

# Formation of Ultrathin, Continuous Metal–Organic Framework Membranes on Flexible Polymer Substrates

Jingwei Hou, Putu D. Sutrisna, Yatao Zhang, and Vicki Chen\*

**Abstract:** Metal–organic framework (MOF) materials have an enormous potential in separation applications, but to realize their potential as semipermeable membranes they need to be assembled into thin continuous macroscopic films for fabrication into devices. By using a facile immersion technique, we prepared ultrathin, continuous zeolitic imidazolate framework (ZIF-8) membranes on titania-functionalized porous polymeric supports. The coherent ZIF-8 layer was surprisingly flexible and adhered well to the support, and the composite membrane could sustain bending and elongation. The membranes exhibited molecular sieving behavior, close to the theoretical permeability of ZIF-8, with hydrogen permeance up to  $201 \times 10^{-7} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$  and an ideal  $\text{H}_2/\text{CO}_2$  selectivity of 7:1. This approach offers significant opportunities to exploit the unique properties of MOFs in the fabrication of separation and sensing devices.

Molecular sieving membranes are promising candidates for applications that require highly selective permeation, such as gas separation, organic solvent nanofiltration, water treatment, and chemical sensors.<sup>[1,2]</sup> The key to their successful implementation is the synthesis of a thin, but defect-free, selective layer. As a consequence of their large porosity, molecule-scale pore size, and good stability, metal–organic frameworks (MOFs) have been extensively studied for their potential as molecular sieving membranes,<sup>[3]</sup> mostly by coating them onto inorganic porous supports.<sup>[4]</sup> However such membranes are hampered considerably by their complex processing, high cost, rigidity, and brittleness. The formation of thin, continuous MOFs on porous polymeric supports has been difficult because of the lack of sufficient heterogeneous nucleation sites and adhesion.<sup>[5]</sup> Chemical modifications of the polymer surface or interfacial precipitation have been used to grow MOFs within porous substrates or as isolated crystals.<sup>[6,7]</sup> Recently, atomic-layer deposition has been applied to grow aggregates of MOF crystals directly on solid polymeric fibers for gas adsorption.<sup>[8]</sup> However, a facile approach to grow thin, continuous MOF films directly from

the synthesis solution has not been available for porous flexible substrates such as polymeric membranes. Theoretically, the MOF layers can exhibit a degree of flexibility as a result of the MOF structure changing reversibly under moderate mechanical loading.<sup>[9]</sup> However, this has yet to be demonstrated in continuous, macroscopic films that act as semipermeable membranes. To achieve this, two major hurdles need to be considered: good adhesion of MOFs on flexible, porous supports and formation of a uniform, coherent film.<sup>[3]</sup>

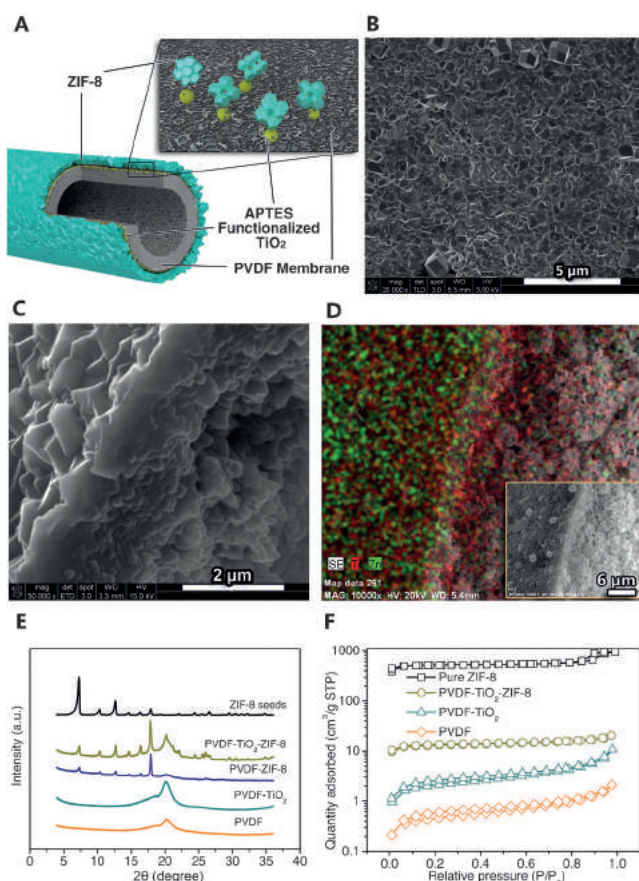
Our group has developed a simple low-temperature hydrothermal sol-gel coating technique to deposit an ultrathin layer of  $\text{TiO}_2$  with a controllable architecture on various polymer supports. The functionalization of the coating layer has been explored for antifouling properties, enzyme immobilization, and superhydrophobicity.<sup>[10,11]</sup> We postulated that the large surface area, nanoscale roughness, and hydrophilicity of the titania layer would promote heterogeneous crystallization on the support. Hence, we explored the formation of MOFs (specifically, ZIF-8) on APTES-functionalized (APTES = (3-aminopropyl)triethoxysilane)  $\text{TiO}_2$ -coated hollow-fiber PVDF (PVDF = polyvinylidene fluoride) membranes with a pore size of  $0.05 \mu\text{m}$  (Figure 1 A). ZIF-8 is a promising candidate for the separation of olefins and hydrogen gas, water treatment, and chemical sensors<sup>[2,12]</sup> (the fabrication process is presented in Figures S1 and S2). The tunable  $\text{TiO}_2$  coating and functionalization conditions can be used for the facile coating of a wide range of polymeric membrane structures, including flat sheets and hollow fibers. The thin and uniform  $\text{TiO}_2$  coating layer has dual hierarchical roughness and provides OH groups for additional functionalization (Figure S3 A,B). The  $\text{TiO}_2$  coating layer has been demonstrated to be stable under significant shear forces and harsh physical/chemical conditions.<sup>[11]</sup> Further functionalization with 2 wt % APTES forms an organosilane monolayer that provides abundant amine groups without compromising the nanoscale architecture of the  $\text{TiO}_2$ .<sup>[13]</sup>

We next deposited ZIF-8 onto PVDF membranes functionalized with APTES-titania by both preseeded growth and direct immersion in the synthesis solution. No apparent difference in the morphology of the ZIF-8 layer was observed, thus the direct immersion without preseeded was applied subsequently. We observed that a continuous polycrystalline ZIF-8 layer about  $1 \mu\text{m}$  thick forms on the supporting membrane surface without any pinholes or cracks after 5 h immersion in a simple ZIF-8 synthesis mixture (Figure 1 B,C). The interface between the ZIF-8 and titania-PVDF support indicates that they are highly intercalated, which ensures stronger bonding. We postulated that the APTES-functionalized  $\text{TiO}_2$  nanoparticles help nucleate the ZIF-8 layer

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**Figure 1.** Description of the ZIF-8 molecular sieving membrane on a PVDF support. A) Schematic representation of the composite membrane. B,C) Surface and cross-sectional SEM images of the ZIF-8 layer. D) EDX elemental map of Ti (red) and Zn (green) (insert: reference SEM image). E,F) XRD patterns and nitrogen adsorption/desorption isotherms of the ZIF-8 crystal and the membranes with different treatment.

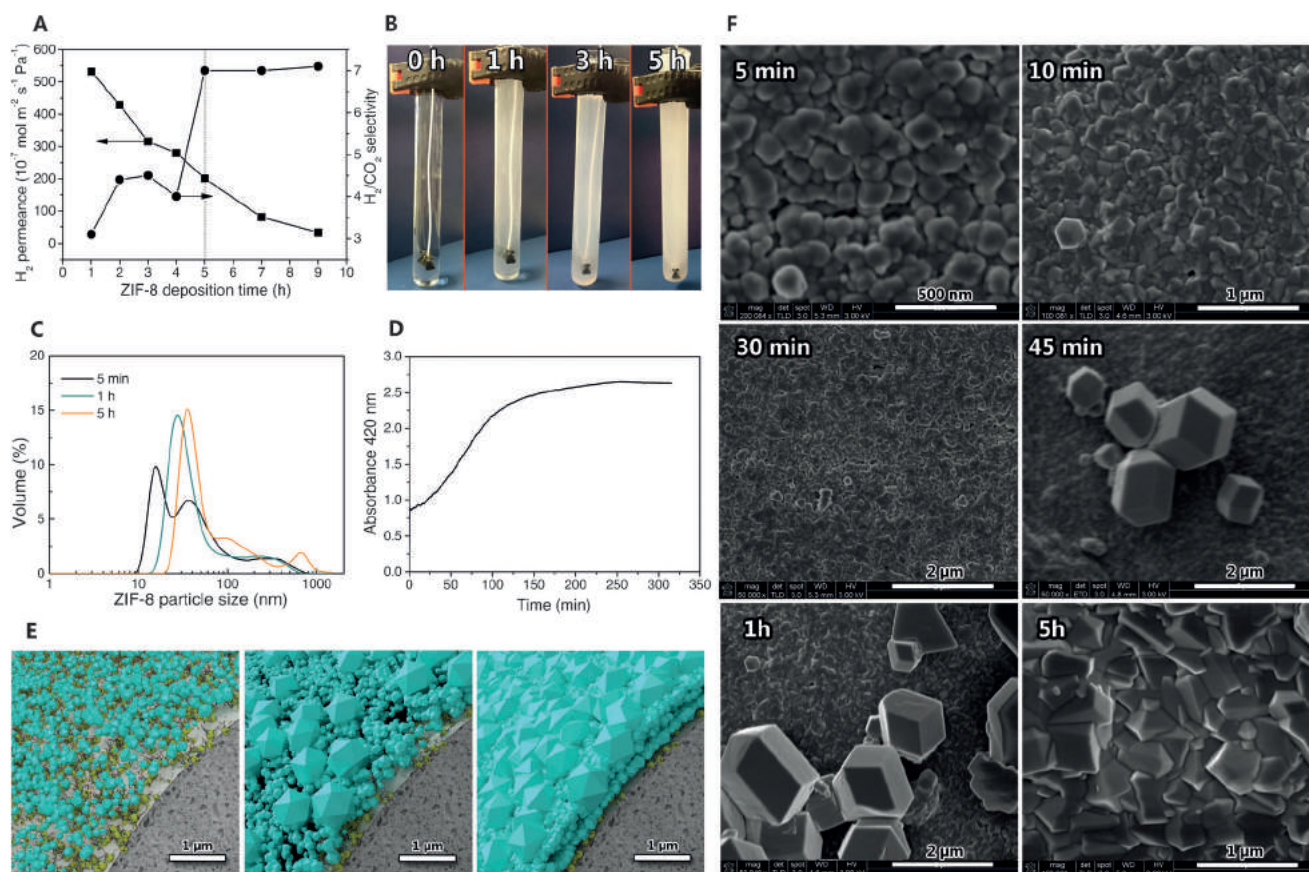
growth through the terminal amine groups which react with the  $\text{Zn}^{2+}$  ions to form complex metal cations.<sup>[6,14]</sup> In addition, the nanostructured titania provides a high surface area, and thus better contact and anchoring during crystallization (Table S1), thereby promoting the nucleation density of ZIF-8 on the polymer membrane surface. Such a high heterogeneous nucleation density promotes the formation of a thin and continuous ZIF-8 membrane.<sup>[15]</sup> Attempts to grow ZIF-8 on pure PVDF and  $\text{TiO}_2$ -coated PVDF (not functionalized with APTES) resulted in poorly intercalated crystals (Figure S3 C,D).

Seeking to confirm the physical, chemical, and structural properties of the ZIF-8 layer, we examined the ZIF-8 membrane by various characterization techniques. EDX (Figure 1D) shows that the Ti is evenly distributed throughout the membrane, while there is a sharp transition of Zn between the ZIF-8 layer and the supporting membrane, thus indicating that the ZIF-8 is mainly present on the membrane surface. Thermogravimetric analysis (TGA; Figure S4) also confirms the low loading of ZIF-8, and the presence of a thin ZIF-8 layer is further supported by the ATR-FTIR results (Figure S5). The XRD patterns (Figure 1E) suggest that the

ZIF structure on the PVDF- $\text{TiO}_2$ -ZIF-8 membrane is highly crystalline. The different relative peak intensity of the PVDF- $\text{TiO}_2$ -ZIF-8 membrane compared with pure ZIF-8, in particular of the (222) plane ( $2\theta = 18.3$ ), can be attributed to the preferential orientation of the crystal growth on the polymer support.<sup>[15]</sup> The gas adsorption isotherms (Figure 1F and Figure S6), together with the results on the surface area (Table S1), are also consistent with the high adsorption area of the ZIF-8 layer.

Gas permeance is usually inversely proportional to the membrane thickness. We hypothesized that a thinner ZIF-8 layer could be obtained by controlling the ZIF-8 deposition time. However, at solution exposure times below 5 h, gas transport is mainly dominated by Knudsen diffusion (Figure 2A and Table S2), thus indicating that the ZIF-8 layer is not continuous. The crystallization of ZIF-8 onto the functionalized PVDF membrane surface immediately resulted in the formation of nanocrystals, which served as the seeds for further crystallization. Microcrystals then formed gradually in both solution and on the membrane surface. The large particles on the membrane ensure efficient surface coverage by ZIF-8, while the fusion of nanoparticles between the large particles sealed the grain boundaries and defects, thereby forming a continuous ZIF-8 layer (Figure 2B–F and Figure S7A). A coherent ZIF-8 layer, formed after 5 h deposition time, clearly displayed molecular sieving properties. A further increase in the deposition time led to a thicker ZIF-8 coating and thus reduced gas permeance (Figure 2A; Table S2 and Figure S7B). Therefore, the ZIF-8 membrane obtained by deposition for 5 h was selected for further study. It has the highest  $\text{H}_2$  permeance among the polymer-supported ZIF-8 molecular sieving membranes (Table S3). Our ZIF-8 membrane has superior performance over most ZIF-8 membranes supported on inorganic membranes, with the exception of the  $\text{Al}_2\text{O}_3$ -supported ZIF-8 produced by contradiffusion methods.<sup>[14]</sup> The ultrahigh  $\text{H}_2$  permeance obtained in the present study can be attributed to the thin and activated ZIF-8 layer as well as the nanoscale surface structure of the ZIF- $\text{TiO}_2$  interface, thereby allowing better contact between the feed gas and the ZIF crystals.<sup>[14,16]</sup> Although the overall ZIF-8 thickness is around 1  $\mu\text{m}$ , the presence of valleys between neighboring microcrystals allows much shorter distances for gas permeance (ca. 400 nm, Figure S8). On the basis of diffusivity measurements using the gas adsorption tests with ZIF-8 crystals, Zhang and co-workers estimated the ideal gas permeability through a ZIF-8 layer ( $\text{H}_2$ : 22000 Barrer,  $\text{CO}_2$ : 3300 Barrer,  $\text{O}_2$ : 2500 Barrer,  $\text{N}_2$ : 1000 Barrer, and  $\text{CH}_4$ : 270 Barrer ( $1 \text{ Barrer} = 10^{-10} \text{ cm}^3 \text{ (STP) cm}^{-1} \text{ s}^{-1} \text{ cmHg}^{-1}$ ).<sup>[17]</sup> Assuming the gas transport is dominated by the thinner part (400 nm) within the ZIF-8 layer, the gas permeability estimated from this present study yielded  $\text{H}_2$ : 24 120 Barrer,  $\text{CO}_2$ : 3444 Barrer,  $\text{O}_2$ : 2940 Barrer,  $\text{N}_2$ : 3096 Barrer, and  $\text{CH}_4$ : 2808 Barrer. The values of  $\text{H}_2$ ,  $\text{CO}_2$ , and  $\text{O}_2$  are in good agreement with the theoretical values, while the higher permeability for  $\text{N}_2$  and  $\text{CH}_4$  can be attributed to the flexibility of the organic ligands on the ultrathin ZIF-8 layer, which allows larger molecules to pass through.<sup>[18]</sup> The gas separation performance easily surpasses the Robeson upper bounds (Figure S9).<sup>[19]</sup>



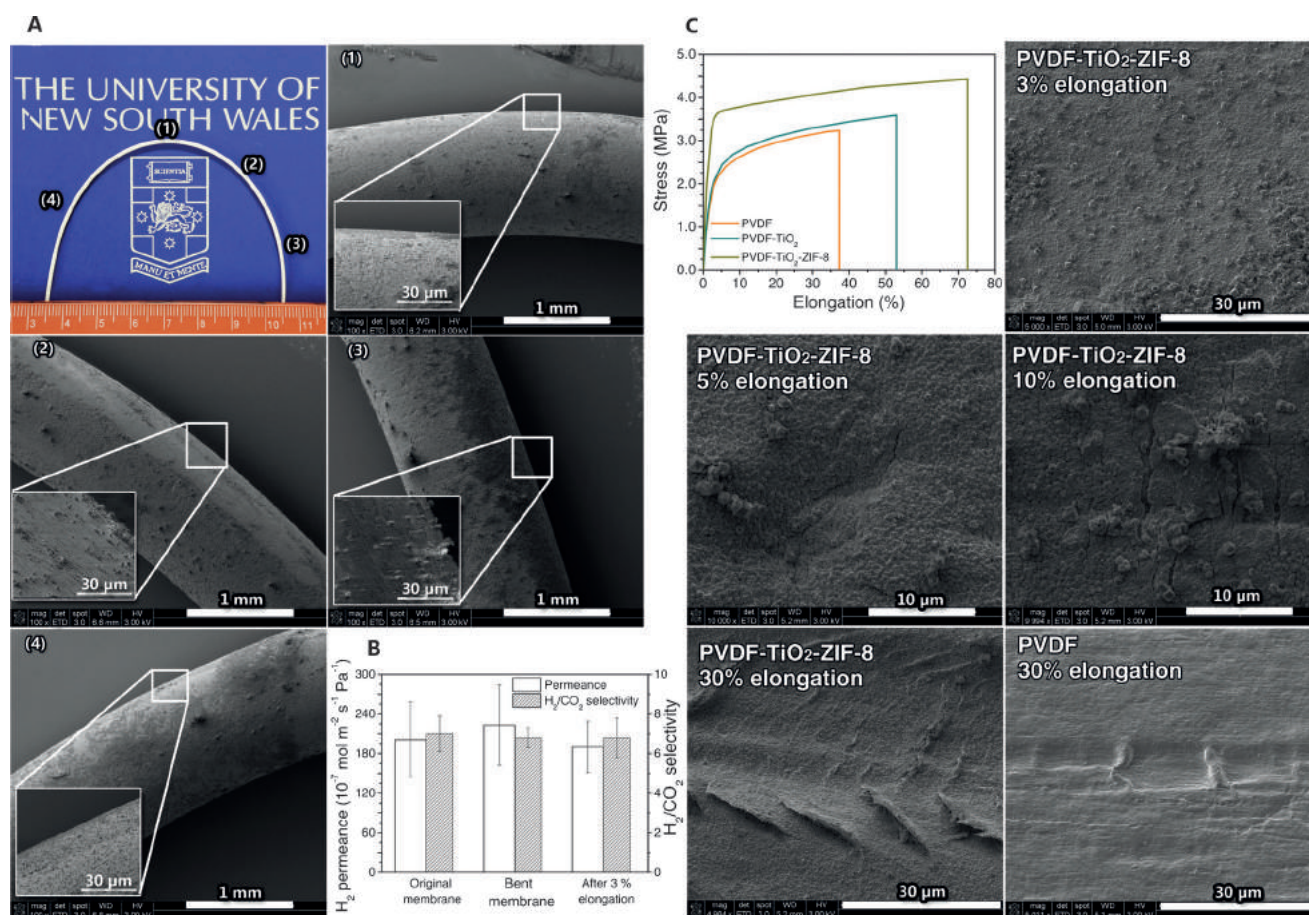


**Figure 2.** Gradual deposition of ZIF-8 onto the support surface. A) H<sub>2</sub> permeance and H<sub>2</sub>/CO<sub>2</sub> ideal selectivity of membranes with different ZIF-8 deposition times. B) Photograph of the ZIF-8 deposition process. C) Change in the ZIF-8 crystal size within the deposition solution. D) Turbidity of the suspension liquid during the deposition process. E) Schematic diagram of the ZIF-8 deposition process. F) Surface SEM images of the ZIF-8 deposition process with different reaction times. Fast amorphization occurred when the nanoscale ZIF-8 was exposed to the electron beam.

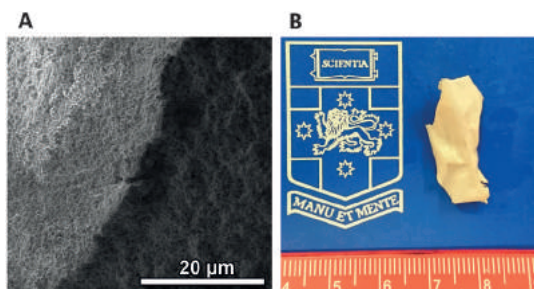
For practical applications, the membrane stability under mechanical stress, long-term operation, elevated temperature, and high pressure are crucial. Tan et al. have demonstrated that the Zn-mIm-Zn linkages (mIm = methylimidazole) in ZIF-8 are more compliant and the structure has greater flexibility than conventional open-framework materials.<sup>[20]</sup> During uniaxial straining, such as stretching and compression, the flexibility is mainly provided by the reversible change in the N-Zn-N and Zn-mIm-Zn bond angles. The ZIF-8 films supported on polymer membranes could be bent up to 180° without creating visible fragmentation and tearing of the ZIF-8 layer (Figure 3A). The gas permeation results also suggest no apparent change in the gas permeation performance after bending (Figure 3B). However, the ZIF-8 layer clearly improves the tensile strength of the whole composite membrane, and it shows elastic behavior at a low elongation rate (< 3%, Figure 3C). The gas permeation tests after 3% elongation further confirms the integrity of the ZIF-8 film. The SEM images (Figure 3C) indicate the formation and growth of ZIF-8 cracks under excessive additional stress. The plastic deformation of the PVDF polymer (elongation higher than 30%) resulted in surface wrinkling for both PVDF and PVDF-TiO<sub>2</sub>-ZIF-8 membranes. However, no detachment of the ZIF layers was observed, thus demonstrating the strong

bonding between the ZIF and the support as well as the flexibility of the ZIF-8 membrane. However, ZIF-8 has a relatively low shear modulus (0.967 GPa) compared with its Young's modulus and bulk modulus (2.98 and 7.751 GPa, respectively).<sup>[20]</sup> Therefore, the shear-induced plasticity and rupture need to be avoided to ensure the integrity of the ZIF-8 layer. This study is one of the first to elucidate the mechanical properties of a continuous MOF film.

The ZIF-8 membrane performed stably for up to 30 days, but thermal treatment led to a significant loss of the permeance. The XRD pattern and the SEM images reveal loss of the crystalline phase after thermal treatment (Figure S10). ZIF-8 has very good thermal stability up to 300°C.<sup>[21]</sup> Thus, amorphization could be caused by thermal-induced expansion of the PVDF, which imposes excessive stress on the ZIF-8 layer.<sup>[20,22]</sup> As a result, the polymer-supported ZIF-8 membrane is considered not suitable for operation at high temperatures. Furthermore, the ZIF-8 film did not collapse and fail under elevated pressures, even when the supporting membrane was severely deformed (Figure S11). To test the stability of the composite membrane against organic solvent, we prepared the flat-sheet composite membrane of ZIF-8 and soaked the membrane in dimethylacetamide (DMAc). This removed the PVDF polymer



**Figure 3.** Behavior of the ZIF-8 membrane under different mechanical stress. A) Optical and SEM images of the bent ZIF-8 membranes. B) Performance of the ZIF-8 membrane before and after the mechanical stability test. C) Tensile testing of the membranes and the SEM images of the membrane with different levels of elongation.



**Figure 4.** Flexible ZIF-8 layer on a TiO<sub>2</sub> exoskeleton. A) Cross-section SEM. B) Optical image of the surface.

support to leave a flexible, free-standing ZIF-8 layer on top of a highly porous TiO<sub>2</sub> exoskeleton, which originally encapsulated the polymer membrane structure (Figure 4 and Figure S12). This could lead to a novel route to develop an ultrathin free-standing MOF layer on a porous TiO<sub>2</sub> support. As a consequence of the mechanical rigidity of the TiO<sub>2</sub> exoskeleton under high temperature,<sup>[11]</sup> the resultant free-standing MOF layer should be able to maintain good thermal stability rather than losing its crystallinity (see Figure S10).

We also obtained a similar coherent ZIF-8 membrane on polyacrylonitrile (PAN) hollow fiber membranes (Table S4 and Figure S13). Compared with the PVDF support, the PAN-supported ZIF-8 membrane has lower permeance, which could be partially attributed to the formation of smaller pores in the support and thicker ZIF-8 layers. The PAN-based ZIF-8 composite membrane possessed comparable selectivity as the PVDF-supported membranes.

In conclusion, we reported here a facile fabrication approach to generate an ultrathin MOF membrane on a polymeric porous support. This study is one of the first to identify the mechanical flexibility of a coherent MOF film. In general, the developed MOF membrane has high permeance, good stability, and mechanical flexibility for many potential separation and sensing applications.

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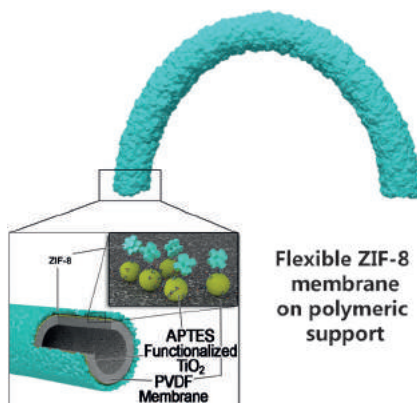
## Communications



## Molecular Sieving Membranes

J. Hou, P. D. Sutrisna, Y. Zhang,  
V. Chen\* ————— ■■■■-■■■■

Formation of Ultrathin, Continuous  
Metal–Organic Framework Membranes  
on Flexible Polymer Substrates



**Molecular sieving behavior**, with a high hydrogen permeance of and a  $H_2/CO_2$  selectivity of 7:1, is shown by ultrathin, continuous zeolitic imidazolate framework (ZIF-8) membranes prepared on polymeric supports. The ZIF-8 layer is flexible and adhered well to the support. This approach offers significant opportunities to exploit the unique properties of metal–organic frameworks in the fabrication of separation and sensing devices.

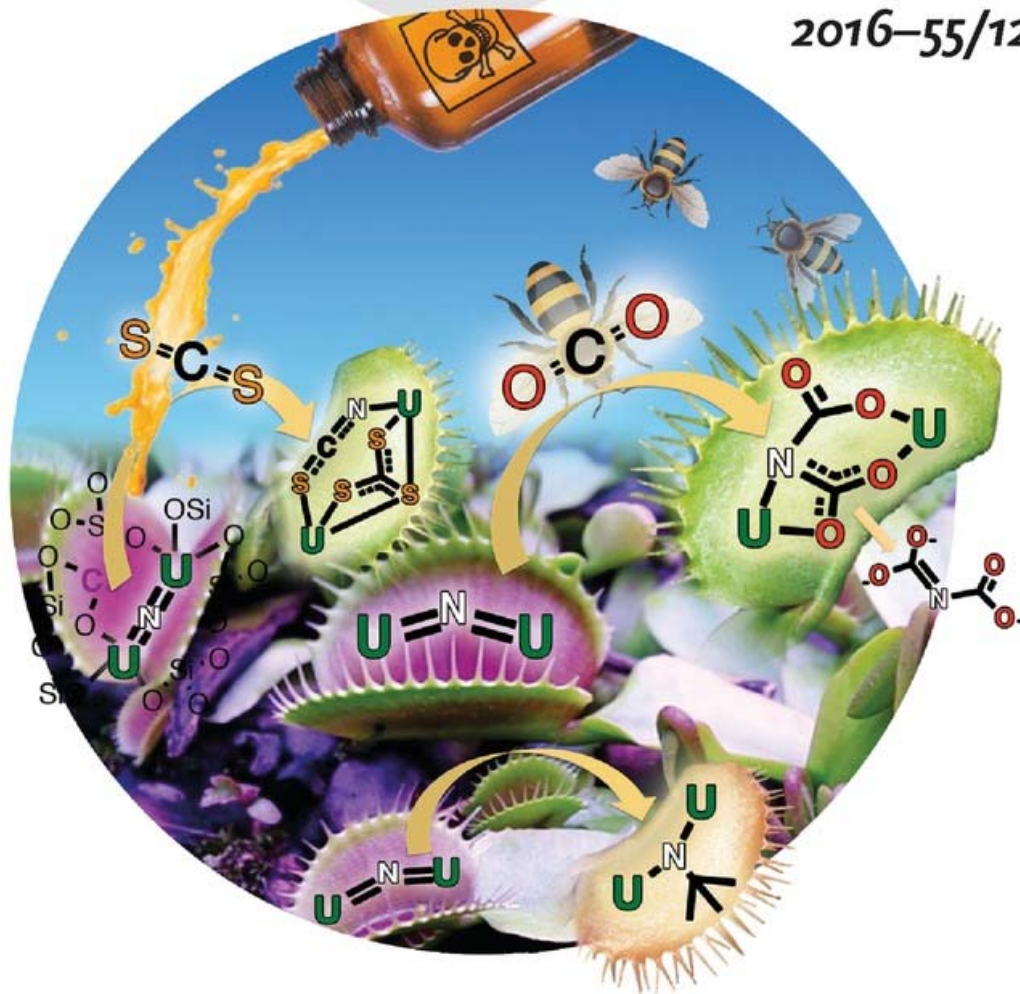


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## Cover Picture

M. Mazzanti et al.

Nucleophilic Reactivity of a Nitride-Bridged Diuranium(IV) Complex:  
CO<sub>2</sub> and CS<sub>2</sub> Functionalization

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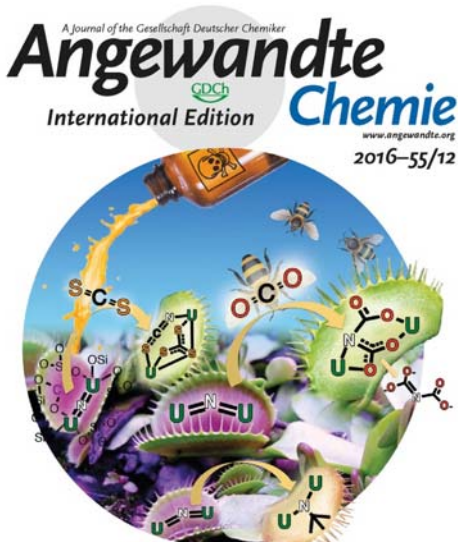
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### Cover Picture: Nucleophilic Reactivity of a Nitride-Bridged Diuranium(IV) Complex: CO<sub>2</sub> and CS<sub>2</sub> Functionalization (Angew. Chem. Int. Ed. 12/2016)

Marta Falcone, Lucile Chatelain, Dr. Marinella Mazzanti

Pages: 3831 | First Published: 29 February 2016



**The Venus flytrap** ... is used to illustrate the importance of a framework built from siloxide ligands and a Cs<sup>+</sup> ion in capturing abundant CO<sub>2</sub> and toxic CS<sub>2</sub> under ambient conditions and in promoting their reactions with a bridging nitride in a diuranium(IV) complex. In their Communication on [page 4074 ff.](#), M. Mazzanti et al. show how these processes lead to N–C bond formation and afford cyanate, thiocyanate, and unprecedented dicarbamate products.

## Cover Picture

M. Mazzanti et al.  
Nucleophilic Reactivity of a Nitride-Bridged Dioxanum(IV) Complex:  
CO<sub>2</sub> and C<sub>2</sub>F<sub>4</sub> Functionalization

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## Inside Cover: Triazine-Based Sequence-Defined Polymers with Side-Chain Diversity and Backbone–Backbone Interaction Motifs (Angew. Chem. Int. Ed. 12/2016)

Dr. Jay W. Grate, Dr. Kai-For Mo, Dr. Michael D. Daily

Pages: 3832 | First Published: 25 February 2016



**Polymer structures** with sequence control will enable new macromolecules and materials with useful functions. In their Communication on [page 3925 ff.](#), J. W. Grate et al. report how they have harnessed nucleophilic aromatic substitution reactions of cyanuric chloride to develop a new architecture for sequence-defined polymers with side-chain diversity and noncovalent backbone–backbone interactions.

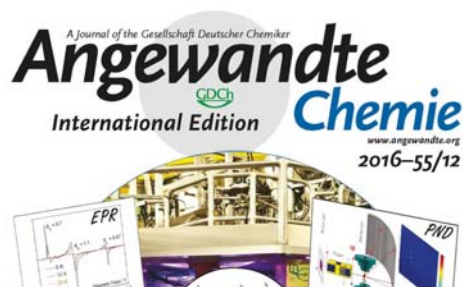
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## Inside Back Cover: Polarized Neutron Diffraction to Probe Local Magnetic Anisotropy of a Low-Spin Fe(III) Complex (Angew. Chem. Int. Ed. 12/2016)

Dr. Karl Ridier, Dr. Abhishake Mondal, Dr. Corentin Boilleau, Dr. Olivier Cador, Dr. Béatrice Gillon, Dr. Grégory Chaboussant, Dr. Boris Le Guennic, Dr. Karine Costuas, Prof. Rodrigue Lescouëzec

Pages: 4101 | First Published: 01 March 2016



**The local magnetic anisotropy** of a low-spin iron(III) complex has been studied. In their Communication on [page 3963 ff.](#), B. Gillon, B. Le Guennic, R. Lescouëzec et al. show how polarized neutron diffraction can provide a unique and straightforward picture of the local magnetic anisotropy and susceptibility tensors in molecules. A clear-cut method is offered to establish



#### The local magnetic anisotropy ...

... of a low-spin iron(III) complex has been studied. In their Communication on page 3952 ff., B. Gillon, B. Le Guennec, R. Lussolteux et al. show how polarized neutron diffraction can provide a unique and insightful picture of the local magnetic anisotropy and susceptibility tensors in molecules. A clear-cut method is offered to establish magneto-structural correlations in paramagnetic complexes.

WILEY-VCH

magneto-structural correlations in paramagnetic complexes.

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## Back Cover: Enantioselective Cyanosilylation of Ketones with Lithium(I) Dicyanotrimethylsilicate(IV) Catalyzed by a Chiral Lithium(I) Phosphoryl Phenoxide (Angew. Chem. Int. Ed. 12/2016)

Dr. Manabu Hatano, Katsuya Yamakawa, Tomoaki Kawai, Dr. Takahiro Horibe, Prof. Dr. Kazuaki Ishihara

Pages: 4102 | First Published: 24 February 2016

A Journal of the Gesellschaft Deutscher Chemiker  
**Angewandte Chemie**  
 International Edition  
 GDCh  
 www.angewandte.org  
 2016–55/12



#### The catalytic enantioselective cyanosilylation ...

... of ketones with highly reactive lithium dicyanotrimethylsilicate involves two lithium centers and is represented by Mr. Fikusuke holding two Engi-kumades (bamboo rakes). In their Communication on page 4021 ff., K. Ishihara and co-workers report on an acid–base cooperative catalytic system, an (*R*)-BINOL-derived lithium phosphoryl phenoxide aqua complex, which activates both the ketone and lithium dicyanotrimethylsilicate.

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**The catalytic enantioselective cyanosilylation** of ketones with highly reactive lithium dicyanotrimethylsilicate involves two lithium centers and is represented by Mr. Fikusuke holding two Engi-kumades (bamboo rakes). In their Communication on [page 4021 ff.](#), K. Ishihara and co-workers report on an acid–base cooperative catalytic system, an (*R*)-BINOL-derived lithium phosphoryl phenoxide aqua complex, which activates both the ketone and lithium dicyanotrimethylsilicate.

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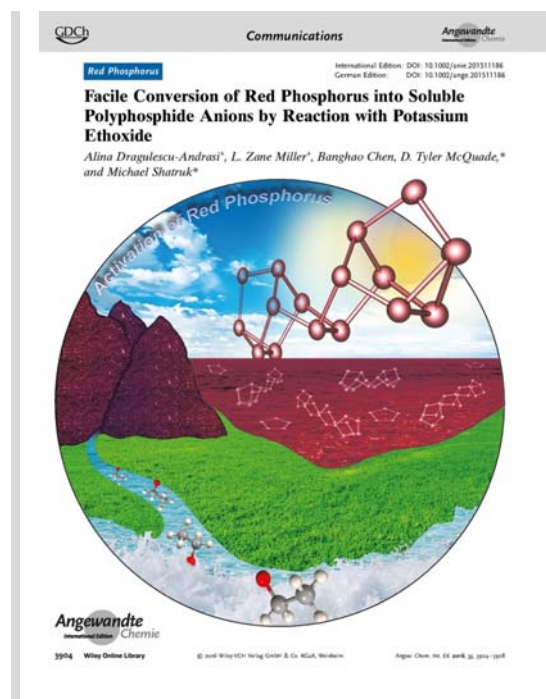
# Frontispiece

[Free Access](#)

## Frontispiece: Facile Conversion of Red Phosphorus into Soluble Polyphosphide Anions by Reaction with Potassium Ethoxide

Dr. Alina Dragulescu-Andrasi, Dr. L. Zane Miller, Dr. Banghao Chen, Prof. D. Tyler McQuade, Prof. Michael Shatruk

First Published: 11 March 2016



**Red Phosphorus** M. Shatruk, D. T. McQuade et al. describe the activation of red phosphorus by nucleophilic attack of KOEt to produce soluble polyphosphide species in their Communication on [page 3904 ff.](#) The reaction was scaled up by using a flow-chemistry approach. Picture designed by Kateryna Zavadska.

[Abstract](#) | [Full text](#) | [PDF](#) | [Request permissions](#)

# Editorials

[Free Access](#)

## Are the Current Changes Good or Bad for Chemistry?

Prof. Ian Manners

Pages: 3834-3835 | First Published: 24 February 2016



*“... The key discoveries on which many commercial products are ultimately based are often the result of fundamental research performed with no (commercial)*



*end-use even contemplated. It is remarkable that the idea of supporting truly fundamental research is under assault to varying degrees worldwide. Humankind will benefit from an environment where the emergence of completely new ideas is encouraged ...”* Read more in the Editorial by Ian Manners

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## Graphical Abstract

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**Graphical Abstract: Angew. Chem. Int. Ed. 12/2016**

Pages: 3837-3849 | First Published: 11 March 2016

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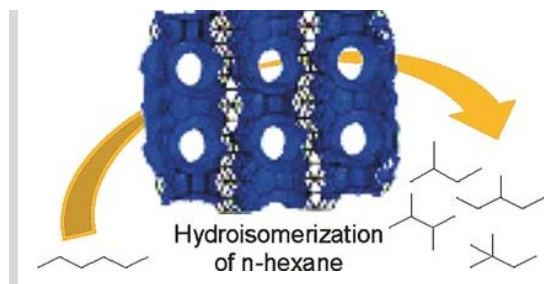
## News

**Spotlights on our sister journals: Angew. Chem. Int. Ed. 12/2016**

Pages: 3850-3853 | First Published: 11 March 2016

Easily produced aluminosilicate **IWV** made in hydroxide media demonstrates catalytic activity





[Abstract](#) | [PDF](#) | [Request permissions](#)

## Author Profiles

[Free Access](#)

**Oliver Trapp**

Pages: 3854 | First Published: 04 February 2016



*“My not-so-secret passion is stereochemistry. If I were not a scientist, I would be an astronaut ...”* This and more about Oliver Trapp can be found on page 3854.

[Abstract](#) | [Full text](#) | [PDF](#) | [References](#) | [Request permissions](#)

# News

**ADUC Prizes: A. Andrieu-Brunsen, I. Siewert, and T. Magauer / Carl Duisberg Memorial Prize: F. R. Fischer / Honorary Membership of the Gesellschaft Deutscher Chemiker: D. Jahn / Windaus Medal and Herbert C. Brown Award: A. Fürstner / Gottfried Wilhelm Leibniz Prize: B. List / Rottendorf Prize: M. Pietsch / Seymour Schulich Lectureship Award: M. T. Reetz / Akademiepreis für Chemie: M. Schnell**

Pages: 3855-3856 | First Published: 24 February 2016



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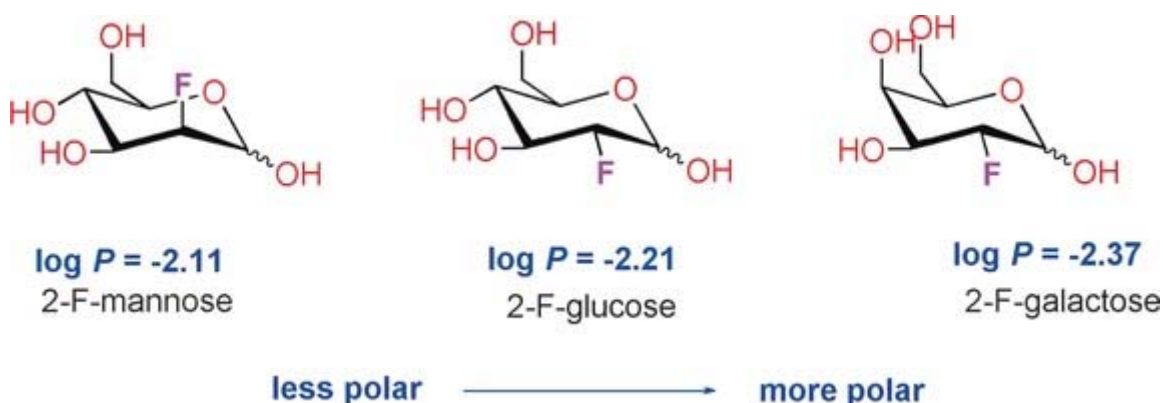
# Highlights

## Fluorocarbons

**Accurate Lipophilicity ( $\log P$ ) Measurements Inform on Subtle Stereoelectronic Effects in Fluorine Chemistry**

Prof. Dr. David O'Hagan, Dr. Robert J. Young

Pages: 3858-3860 | First Published: 19 February 2016





**Polar exploration:** Recently, Linclau and co-workers disclosed a straightforward  $^{19}\text{F}$ -NMR method for determining the log  $P$  values of fluorocarbons. The method is particularly useful for most polar compounds and provides a quantitative way to rationalize the more subtle stereoelectronic consequences of fluorine introduction.

[Abstract](#) | [Full text](#) | [PDF](#) | [References](#) | [Request permissions](#)

## Reviews

### 3D Printing

#### 3D-Printed Microfluidics

Dr. Anthony K. Au, Wilson Huynh, Dr. Lisa F. Horowitz, Dr. Albert Folch

Pages: 3862-3881 | First Published: 08 February 2016



**3D printing techniques** are ideally suited to the fabrication of microfluidic devices. As structures are created by adding materials without the need for etching or dissolution, processing is environmentally friendly and economically efficient. It is expected that in the next few years, 3D printing will replace molding techniques for the fabrication of microfluidic devices.

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### Drug Discovery

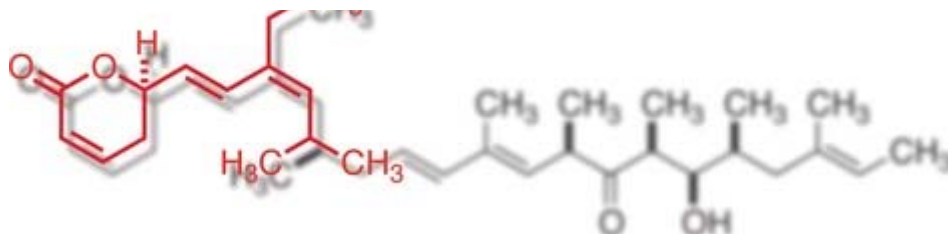
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#### Capturing Biological Activity in Natural Product Fragments by Chemical Synthesis

Dr. Erika A. Crane, Prof. Dr. Karl Gademann

Pages: 3882-3902 | First Published: 02 February 2016





**Less is more:** The desired biological activity of structurally complex natural products can be captured in small, truncated fragments. This process is enabled by chemical synthesis, which guides the discovery process and allows the secrets enshrined in natural products to be unlocked.

[Abstract](#) | [Full text](#) | [PDF](#) | [References](#) | [Request permissions](#)

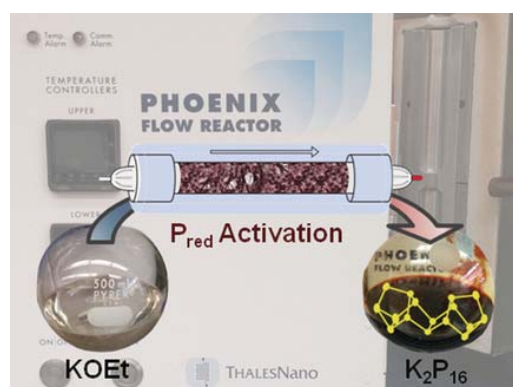
## Communications

### Red Phosphorus

#### Facile Conversion of Red Phosphorus into Soluble Polyphosphide Anions by Reaction with Potassium Ethoxide

Dr. Alina Dragulescu-Andrasi, Dr. L. Zane Miller, Dr. Banghao Chen, Prof. D. Tyler McQuade, Prof. Michael Shatruk

Pages: 3904-3908 | First Published: 01 March 2016



**Let's get active!** The reaction between red phosphorus ( $P_{red}$ ) and potassium ethoxide (KOEt) in various organic solvents yields soluble polyphosphide anions. This simple method for the activation of  $P_{red}$  avoids the use of strong reducing agents such as alkali metals. The reaction was adapted to a packed-bed flow reactor to afford continuous, multigram synthesis of soluble polyphosphides.

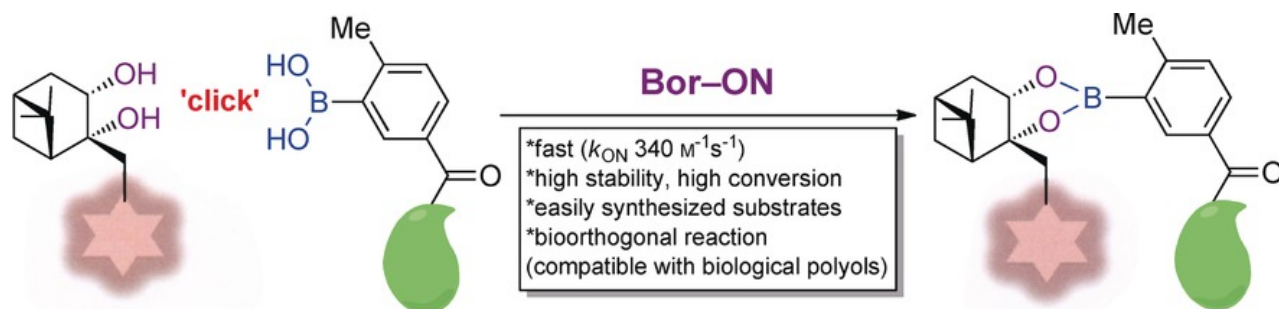
[Abstract](#) | [Full text](#) | [PDF](#) | [References](#) | [Request permissions](#)

### Bioconjugation

#### Fast and Tight Boronate Formation for Click Bioorthogonal Conjugation

Burcin Akgun, Prof. Dr. Dennis G. Hall

Pages: 3909-3913 | First Published: 23 February 2016



**A click bioorthogonal** reaction system was devised for the fast ligation of conjugatable derivatives of a rigid cyclic diol (nopoldiol) with an optimal boronic acid partner, 2-methyl-5-carboxymethylphenylboronic acid. The resulting boronates form reversibly within minutes at low concentrations in water. Efficient protein conjugation under physiological conditions was demonstrated with model proteins thioredoxin and albumin.

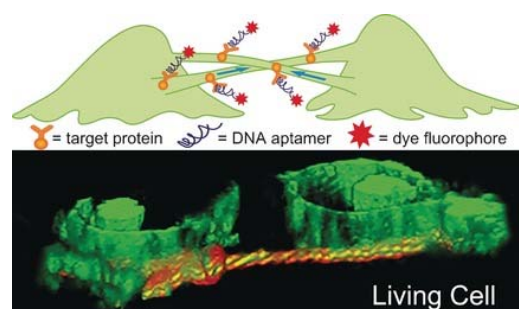
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## Aptamer Probes

### Intercellular Connections Related to Cell–Cell Crosstalk Specifically Recognized by an Aptamer

Nan Zhang, Prof. Tao Bing, Luyao Shen, Rusheng Song, Linlin Wang, Prof. Xiangjun Liu, Meirong Liu, Dr. Juan Li, Prof. Weihong Tan, Prof. Dihua Shanguan

Pages: 3914-3918 | First Published: 17 February 2016



**Unusual connections:** Aptamer M17A2 generated by cell-SELEX specifically recognizes intercellular connections related to cell-cell crosstalk. These connections are homologous with tunneling nanotubes. Proteins were found to be transported through M17A2-binding connections.

[Abstract](#) | [Full text](#) | [PDF](#) | [References](#) | [Request permissions](#)

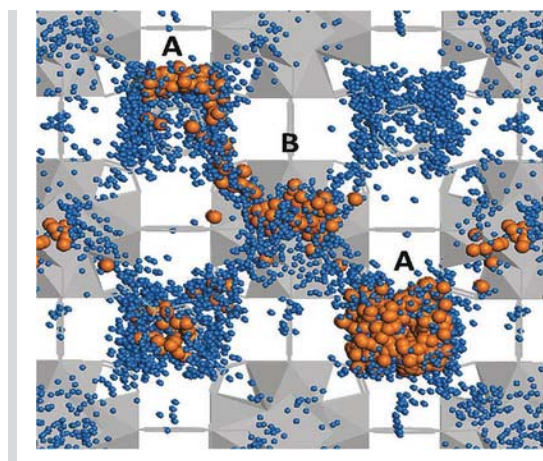
## Metal–Organic Frameworks

### Proton Transport in a Highly Conductive Porous Zirconium-Based Metal–Organic Framework: Molecular Insight

Dr. Daiane Damasceno Borges, Dr. Sabine Devautour-Vinot, Dr. Hervé Jobic, Dr. Jacques Ollivier, Dr.

Farid Nouar, Dr. Rocio Semino, Dr. Thomas Devic, Dr. Christian Serre, Prof. Francesco Paesani, Prof. Guillaume Maurin

Pages: 3919-3924 | First Published: 17 February 2016



**Proton conductivity:** The superprotonic behavior of the water-stable UiO-66(Zr)-(CO<sub>2</sub>H)<sub>2</sub> metal–organic framework (MOF) was examined at the molecular level by combining quasi-elastic neutron scattering measurements and aMS-EVB3 molecular dynamics simulations. The proton transport is shown to be assisted by the formation of a hydrogen-bonded network of water molecules that spans from the tetrahedral to the octahedral cages of this MOF.

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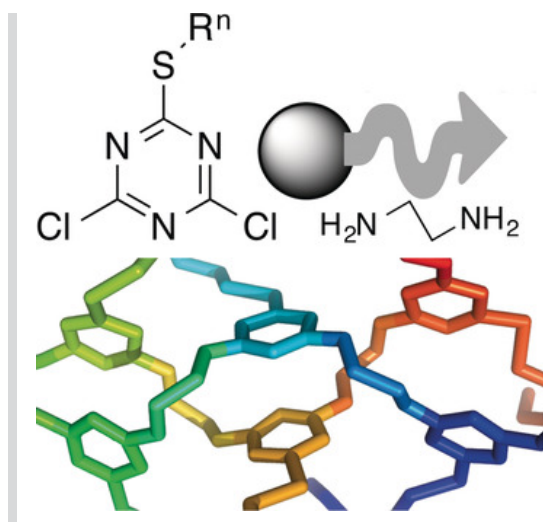
## Sequence-Defined Polymers | Hot Paper

[Open Access](#)

### Triazine-Based Sequence-Defined Polymers with Side-Chain Diversity and Backbone–Backbone Interaction Motifs

Dr. Jay W. Grate, Dr. Kai-For Mo, Dr. Michael D. Daily

Pages: 3925-3930 | First Published: 10 February 2016



**The sequence of monomers** in macromolecules leads to diverse structures and functions. Nucleophilic aromatic substitution reactions of cyanuric chloride, using a submonomer solid-phase synthesis, have been harnessed to develop a new architecture for sequence-defined polymers with side-chain diversity and noncovalent backbone–backbone interactions.

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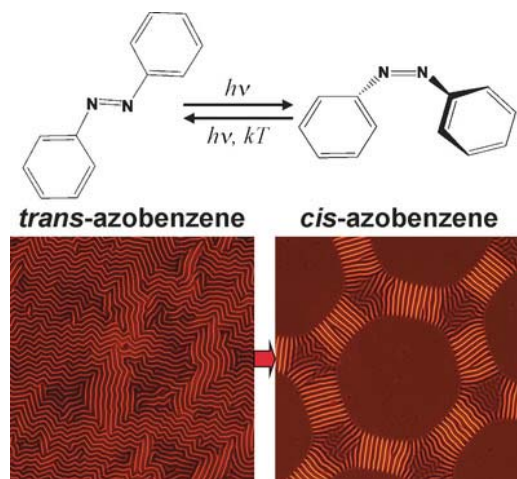
## Azopolymers | Very Important Paper



## Tuning and Erasing Surface Wrinkles by Reversible Visible-Light-Induced Photoisomerization

Chuanyong Zong, Yan Zhao, Haipeng Ji, Xue Han, Jixun Xie, Juanjuan Wang, Prof. Yanping Cao, Prof. Shichun Jiang, Prof. Conghua Lu

Pages: 3931-3935 | First Published: 19 February 2016



**Surface wrinkles** on azopolymer films can be optically erased by visible-light irradiation. The rapid reversible photoisomerization of the azobenzene units generates a significant local nanoscale force throughout the film, which leads to stress release and erasure of the wrinkles. Highly ordered wrinkling patterns with well-defined microstructures were fabricated by selective light exposure.

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## Graphene Materials

### A Phytic Acid Induced Super-Amphiphilic Multifunctional 3D Graphene-Based Foam

Xinhong Song, Yiying Chen, Mingcong Rong, Prof. Dr. Zhaoxiong Xie, Tingting Zhao, Prof. Yiru Wang, Prof. Dr. Xi Chen, Prof. Dr. Otto S. Wolfbeis

Pages: 3936-3941 | First Published: 18 February 2016



**A super-amphiphilic 3D graphene-based foam** was prepared through a single-step approach using phytic acid, which acts as both a gelator and a dopant. The product shows  $0^\circ$  contact angle for both oil and water. The graphene foam was impregnated with a catalyst for epoxidation of alkenes and then showed very high catalytic efficiency even in the absence of a co-solvent and without need for stirring.

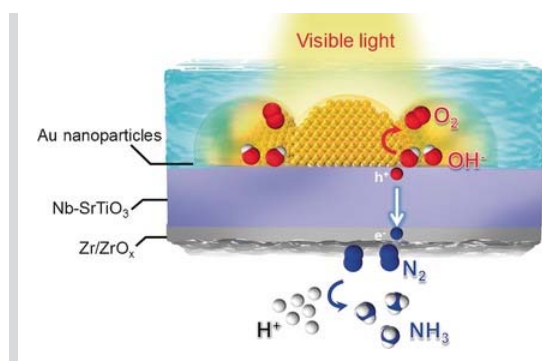
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# Nitrogen Photofixation

## Selective Dinitrogen Conversion to Ammonia Using Water and Visible Light through Plasmon-induced Charge Separation

Dr. Tomoya Oshikiri, Prof. Kosei Ueno, Prof. Hiroaki Misawa

Pages: 3942-3946 | First Published: 17 February 2016



**NH<sub>3</sub> sees the light of day:** A highly selective and visible-light-responsive plasmonic NH<sub>3</sub> synthesis device containing a Au nanostructure and a Zr/ZrO<sub>x</sub> thin film was developed. N<sub>2</sub> gas was reduced to NH<sub>3</sub> by using water as an electron donor through plasmon-induced charge separation under visible light irradiation.

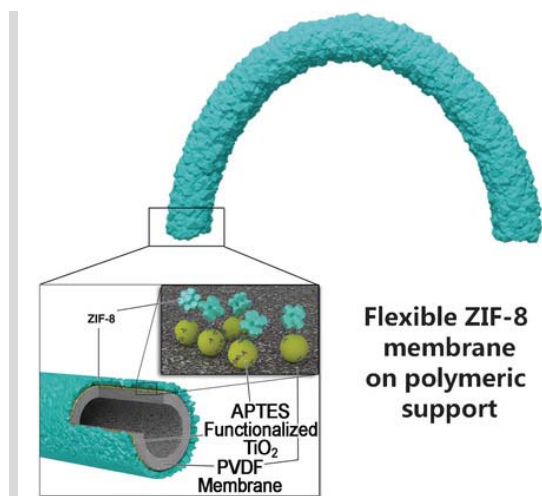
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# Molecular Sieving Membranes

## Formation of Ultrathin, Continuous Metal–Organic Framework Membranes on Flexible Polymer Substrates

Dr. Jingwei Hou, Putu D. Sutrisna, Prof. Yatao Zhang, Prof. Vicki Chen

Pages: 3947-3951 | First Published: 23 February 2016



**Molecular sieving behavior**, with a high hydrogen permeance and a H<sub>2</sub>/CO<sub>2</sub> selectivity of 7:1, is shown by ultrathin, continuous zeolitic imidazolate framework (ZIF-8) membranes prepared on polymeric supports. The ZIF-8 layer is flexible and adhered well to the support. This approach offers significant opportunities to exploit the unique properties of metal–organic frameworks in the fabrication of separation and sensing devices.

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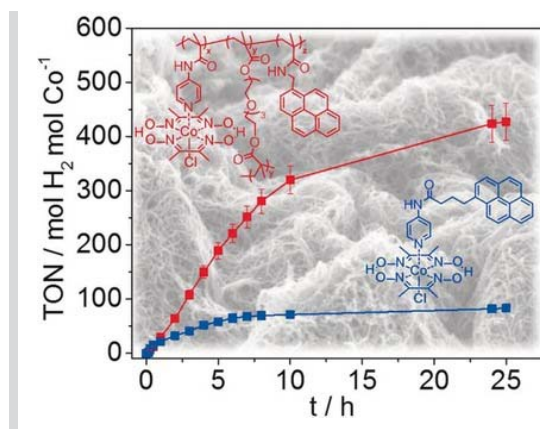
## H<sub>2</sub> Evolution | Hot Paper

[Open Access](#)

### A Poly(cobaloxime)/Carbon Nanotube Electrode: Freestanding Buckypaper with Polymer-Enhanced H<sub>2</sub>-Evolution Performance

Dr. Bertrand Reuillard, Dr. Julien Warnan, Jane J. Leung, David W. Wakerley, Dr. Erwin Reisner

Pages: 3952-3957 | First Published: 18 February 2016



**Giving it some gas:** Hybrid carbon-nanotube-based electrodes can be constructed in a straightforward manner by the integration of a cobaloxime-containing copolymeric structure into a multiwall carbon nanotube matrix. The use of the cobaloxime-containing copolymer led to a significant improvement in the electrocatalytic hydrogen-evolution performance compared to the use of a monomeric cobaloxime (see figure).

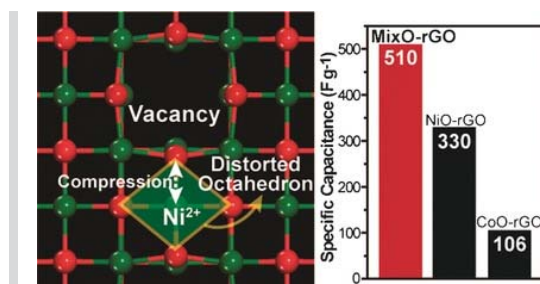
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## Energy Storage

### Enhanced Pseudocapacitance in Multicomponent Transition-Metal Oxides by Local Distortion of Oxygen Octahedra

Hyeon Jeong Lee, Ji Hoon Lee, Prof. Sung-Yoon Chung, Prof. Jang Wook Choi

Pages: 3958-3962 | First Published: 17 February 2016



**In full swing:** A synergistic effect of transition-metal mixing led to anomalously high pseudocapacitance of a multicomponent transition-metal oxide, MixO-rGO, containing nickel, cobalt, and manganese cations. The high pseudocapacitance results from local distortions of the oxygen framework, which facilitate the redox swing of the key transition metal (see picture).

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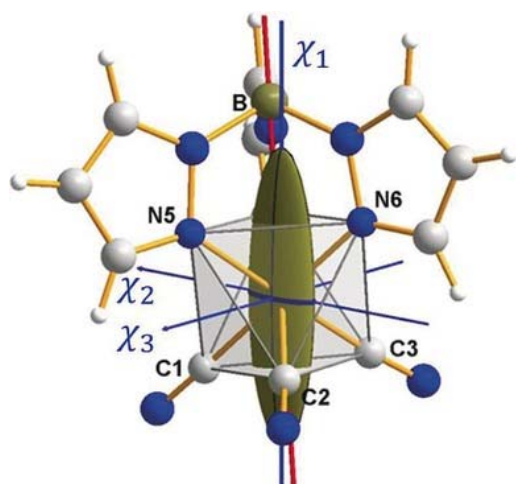
## Magnetic Materials

### Polarized Neutron Diffraction to Probe Local Magnetic Anisotropy of a Low-Spin

## Fe(III) Complex

Dr. Karl Ridier, Dr. Abhishake Mondal, Dr. Corentin Boilleau, Dr. Olivier Cador, Dr. Béatrice Gillon, Dr. Grégory Chaboussant, Dr. Boris Le Guennic, Dr. Karine Costuas, Prof. Rodrigue Lescouëzec

Pages: 3963-3967 | First Published: 17 February 2016



**Molecular Magnetism:** The low-temperature molecular magnetic susceptibility tensor of the anisotropic low-spin  $[\text{Fe}^{\text{III}}(\text{Tp})(\text{CN})_3]^-$  complex (Tp=tris(pyrazolyl)borate; see picture) was determined by polarized neutron diffraction (PND). PND provided a unique, direct, and straightforward picture of the magnetic anisotropy and susceptibility tensors.

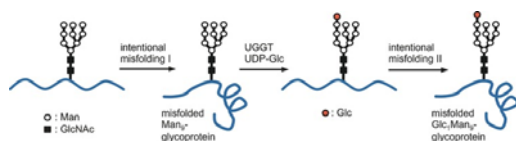
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## Protein Folding | Very Important Paper

### Synthesis of $\text{Glc}_1\text{Man}_9$ -Glycoprotein Probes by a Misfolding/Enzymatic Glucosylation/Misfolding Sequence

Dr. Masayuki Izumi, Yukiho Oka, Dr. Ryo Okamoto, Dr. Akira Seko, Dr. Yoichi Takeda, Prof. Dr. Yukishige Ito, Prof. Dr. Yasuhiro Kajihara

Pages: 3968-3971 | First Published: 17 February 2016



**Go native:** Native and misfolded  $\text{Glc}_1\text{Man}_9$ -glycoprotein probes were synthesized by using the title sequence. These probes were used to analyze the substrate preference of glucosidase II and the binding

of misfolded glycoproteins to the lectin chaperone calreticulin, both of which are involved in the glycoprotein quality control system. Calreticulin was found to bind preferentially to a hydrophobic non-native glycoprotein whereas glucosidase II activity was not affected by glycoprotein conformation.

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## CO<sub>2</sub> Fixation

### Substrate-Controlled Product Divergence: Conversion of CO<sub>2</sub> into Heterocyclic



## Products

Jeroen Rintjema, Roel Epping, Dr. Giulia Fiorani, Dr. Eddy Martín, Eduardo C. Escudero-Adán, Prof. Dr. Arjan W. Kleij

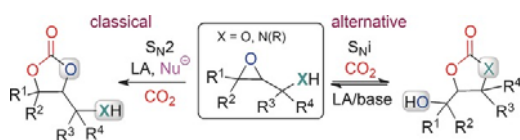
Pages: 3972-3976 | First Published: 19 February 2016

**!** [Correction\(s\) for this article >](#)

### Corrigendum: Substrate-Controlled Product Divergence: Conversion of CO<sub>2</sub> into Heterocyclic Products

Jeroen Rintjema, Roel Epping, Giulia Fiorani, Eddy Martín, Eduardo C. Escudero-Adán, Arjan W. Kleij

Volume 55, Issue 40, Angewandte Chemie International Edition | pages: 12136-12136 | First Published online: May 9, 2016



**Two ways:** A novel substrate-driven and divergent approach leads to a variety of heterocyclic products from a single epoxy alcohol/amine substrate. The different reactions paths are controlled by the

reaction conditions. A new mechanism is proposed wherein the substrate activates the CO<sub>2</sub> molecule, thus leading to an intramolecular process and giving rise to formation of an alternative product. LA=Lewis acid.

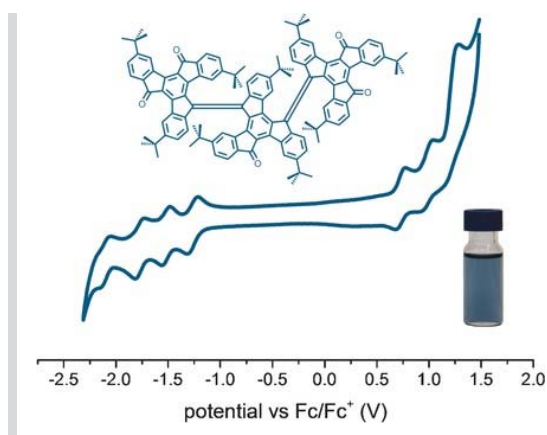
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## Extended $\pi$ Systems

### Rigid Conjugated Twisted Truxene Dimers and Trimers as Electron Acceptors

Dr. Gang Zhang, Vincent Lami, Dr. Frank Rominger, Prof.Dr. Yana Vaynzof, Prof.Dr. Michael Mastalerz

Pages: 3977-3981 | First Published: 18 February 2016



**Twisted:** Rigid truxenone oligomers with an extended  $\pi$  backbone (see example structure) were synthesized by oxidative dimerization reactions. The resulting extended conjugated systems, which dissolve well in common organic solvents, have large extinction coefficients and low-lying LUMO levels. Their suitability as electron acceptors for organic electronics has been demonstrated in bulk-heterojunction organic solar cells.

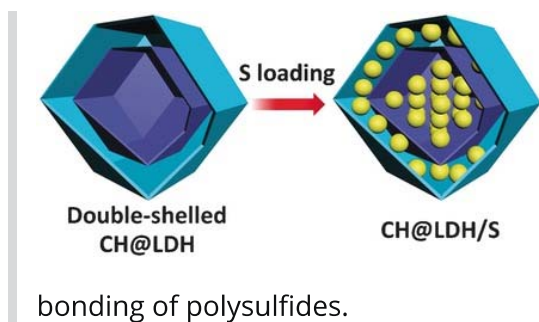
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## Lithium–Sulfur Batteries | Hot Paper

### Double-Shelled Nanocages with Cobalt Hydroxide Inner Shell and Layered Double Hydroxides Outer Shell as High-Efficiency Polysulfide Mediator for Lithium–Sulfur Batteries

Jintao Zhang, Dr. Han Hu, Dr. Zhen Li, Prof. Xiong Wen (David) Lou

Pages: 3982-3986 | First Published: 19 February 2016



**Nanocages with two shells** of cobalt hydroxide and layered double hydroxides (CH@LDH) have been synthesized as a sulfur host for lithium–sulfur batteries. The hollow CH@LDH polyhedra with their complex shell structures were not only used for encapsulating a high content of sulfur, but they also provided functionalized surfaces for chemically

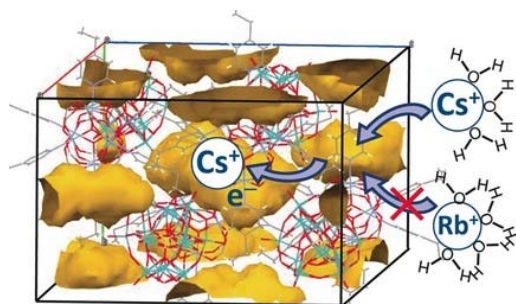
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## Ion-Selective Uptake

### Reduction-Induced Highly Selective Uptake of Cesium Ions by an Ionic Crystal Based on Silicododecamolybdate

Saori Seino, Ryosuke Kawahara, Dr. Yoshiyuki Ogasawara, Prof. Noritaka Mizuno, Dr. Sayaka Uchida

Pages: 3987-3991 | First Published: 19 February 2016



**Highly selective uptake of Cs<sup>+</sup>** by an ionic crystal based on silicododecamolybdate is reported. Up to 3.8 mol(Cs<sup>+</sup>) per mole of solid by cation-exchange and reduction of silicododecamolybdate. Other alkali-metal and alkaline-earth-metal cations were almost completely excluded (<0.2 mol mol(solid)<sup>-1</sup>).

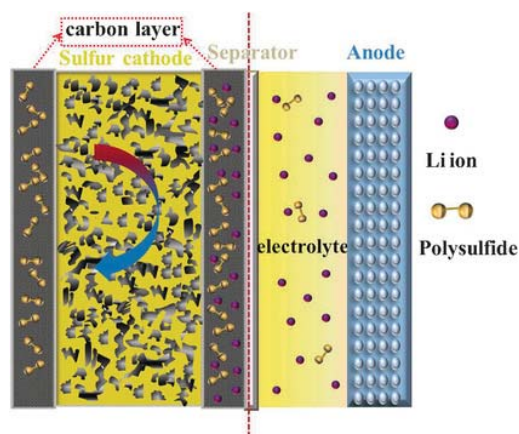
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## Electrochemistry

### A Strategy for Configuration of an Integrated Flexible Sulfur Cathode for High-Performance Lithium–Sulfur Batteries

Hongqiang Wang, Wenchao Zhang, Prof. Huakun Liu, Prof. Zaiping Guo

Pages: 3992-3996 | First Published: 17 February 2016



**An integrated flexible sulfur cathode** consisting of a carbon/sulfur/carbon sandwich structure coated on a polypropylene separator was prepared by the doctor-blade method. This sulfur cathode could enhance the electronic conductivity, toleration of volume expansion, and control of the polysulfide diffusion, thereby improving the electrochemical performance of lithium–sulfur batteries.

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## Heterocycles

## Water-Assisted Nitrile Oxide Cycloadditions: Synthesis of Isoxazoles and Stereoselective Syntheses of Isoxazolines and 1,2,4-Oxadiazoles

Chatchai Kesornpun, Dr. Thammarat Aree, Prof. Dr. Chulabhorn Mahidol, Prof. Dr. Somsak Ruchirawat, Dr. Prasat Kittakoop

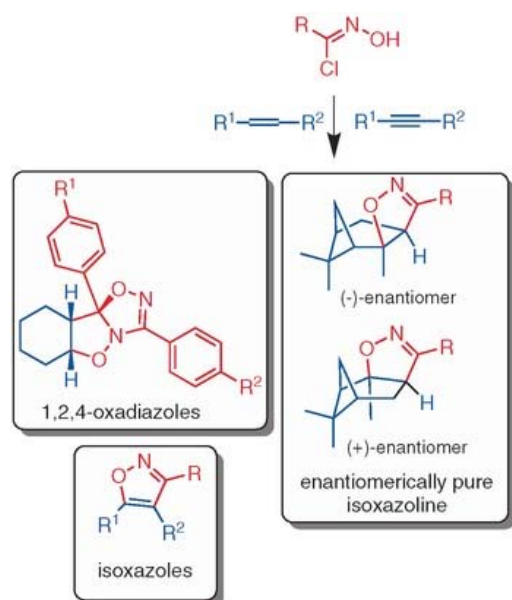
Pages: 3997-4001 | First Published: 23 February 2016

[! Correction\(s\) for this article >](#)

### Corrigendum: Water-Assisted Nitrile Oxide Cycloadditions: Synthesis of Isoxazoles and Stereoselective Syntheses of Isoxazolines and 1,2,4-Oxadiazoles

Chatchai Kesornpun, Thammarat Aree, Chulabhorn Mahidol, Somsak Ruchirawat, Prasat Kittakoop

Volume 55, Issue 36, Angewandte Chemie International Edition | pages: 10548-10548 | First Published online: August 23, 2016



**Water changes things:** In water, nitrile oxides can be generated from oxime halides without using catalysts. Unexpectedly, the nitrile oxide generation proceeds under mild acidic conditions. The catalyst-free cycloaddition of the formed nitrile oxides with certain alkenes in water had excellent stereoselectivity, and it was applied to the synthesis of isoxazoles, novel oxadiazoles, and enantiomerically pure isoxazolines.

[Abstract](#) | [Full text](#) | [PDF](#) | [References](#) | [Request permissions](#)

## Protein Modifications | Very Important Paper

### Glycation Reactivity of a Quorum-Sensing Signaling Molecule

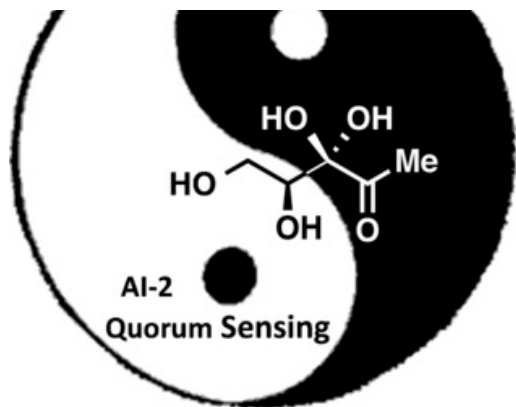
Dr. Kyoji Tsuchikama, Dr. Major Gooyit, Dr. Tyler L. Harris, Dr. Jie Zhu, Dr. Daniel Globisch, Dr. Gunnar F. Kaufmann, Prof. Dr. Kim D. Janda

Pages: 4002-4006 | First Published: 17 February 2016



**Yin and yang:** The quorum-sensing signaling molecule (4S)-4,5-dihydroxy-2,3-pentanedione





assumes a yet unrecognized biochemical role and is capable of glycosylating DNA and proteins in a rapid and stereospecific manner. These results open new avenues toward tracking and understanding the fate and function of the elusive quorum-sensing signaling molecule.

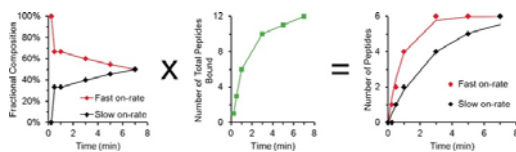
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## Affinity Measurements | Hot Paper

### High-Throughput Measurement of Binding Kinetics by mRNA Display and Next-Generation Sequencing

Dr. Farzad Jalali-Yazdi, Lan Huong Lai, Prof. Terry T. Takahashi, Prof. Richard W. Roberts

Pages: 4007-4010 | First Published: 23 February 2016



**The kinetic** on- and off-rates of over 20 000 individual ligands for their target protein were determined without the need to synthesize each individual ligand separately. Reproducible and accurate results were obtained by combining mRNA display and high-

throughput DNA sequencing.

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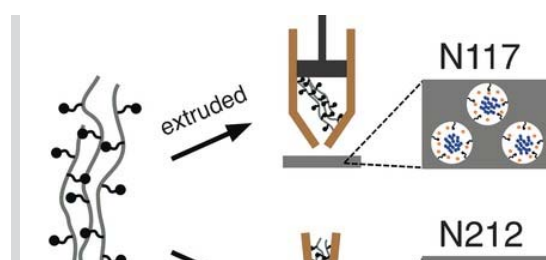
## Water and Proton Transport

[Open Access](#)

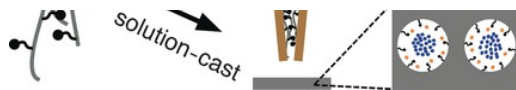
### Nanoscale Distribution of Sulfonic Acid Groups Determines Structure and Binding of Water in Nafion Membranes

Xiao Ling, Prof. Dr. Mischa Bonn, Dr. Sapun H. Parekh, Dr. Katrin F. Domke

Pages: 4011-4015 | First Published: 19 February 2016



**Casting is important:** Differently cast Nafion membranes of identical chemical composition exhibit distinct nanoscale chemical constitutions of the water channels. The observed disproportional increase of undercoordinated water molecules in membranes with smaller channel diameters correlates with



improved macroscopic water and proton transport properties under the same conditions.

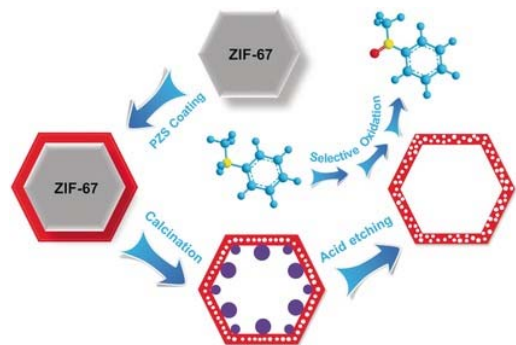
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## Heterogeneous Catalysis | Hot Paper

### Nitrogen, Phosphorus, and Sulfur Co-Doped Hollow Carbon Shell as Superior Metal-Free Catalyst for Selective Oxidation of Aromatic Alkanes

Shuliang Yang, Dr. Li Peng, Peipei Huang, Xiaoshi Wang, Yongbin Sun, Dr. Changyan Cao, Prof. Weiguo Song

Pages: 4016-4020 | First Published: 17 February 2016



**Highly efficient and selective:** A synthetic route was developed to produce nitrogen, phosphorus, and sulfur co-doped hollow carbon shells which were used as a metal-free carbocatalyst. The carbocatalyst had a high surface area and showed superior activity in the selective oxidation of aromatic alkanes in aqueous solution.

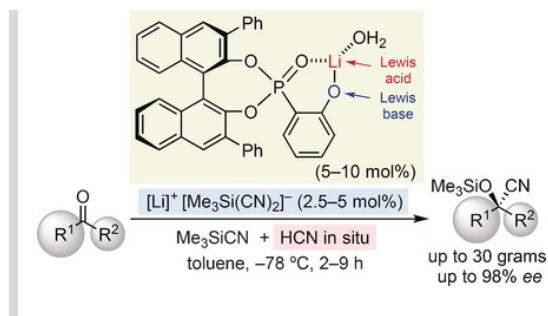
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## Homogeneous Catalysis

### Enantioselective Cyanosilylation of Ketones with Lithium(I) Dicyanotrimethylsilicate(IV) Catalyzed by a Chiral Lithium(I) Phosphoryl Phenoxide

Dr. Manabu Hatano, Katsuya Yamakawa, Tomoaki Kawai, Dr. Takahiro Horibe, Prof. Dr. Kazuaki Ishihara

Pages: 4021-4025 | First Published: 02 February 2016



**Joint effort:** A highly enantioselective cyanosilylation of ketones was developed by using a chiral lithium phosphoryl phenoxide aqua complex as an acid/base cooperative catalyst. The pentacoordinate silicate generated in situ from  $\text{Me}_3\text{SiCN}/\text{LiCN}$  acts as a reactive cyano reagent. Moreover, a 30 gram scale reaction and the synthesis of the key precursor to (+)-13-hydroxyisocyclocelabenzine are described.

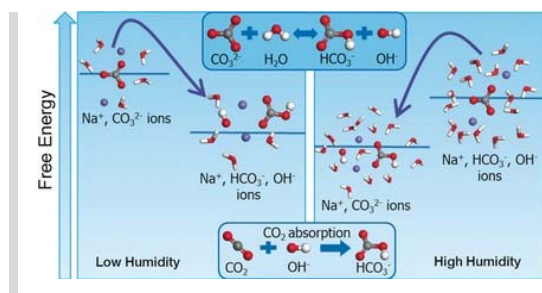
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## CO<sub>2</sub> Capture

### Capture CO<sub>2</sub> from Ambient Air Using Nanoconfined Ion Hydration

Xiaoyang Shi, Hang Xiao, Prof. Klaus S. Lackner, Prof. Xi Chen

Pages: 4026-4029 | First Published: 23 February 2016



**The presence of water** confined in nanoscopic pores was shown to control the equilibrium between CO<sub>2</sub> and bicarbonate on adsorbent surfaces. This control allowed for facile sequestration of CO<sub>2</sub> from the air, and may influence the design of other adsorbent materials.

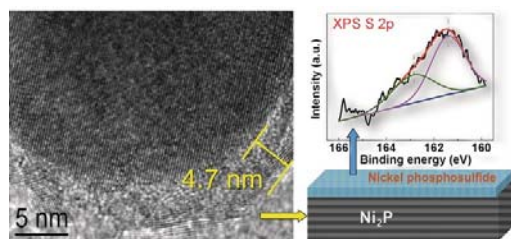
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## Hydrocarbon Processing

### Facile Preparation of Ni<sub>2</sub>P with a Sulfur-Containing Surface Layer by Low-Temperature Reduction of Ni<sub>2</sub>P<sub>2</sub>S<sub>6</sub>

Song Tian, Dr. Xiang Li, Prof. Dr. Anjie Wang, Prof. Dr. Roel Prins, Yongying Chen, Prof. Yongkang Hu

Pages: 4030-4034 | First Published: 17 February 2016



**Sulfur icing on the cake:**  $\text{Ni}_2\text{P}$ , which is highly active in hydrodesulfurization and selective hydrogenation reactions, was obtained by reduction of  $\text{Ni}_2\text{P}_2\text{S}_6$  at temperatures as low as 200–220 °C. A distinct sulfur-containing layer (see picture: blue) was observed at the surface of the  $\text{Ni}_2\text{P}$  catalyst, which is a direct observation of the so-called nickel phosphosulfide

phase.

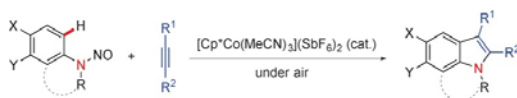
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## C–H Activation

### Cationic Cobalt(III) Catalyzed Indole Synthesis: The Regioselective Intermolecular Cyclization of N-Nitrosoanilines and Alkynes

Yujie Liang, Dr. Ning Jiao

Pages: 4035-4039 | First Published: 17 February 2016



**Cp\* and Co.:** The unique regioselectivity and reactivity of cobalt(III) in the direct cyclization of N-nitrosoanilines with alkynes for the expedient

synthesis of a variety of N-substituted indoles is demonstrated. In the presence of a cobalt(III) catalyst, high regioselectivity was observed when using unsymmetrical *meta*-substituted N-nitrosoanilines.  $\text{Cp}^* = \text{C}_5\text{Me}_5$ .

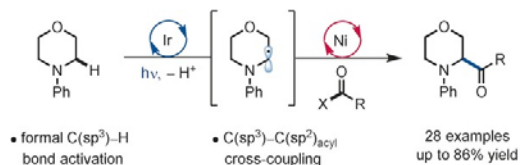
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## Cross-Coupling

### Direct Acylation of C(sp<sup>3</sup>)-H Bonds Enabled by Nickel and Photoredox Catalysis

Dr. Candice L. Joe, Prof. Abigail G. Doyle

Pages: 4040-4043 | First Published: 17 February 2016



**Teamwork:** The direct functionalization of C(sp<sup>3</sup>)-H bonds of *N*-aryl amines by acyl electrophiles is achieved, thus affording a diverse range of  $\alpha$ -amino ketones at room temperature. C(sp<sup>3</sup>)-H activation occurs by photoredox-mediated oxidation to generate  $\alpha$ -amino radicals which are intercepted by nickel in catalytic C(sp<sup>3</sup>)-C coupling.

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## Natural Product Synthesis

### Iridium-Catalyzed Enantioselective Indole Cyclization: Application to the Total Synthesis and Absolute Stereochemical Assignment of (-)-Aspidophylline A

Dr. Shi-Zhi Jiang, Xue-Yi Zeng, Xiao Liang, Ting Lei, Dr. Kun Wei, Prof. Yu-Rong Yang

Pages: 4044-4048 | First Published: 17 February 2016



**The enantioselective total synthesis** of (-)-aspidophylline A, including assignment of its absolute configuration has been accomplished. A key element of the synthesis is a highly enantioselective indole allylic alkylation/iminium cyclization cascade which

was developed by employing a combination of Lewis acid activation and an iridium/ligand catalyst. cod=1,5-cyclooctadiene, Tf=trifluoromethanesulfonyl.

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## Biological Inhibitors

### Bioisosteric Exchange of C<sub>sp<sup>3</sup></sub>-Chloro and Methyl Substituents: Synthesis and Initial Biological Studies of Atpenin A5 Analogues

Simon Krautwald, Dr. Christian Nilewski, Dr. Mihoko Mori, Prof. Dr. Kazuro Shiomi, Prof. Dr. Satoshi Ōmura, Prof. Dr. Erick M. Carreira

Pages: 4049-4053 | First Published: 17 February 2016





**Changing places:** Asymmetric synthesis and initial biological studies of two analogues of the complex II inhibitor atpenin A5 are reported. Bioisosteric exchange of C<sub>sp<sup>3</sup></sub>-chloro and methyl substituents resulted in analogues with essentially identical biological activity as inhibitors of complex II.

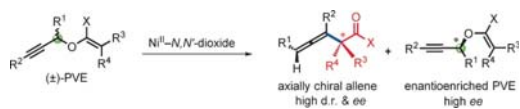
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## Chirality Transfer

### Synergistic Kinetic Resolution and Asymmetric Propargyl Claisen Rearrangement for the Synthesis of Chiral Allenes

Yangbin Liu, Prof. Dr. Xiaohua Liu, Haipeng Hu, Jing Guo, Yong Xia, Dr. Lili Lin, Prof. Dr. Xiaoming Feng

Pages: 4054-4058 | First Published: 17 February 2016



**Selective about neighbors:** A chiral nickel(II)-N,N'-dioxide complex catalyzed the highly efficient kinetic resolution and rearrangement of racemic

propargyl vinyl ethers (PVEs) to afford a chiral allene along with the enantiomerically enriched substrate (see scheme). The complete chirality transfer and facially selective rearrangement enabled the simultaneous construction of an axially chiral allenic unit and a neighboring quaternary stereocenter.

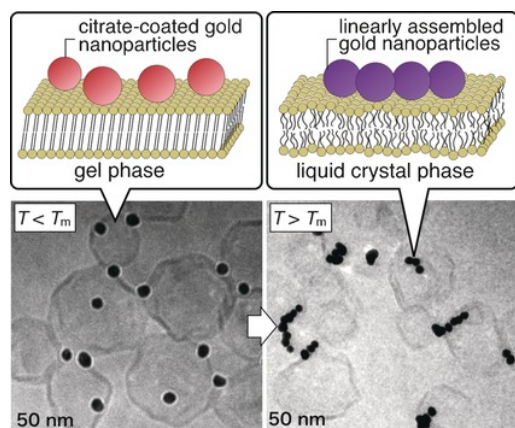
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## Liposome Functionalization

### Anisotropic Self-Assembly of Citrate-Coated Gold Nanoparticles on Fluidic Liposomes

Dr. Kouta Sugikawa, Tatsuya Kadota, Dr. Kazuma Yasuhara, Prof. Atsushi Ikeda

Pages: 4059-4063 | First Published: 23 February 2016



**Burnt orange peel:** Citrate-coated gold nanoparticles self-assembled into an anisotropic structure on lipid membranes by heating above the transition temperature of the lipid bilayer. The critical temperature for self-assembly was determined by lipid components.

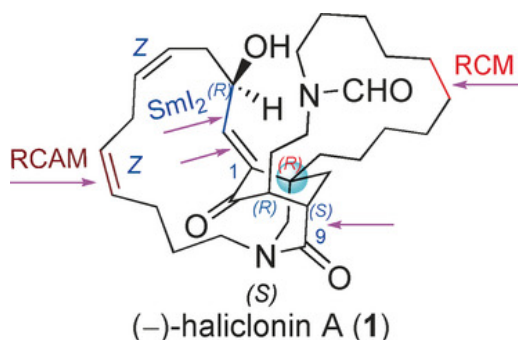
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## Total Synthesis

### Organocatalytic, Asymmetric Total Synthesis of (–)-Haliclونin A

Lian-Dong Guo, Xiong-Zhi Huang, Shi-Peng Luo, Wen-Sen Cao, Prof. Yuan-Ping Ruan, Dr. Jian-Liang Ye, Prof. Dr. Pei-Qiang Huang

Pages: 4064-4068 | First Published: 17 February 2016



**A natural product from the bench:** Key steps in the first total synthesis of (–)-haliclونin A include an organocatalytic asymmetric conjugate addition of nitromethane with 3-substituted cyclohexenone, Pd-promoted cyclization,  $Sml_2$ -mediated intermolecular reductive coupling of enone with aldehyde, metathesis reactions (RCM and RCAM) to build the macrocyclic ring systems, and a direct oxidation of enol to enone.

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## Natural Products

### Structural Revisions of a Class of Natural Products: Scaffolds of Aglycon Analogues of Fusicoccins and Cotylenins Isolated from Fungi

Dr. Ying Tang, Dr. Yongbo Xue, Prof. Guang Du, Prof. Jianping Wang, Prof. Junjun Liu, Bin Sun, Dr. Xiao-Nian Li, Prof. Guangmin Yao, Dr. Zengwei Luo, Prof. Yonghui Zhang

Pages: 4069-4073 | First Published: 24 February 2016

[Correction\(s\) for this article >](#)

#### Corrigendum: Structural Revisions of a Class of Natural Products: Scaffolds of Aglycon Analogues of Fusicoccins and Cotylenins Isolated from Fungi

Ying Tang, Yongbo Xue, Guang Du, Jianping Wang, Junjun Liu, Bin Sun, Xiao-Nian Li, Guangmin Yao, Zengwei Luo, Yonghui Zhang

Volume 57, Issue 46, Angewandte Chemie International Edition | pages: 14970-14970 | First Published online: September 11, 2018



**Brass tacks:** A class of natural products (NPs)



regarding the brassicene C-type diterpenoids was revised and characterized as the first class of bridgehead double-bond-containing NPs with a bicyclo[6.2.1]undecane carbon skeleton. This study

shows the potential of the application of computational prediction methods and biosynthetic logic-based structure elucidation to determining the structure and stability of NPs.

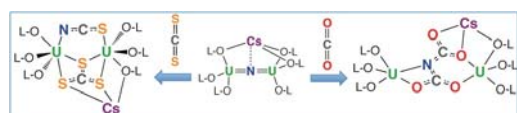
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## N–C Bond Formation

### Nucleophilic Reactivity of a Nitride-Bridged Diuranium(IV) Complex: CO<sub>2</sub> and CS<sub>2</sub> Functionalization

Marta Falcone, Lucile Chatelain, Dr. Marinella Mazzanti

Pages: 4074-4078 | First Published: 23 February 2016



**A nitride-bridged** diuranium(IV) complex rapidly reacts with CS<sub>2</sub> and CO<sub>2</sub> at room temperature. These transformations afford cyanate and thiocyanate species by N–C bond formation and enable the

unprecedented construction of a dicarbamate product (see scheme).

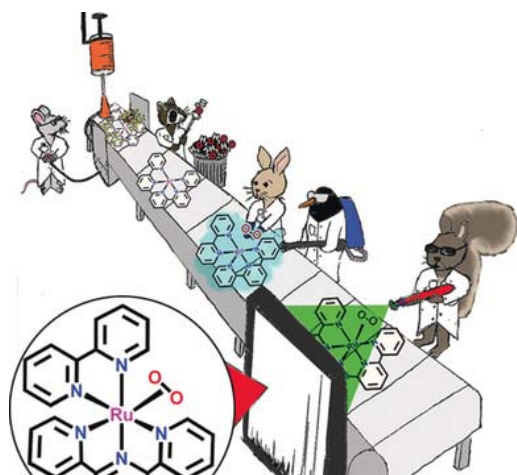
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## Vibrational Spectroscopy

### Characterization of the Oxygen Binding Motif in a Ruthenium Water Oxidation Catalyst by Vibrational Spectroscopy

Erin M. Duffy, Dr. Brett M. Marsh, Jonathan M. Voss, Prof. Etienne Garand

Pages: 4079-4082 | First Published: 17 February 2016



**The O<sub>2</sub> binding motif** in the [Ru(tpy)(bpy)(O<sub>2</sub>)]<sup>2+</sup> catalytic water oxidation intermediate is determined by using mass spectrometry and cryogenic ion IR predissociation spectroscopy. The O–O stretch, identified through <sup>18</sup>O<sub>2</sub> substitution, points to a singlet side-on bidentate O<sub>2</sub> complex. A comparison of experimental results with those from calculations show that DFT may inaccurately describe the Ru–O<sub>2</sub> interaction.



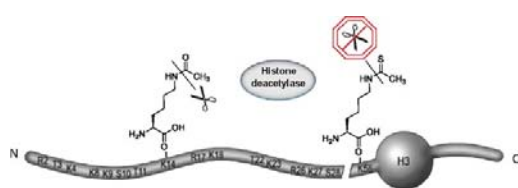
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## Post-translational Modifications

### Dual Genetic Encoding of Acetyl-lysine and Non-deacetylatable Thioacetyl-lysine Mediated by Flexizyme

Dr. Hai Xiong, Dr. Noah M. Reynolds, Dr. Chenguang Fan, Dr. Markus Englert, Dr. Denton Hoyer, Prof. Scott J. Miller, Prof. Dieter Söll

Pages: 4083-4086 | First Published: 23 February 2016



The modified histone H3 could not be removed by the histone deacetylase sirtuin.

**Director's cut:** The Flexizyme technique is used to incorporate acetyl-lysine and the non-hydrolyzable thioacetyl-lysine into full-length proteins in vitro and site-specifically into human histone H3, either individually or in pairs. The thioacetyl group of the

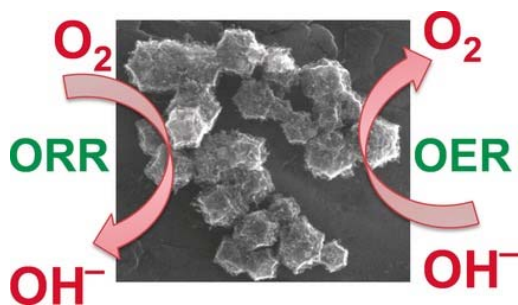
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## Electrocatalysis

### Co@Co<sub>3</sub>O<sub>4</sub> Encapsulated in Carbon Nanotube-Grafted Nitrogen-Doped Carbon Polyhedra as an Advanced Bifunctional Oxygen Electrode

Dr. Arshad Aijaz, Dr. Justus Masa, M. Sc. Christoph Rösler, Dr. Wei Xia, M. Sc. Philipp Weide, M. Sc. Alexander J. R. Botz, Prof. Dr. Roland A. Fischer, Prof. Dr. Wolfgang Schuhmann, Prof. Dr. Martin Muhler

Pages: 4087-4091 | First Published: 23 February 2016



**An electrocatalyst** consisting of Co@Co<sub>3</sub>O<sub>4</sub> embedded in carbon nanotube-grafted N-doped carbon polyhedra formed in situ has been fabricated from metal-organic frameworks. The electrocatalyst shows high activity towards water oxidation and oxygen reduction and outperforms Pt-, Ir-, and Ru-based electrocatalysts.

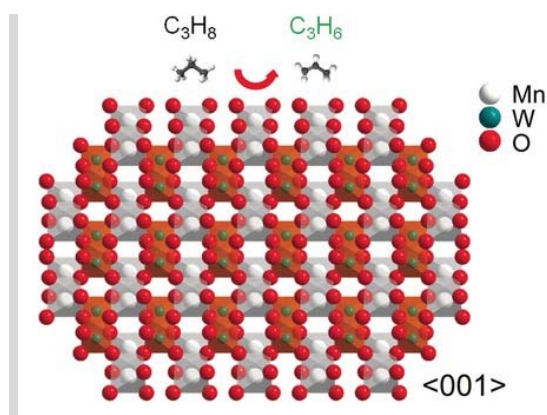
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# Heterogeneous Catalysis

## Selective Alkane Oxidation by Manganese Oxide: Site Isolation of $\text{MnO}_x$ Chains at the Surface of $\text{MnWO}_4$ Nanorods

Xuan Li, Dr. Thomas Lunkenbein, Verena Pfeifer, Mateusz Jastak, Pia Kjaer Nielsen, Dr. Frank Girgsdies, Dr. Axel Knop-Gericke, Dr. Frank Rosowski, Prof. Dr. Robert Schlögl, Dr. Annette Trunschke

Pages: 4092-4096 | First Published: 23 February 2016



**Site isolation** of surface manganese oxide species was obtained by the hydrothermal synthesis of nanostructured  $\text{MnWO}_4$ . In contrast to manganese oxide, which is a combustion catalyst,  $\text{MnO}_x$  chains on the exposed (010) crystal plane of  $\text{MnWO}_4$  selectively catalyze the oxidative dehydrogenation of propane to propene.

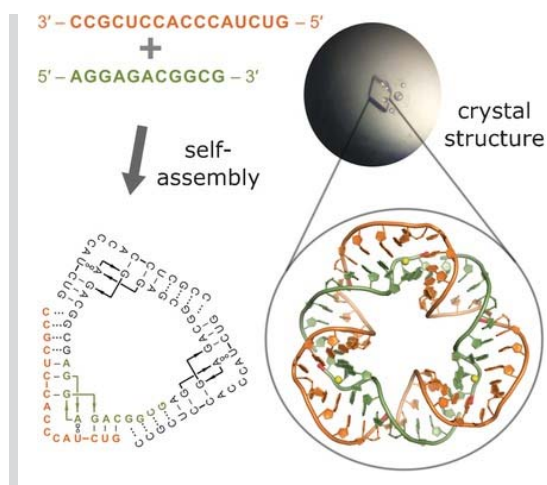
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# RNA Nanostructures | Hot Paper

## Crystal-Structure-Guided Design of Self-Assembling RNA Nanotriangles

Mark A. Boerneke, Sergey M. Dibrov, Thomas Hermann

Pages: 4097-4100 | First Published: 23 February 2016



### Design and characterization of RNA

**nanostructures:** Two short oligonucleotides self-assemble cooperatively in solution to form the so far smallest circularly closed nanotriangle made entirely of double-stranded RNA. This nanoobject formed crystals, and its structure was determined by X-ray diffraction at 2.6 Å resolution.

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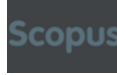
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