossover (SCO) complexes have been extensively studied for their magnetic and optical properties. In those complexes,



$3d^6$  iron(II) displays a chemical equilibrium between the low spin ( $^1A_{1g}$ ,  $S = 0$ , diamagnetic) and high spin ( $^5T_{2g}$ ,  $S = 2$ , paramagnetic) electronic configurations.

A subtle balance between ligand-field splitting and spin-pairing energies controls the thermodynamic parameters of the SCO process, which makes these complexes challenging for chemical tuning. In this work, the iron(II) spin transitions occurring in the mononuclear  $[\text{Fe}(\text{Lk})_3]^{2+}$  ( $\text{Lk} = \text{L1}, \text{L3}$ ) complexes were studied both in solution and in the solid state in order to evaluate the effects of intermolecular packing on the enthalpic and entropic contributions. Moreover, the use of non-symmetrical didentate ligands **L1**, **L3** open novel avenues for programming SCO transition temperatures because the resulting complexes exist as two structural isomers<sup>1</sup> (meridional and facial). The influence of this considerable constraint on SCO spin transitions mainly escaped attention in this well-explored field, this probably because of the very difficult (impossible?) isolation of pure isomers for labile coordination complexes. Taking advantage of the templating effect<sup>2</sup> brought by trivalent lanthanides,  $\text{Ln}^{\text{III}}$  in the triple-stranded helicates  $[\text{LnFe}(\text{Lk})_3]^{5+}$  ( $\text{Lk} = \text{L4}, \text{L6}$ ), this challenge is solved with the estimation of complete sets of thermodynamic SCO parameters for both meridional and facial isomers.



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## DNA probes at liquid interfaces

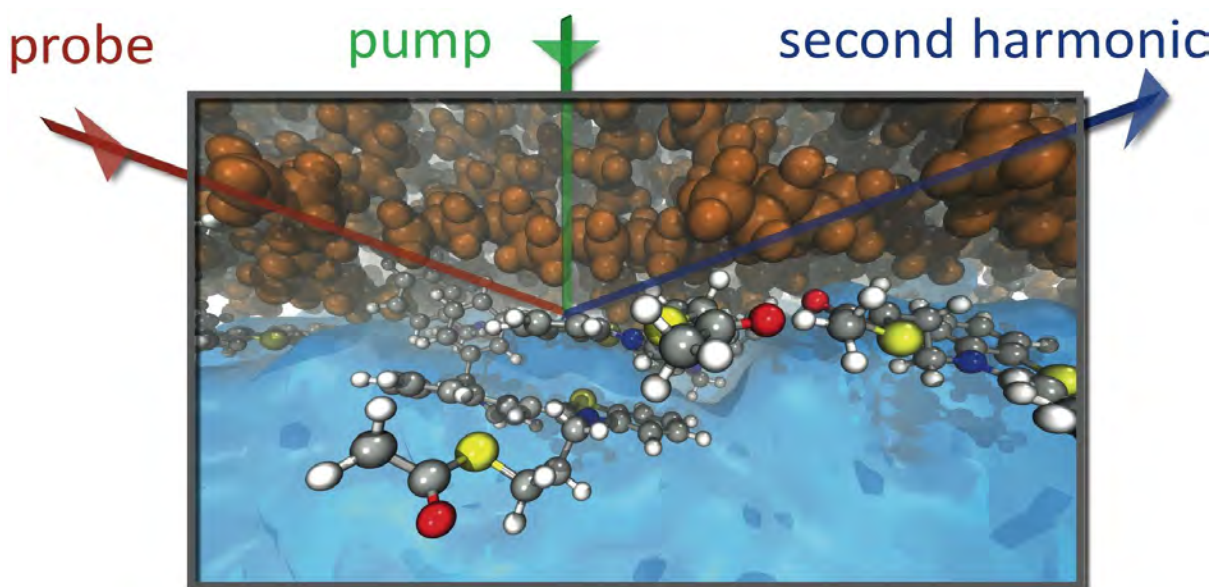
Giuseppe LICARI

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The interfaces between two isotropic liquids, such as membranes in a living cell, are of primary importance in many areas of science and technology. Nevertheless, the properties of interfaces are not fully understood because the signal is buried by the bulk response. We are spectroscopically investigating surface-active compounds using one of the few interface-specific techniques, Surface Second Harmonic Generation (SSHG)<sup>1</sup>. The dipolar origin of SSHG forbids generation of the signal from media with inversion symmetry and therefore can be used to answer fundamental questions concerning interfaces.

We have performed SSHG investigations on a series of yellow oxazole cyanine dyes, which belongs to a well-known class of DNA probes<sup>2</sup>. SSHG revealed that the behavior of the dyes can be very different from that in the bulk. Many information regarding the adsorption, aggregation, orientation and dynamics can be extracted from the experiments<sup>3,4</sup>. Moreover, computational methods such as Density Functional Theory and Molecular Dynamics have been combined to the SSHG data to obtain a better microscopic picture of the interfacial processes.



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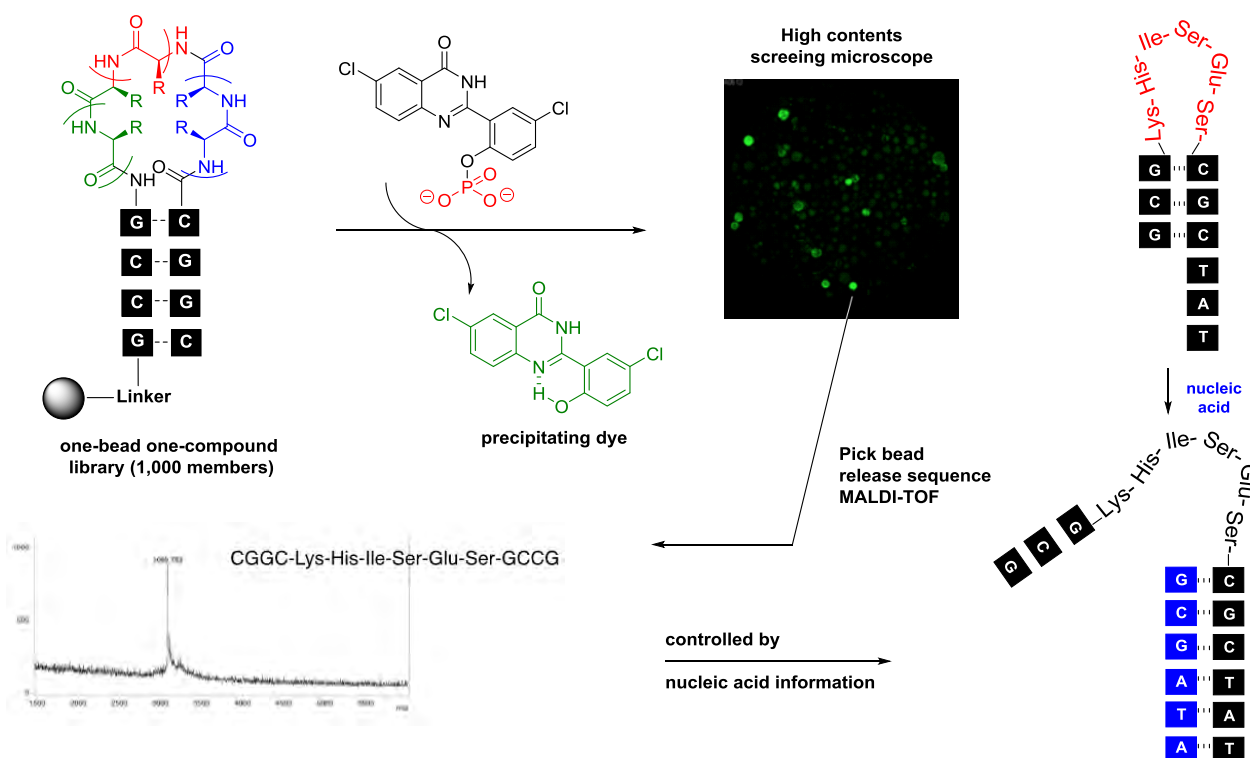
## Phosphatase activity screening based on constrained peptide-peptide nucleic acid (PNA) conjugates<sup>1</sup>

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The importance of spatial organization in short peptide catalysts is well recognized<sup>2</sup>. We designed and screened peptide-PNA conjugate library such that the peptide would be constrained in a hairpin loop upon hybridization. To identify phosphatase like activity, a fluorescence precipitating dye which is turned on by phosphate hydrolysis was used to screen 1,000 one-bead one-compound peptide-PNA library. Bright beads monitored by high contents screening microscope were picked up and the sequences were de-convoluted by MALDI-TOF mass. Hit sequence had hybridization dependent activity and the activity could be controlled by toe hold strand displacement with complimentary sequence.



Schematic representation of 1,000 peptide-PNA library screening for phosphatase activity.

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## The anion- $\pi$ interaction: A tuneable non-covalent interaction for catalysis

François MIROS

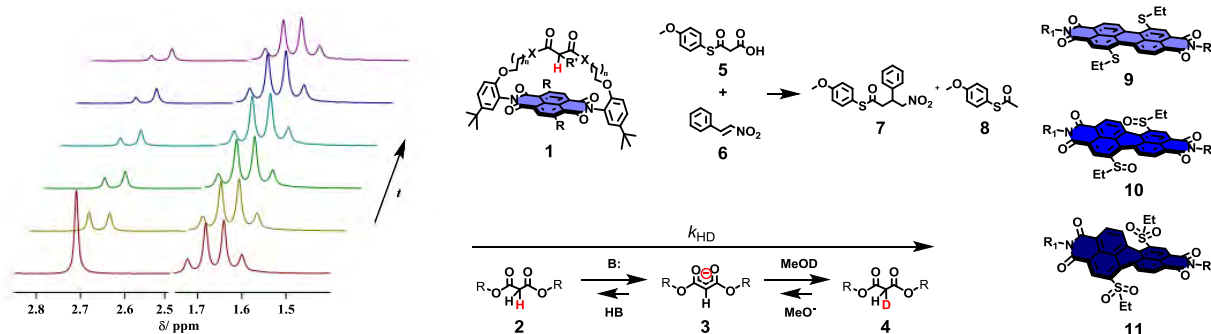
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The now well established exotic anion- $\pi$  interaction has been used to stabilize anionic transition states of a variety of reactions<sup>1</sup>. However, direct quantification of this transition state stabilization and “ $\pi$ -acid strength” requires further study.

Macrolactones **1** comprising a variety of core-substituted NDIs and covalently linked malonate bridge were created<sup>2</sup>. Exchange of the most acidic malonate bridge protons with a deuterium source was recorded through <sup>1</sup>H NMR spectroscopy to obtain exchange rates. As the malonate protons of **2** are removed with base, anionic enolate **3** above the NDI surface is formed. NDIs should thus stabilize the enolate, promoting exchange towards **4**. Rates were compared with a control to obtain stabilization energies of 19 kJ/mol and  $\Delta pK_a \sim 6.0$  with the strongest NDI  $\pi$ -acids. This suggests that increases in malonate acidity are due to improved enolate stabilization by strong anion- $\pi$  interactions.

Larger perylenediimides (PDIs) were also studied. PDI catalysts for malonic acid half thioester (MAHT) **5** were made.<sup>3</sup> Results with NDIs favor formation of **7** over **8** with increasing  $\pi$ -acidity through oxidation of sulfide to sulfoxide and sulfone.<sup>4</sup> PDIs showed a reverse trend, attenuated to twisting of the PDI core due to the increase in substituent size from sulfide **9** to sulfoxide **10** and sulfone **11**. This study suggested that  $\pi$ -system deplanarisation disrupts the anion- $\pi$  interaction.



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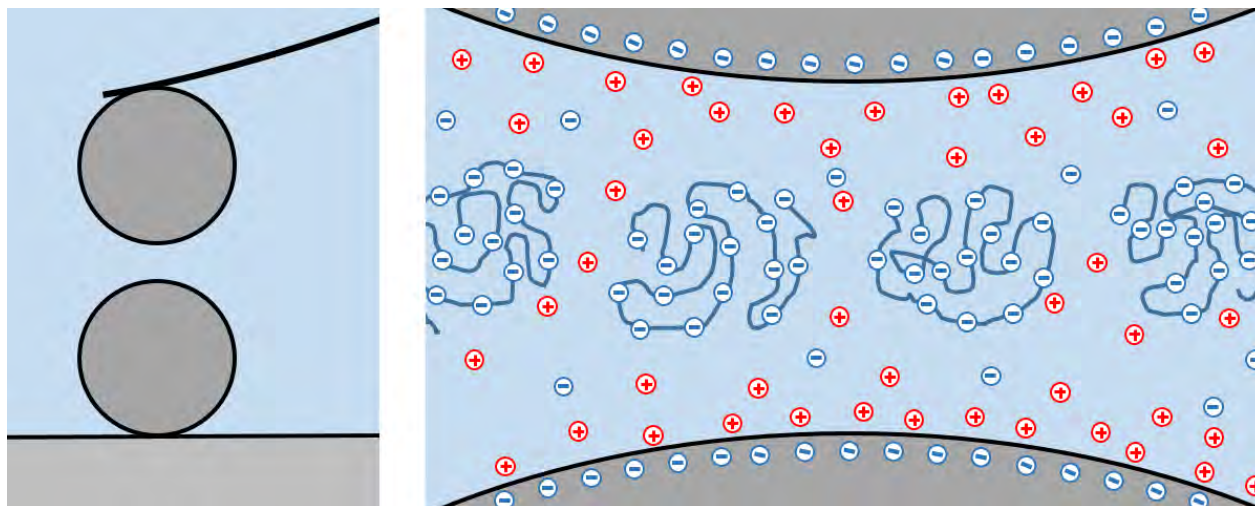
## Depletion and double layer forces in the presence of like-charged polyelectrolytes

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The colloidal probe technique was used to measure interaction forces between silica particles in aqueous solutions of negatively charged polyelectrolytes and monovalent salt<sup>1</sup>. The measured forces can be quantitatively interpreted through a superposition of depletion and double layer forces. The depletion forces appear in the form of a damped oscillatory profile due to the depletion and structuring of like-charged polyelectrolyte at the silica-water interface. The double layer forces can be accurately described using Poisson-Boltzmann theory for a mixture of monovalent symmetric salt and a highly asymmetric electrolyte, whereby the multivalent coions represents the polyelectrolyte chains. The valence of the asymmetric coions depends on molecular mass of polyelectrolyte but it is smaller than the nominal number of charged group of one polyelectrolyte molecules. This is consistent with counterion condensation. The highly charged nature of polyelectrolyte molecules led to their exclusion from the vicinity of the interface and in turn formation of highly non-exponential diffuse ionic layer and double layer forces. Exclusion of polyelectrolyte molecules from the interface forms a depletion layer where oscillatory force sets in. The phase of oscillation can be predicted through the interplay of double layer and depletion forces.

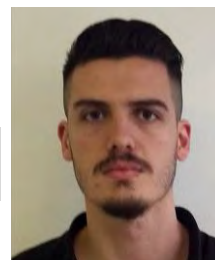


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## Stabilization of enzyme-clay nanocomposite in dispersion by heparin adsorption

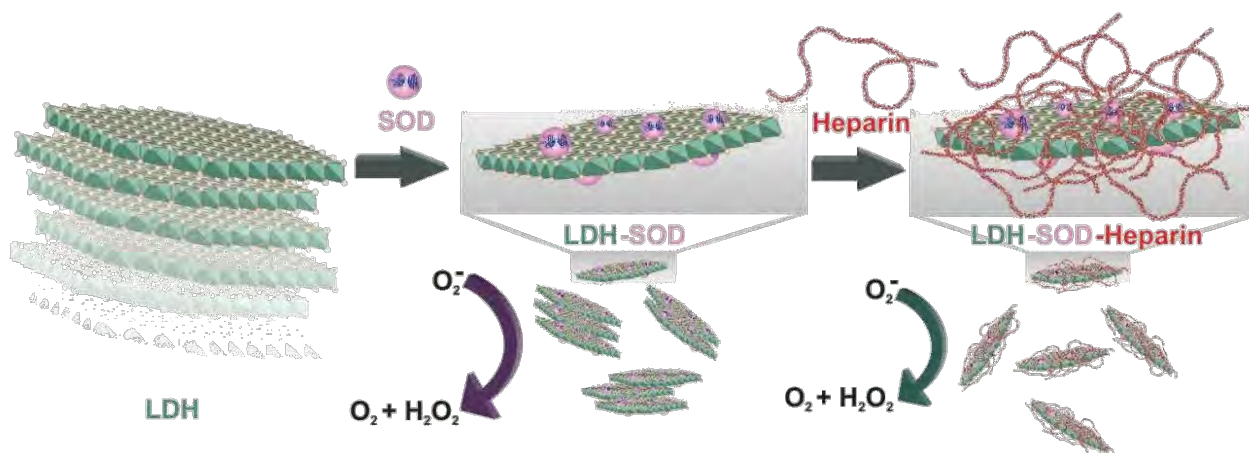
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Enzyme delivery in biomedical processes is challenging due to the complex environment in the biofluids. Immobilizing enzymes on nanoparticles may lead to successful targeted transport and can also protect the biocatalysts. However, colloidal stability has to be well-controlled since the aggregation of the carrier particles gives rise to inefficient delivery<sup>1</sup>.

The present study focuses on a preparation of hybrid nanomaterial by combining layered double hydroxide (LDH) nanoparticles as a solid support, superoxide dismutase (SOD) as attached enzyme and biocompatible polyelectrolyte, heparin, as additional stabilizing agent. Structural features of the novel bionanomaterial was studied by XRD, various spectroscopic methods (IR, UV-Vis and fluorescence) and TEM, while colloidal stability of the obtained materials was investigated by electrophoresis and light scattering in aqueous dispersions. The SOD quantitatively adsorbed on the LDH by electrostatic and hydrophobic interactions and kept its integrity upon immobilization. The composite material showed moderate resistance against salt-induced aggregation in dispersions, therefore, polyelectrolyte was used to improve the colloidal stability of the system. Heparin of highly negative line-charge density strongly adsorbed on the oppositely charged hybrid particles leading to charge neutralization and overcharging at appropriate polyelectrolyte loading<sup>1</sup>. Obtained LDH-SOD-Hep nanocomposites showed good SOD-like activity. Due to the improved colloidal stability, the developed enzymatic system is a promising antioxidant candidate for biomedical or other manufacturing processes wherever the aim is to decompose reactive oxygen species in suspensions.



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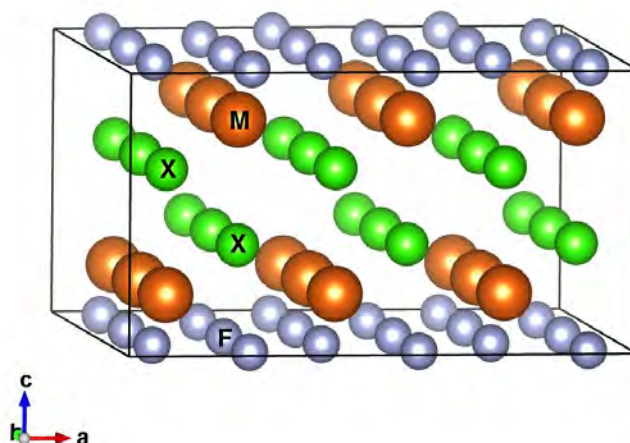
## Influence of van der Waals dispersion correction on the DFT description of alkaline-earth fluorohalides

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Alkaline-earth fluorohalides, MFX, are receiving much attention in material and optical sciences owing to the remarkable photophysical and photochemical properties, which they can exhibit upon incorporation of photoactive rare-earth ions<sup>1</sup>. The MFX host compounds crystallize in the tetragonal  $P4/nmm$  Matlockite structure: they exhibit a layered ionic structure which corresponds to a simple  $F^- - M^{2+} - X^- - X^- - M^{2+} - F^-$  stacking of the ion layers along the  $c$  axis (see Figure below). Due to their layered structure and especially the presence of the anionic double layer, the description of the structures and properties of the MFX compounds within density functional theory (DFT) may suffer from the inaccurate description of the dispersion interactions, which can be observed with local, semilocal, and hybrid density functionals<sup>2</sup>. This deficiency of standard approximate functionals could indeed explain the overestimation of the  $c$  parameter reported for  $PbFCl$ <sup>3</sup>. To investigate this question, periodic DFT calculations have been performed to study the crystal structures, the vibrational frequencies, the bulk modulus ( $B_0$ ) and its derivatives ( $B_0'$ ), and the mode Grüneisen parameters of the MFX compounds ( $M = Ca, Sr, Ba, Pb$  and  $X = Cl, Br, I$ ). The PBE and B3LYP functionals and their DFT-D2 dispersion-corrected variants<sup>4,5</sup> were employed. For these layered ionic compounds, it proved necessary to modify the semi-empirical D2 dispersion correction.



The tetragonal  $P4/nmm$  Matlockite structure of MFX alkaline-earth fluorohalides :  
3 x 3 x 1 supercell showing the layer stacking.

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## Stochastic switching of Min proteins in short *Escherichia coli* cells

Lukas WETTMANN

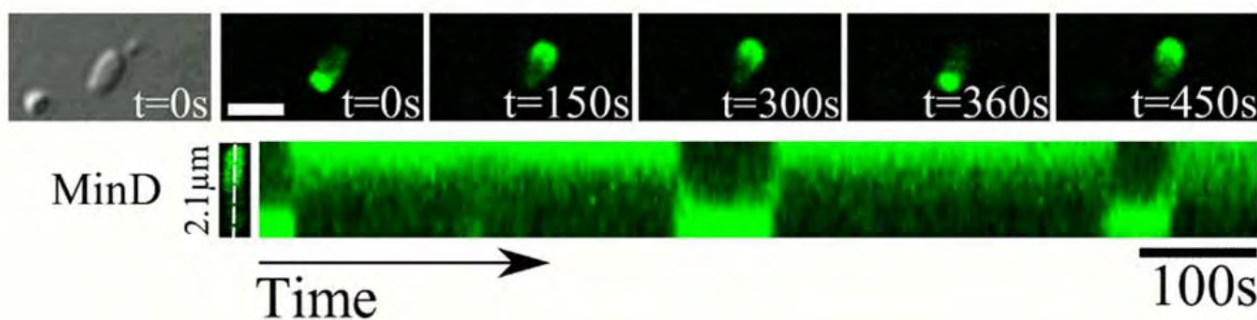
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Intracellular processes are subjected to noise, be it through thermal fluctuations or, for example, molecular noise. The latter case is especially true when the total number of involved molecules inside the cell is low.

One important process influenced by molecular noise is gene expression, where small changes in the protein number can cause phenomenological changes of the cell itself. More interesting than the, for the case of gene expression, local changes is the stochastic behaviour of spatially inhomogeneous protein distributions inside the cell.

To this end, we study the influence of noise on the dynamics of the Min system. The Min proteins are a family of proteins which through self-organization are able to exert spatial oscillations in rod-shaped *E. coli* cells, ranging from one pole of the cell to the other. These oscillations cause the division site to be located along the symmetry axis of the cell, ensuring equal-sized daughter cells. While this is the case for wild type cells, there are several other patterns which have been studied in experiments. For example, in vitro, travelling waves have been observed.



In contrast, for short cells, the oscillations are replaced by stochastic switching of the proteins between two stable polar configurations<sup>1</sup>. This behaviour can cause the emergence of mini cells if the residence times are sufficiently long.

We developed a mechanism based on the underlying molecular processes to study the dynamics of the Min proteins. With this mechanism, we are able to reproduce all of the patterns found in experiments including the case of two symmetric stable states. We then use a framework developed in earlier work<sup>2</sup> to analyse the behaviour of the Min proteins in the limit of weak noise and calculate the residence times as a function of the cell length.

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## Voltammetric thin layer ionophore based membranes for ion activity detection: Two sensing modes

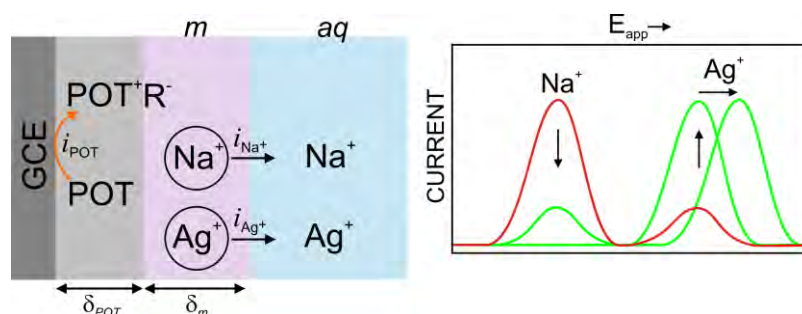
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The benefit of using electrodes based on thin layer ionophore-based membranes (~200 nm) backside contacted with a film of poly(3-octylthiophene) (POT) (~50 nm) for multi-analyte detection has been recently demonstrated<sup>1</sup>. An anodic potential scan partially oxidizes the POT film, thereby initiating the release of hydrophilic cations from the membrane to the sample solution at a certain potential. This ion transfer potential is related to the selectivity of the membrane and an interesting pattern is observed with the presence of multiple ionophores that are selective for different cations (such as lithium, potassium and sodium). Since the cation-ionophore binding constants are distinct, voltammograms based on multiple peaks (one for each cation) are obtained allowing multi-analyte detection in real samples<sup>2</sup>.

Interestingly, the electrode can operate under two sensing modes depending on the ion concentration in the sample solution. At high concentrations, the membrane behaves under thin layer control and consequently the peak potential shifts to more positive values for increasing ion concentrations in analogy to a potentiometric sensor<sup>1</sup>. On the other hand, at lower concentrations, ion-transfer processes through the membrane depend on mass transport in the solution, resulting in an increase of peak current with ion concentration similar to voltammetric sensors. While the potentiometric readout can be used for multi-analyte detection as aforementioned, the voltammetric mode is useful for lowering the limit of detection of the sensor using stripping voltammetry<sup>3</sup>.



In the present work, we explored the limitations of the use of these two sensing modes. A silver-selective membrane is selected as proof-of-concept. Apart from experimental evidences, a numerical simulation is developed considering an idealized behavior of the conducting polymer. This approach allows one to predict the experimental behavior of the electrode under either diffusion or thin layer control<sup>4</sup>. Finally, a simplified semi-empirical treatment is developed and permits to quite easily predict relevant experimental conditions for this emergent methodology<sup>5</sup>.

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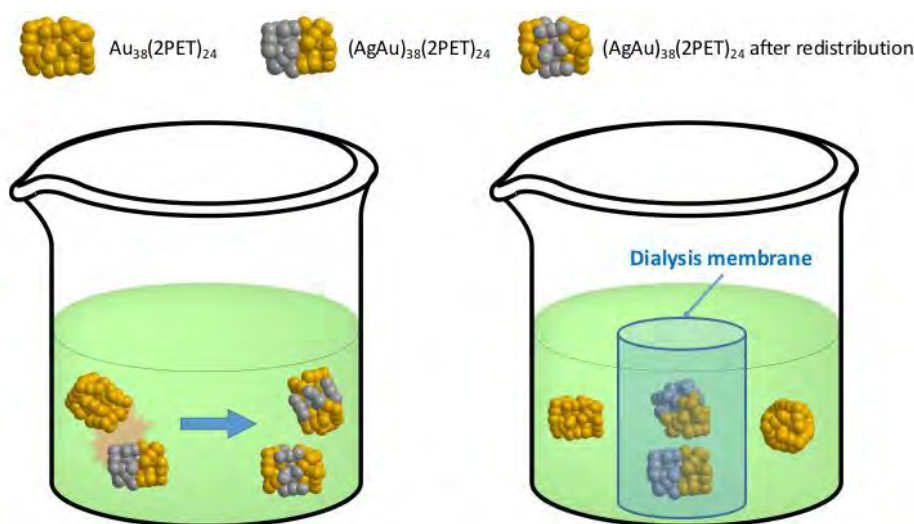
## Ag doped $\text{Au}_{38}(\text{SR})_{24}$ nanocluster: Surface flexibility and silver migration

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Thiolate-protected gold clusters and heteroatoms doped gold clusters have attracted increased attention by their well-defined structure and size-dependent properties<sup>1</sup>. Among them, the  $\text{Au}_{38}(\text{SR})_{24}$  cluster bears intrinsically chiral features due to the arrangement of the protecting ligands on the surface of the cluster<sup>2</sup>. Enhanced stability and surface flexibility of gold nanoclusters have been achieved by doping hetero metal atoms inside  $\text{Au}_{38}$ <sup>3</sup>. However, the effect of the number of dopant atoms on the nanocluster stability and flexibility has not been fully understood. Multiple Ag atoms were doped inside  $\text{Au}_{38}$  by time-dependent metal exchange. The temperature required for complete racemization of  $\text{Ag}_x\text{Au}_{38-x}$  enantiomer follows  $\text{Au}_{38} > \text{Ag}_x\text{Au}_{38-x}$  ( $x=6.5$ )  $>$   $\text{Ag}_x\text{Au}_{38-x}$  ( $x=7.9$ ), which shows an increased flexibility of the cluster with increasing silver content. Unexpected spontaneous redistribution of Ag atoms occurs upon mixing between  $\text{Ag}_x\text{Au}_{38-x}$  and  $\text{Au}_{38}$  nanoclusters in solution, as observed by MALDI. Physical separation of  $\text{Ag}_x\text{Au}_{38-x}$  and  $\text{Au}_{38}$  species by a dialysis membrane prohibits the metal migration, which suggests that collisions between the reacting clusters are at the origin of the redistribution of Ag atoms (Figure).



**Left:** Reaction between  $\text{Au}_{38}$  and  $\text{Ag}_x\text{Au}_{38-x}$  that led to silver migration among the clusters.

**Right:** Reaction in the presence of a dialysis membrane that prevents the direct physical contact between  $\text{Au}_{38}$  and  $\text{Ag}_x\text{Au}_{38-x}$ .

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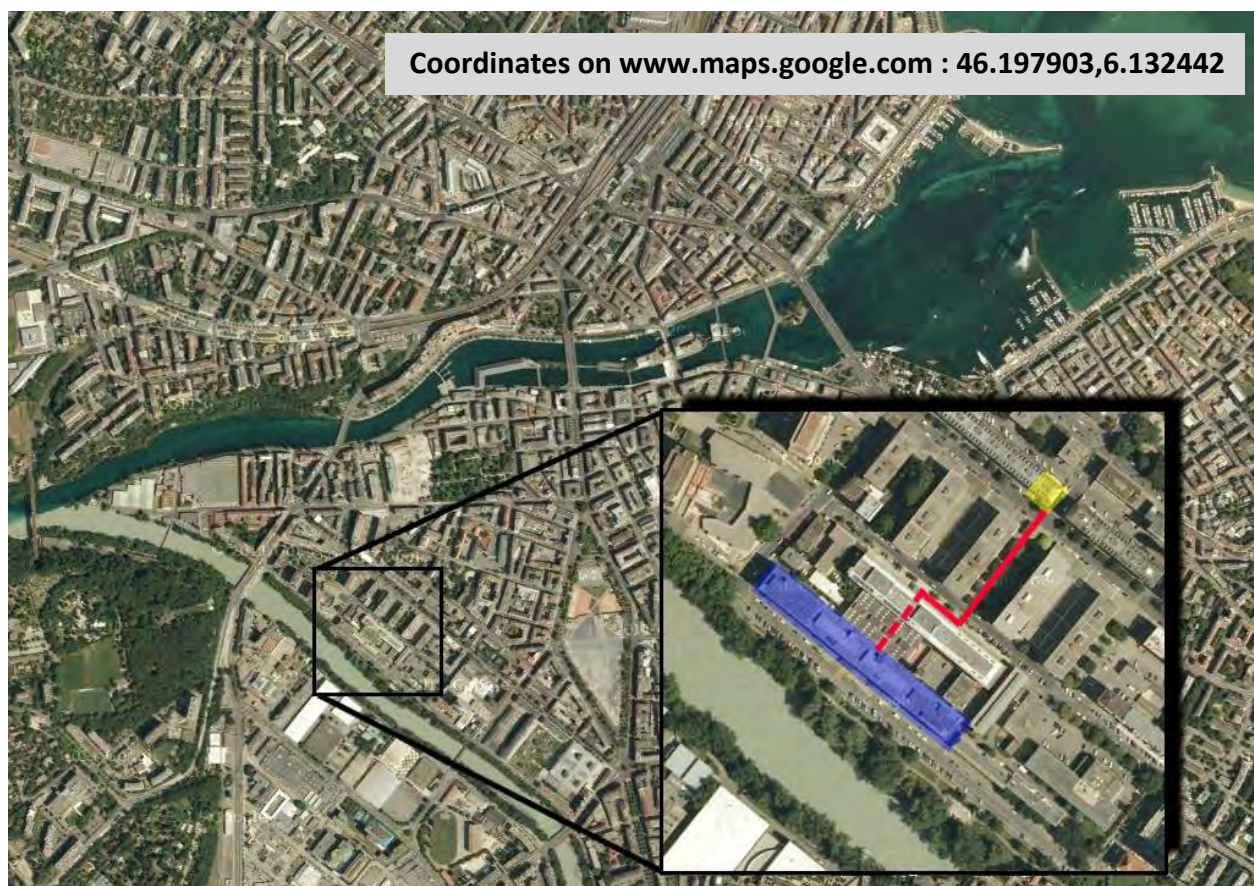
## THE SCHOOL OF CHEMISTRY AND BIOCHEMISTRY

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- Elaboration of new supramolecular architectures on the nanometre scale with promising microscopic and macroscopic properties.
- Development of analytical techniques surpassing today's frontiers of precision, in order to identify ultra-trace species in complex environments.
- Development and optimisation of alternatives to fossil fuels as sources of energy.
- Development of highly selective and environmentally benign methods of organic synthesis.
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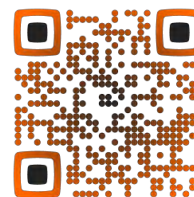
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