



The following Communications have been judged by at least two referees to be “very important papers” and will be published online at [www.angewandte.org](http://www.angewandte.org) soon:

I. Garcia-Bosch, A. Company, C. W. Cady, S. Styring, W. R. Browne, X. Ribas, M. Costas\*

**Evidence for a Precursor Complex in C–H Hydrogen-Atom-Transfer Reactions Mediated by a Manganese(IV) Oxo Complex**

G. N. Newton, S. Yamashita, K. Hasumi, J. Matsuno, N. Yoshida, M. Nihei, T. Shiga, M. Nakano, H. Nojiri, W. Wernsdorfer, H. Oshio\*

**Redox-Controlled Optimization of the Magnetic Properties of Keggin-Type {Mn<sub>13</sub>} Clusters**

C. C. Lee, Y. Hu,\* M. W. Ribbe\*

**Tracing the Hydrogen Source of Hydrocarbons Formed by Vanadium Nitrogenase**

S. R. Waldvogel,\* J. Kulisch, M. Nieger, F. Stecker, A. Fischer  
**Efficient and Stereodiverse Electrochemical Synthesis of Optically Pure Menthylamines**

J. Liu, S. Z. Qiao,\* H. Liu, J. Chen, A. Orpe, D. Zhao, G. Q. Lu\*  
**Extension of the Stöber Method to the Preparation of Monodisperse Spheres of Resorcinol–Formaldehyde Resin Polymer and Carbon**

T. Lewis, M. Faubel, B. Winter, J. C. Hemminger\*

**CO<sub>2</sub> Capture in an Aqueous Solution of an Amine: Role of the Solution Interface**

Y. H. Kim, S. Banta\*

**Complete Oxidation of Methanol in an Enzymatic Biofuel Cell by a Self-Assembling Hydrogel Created from Three Modified Dehydrogenases**

R. B. Bedford,\* M. F. Haddow, C. J. Mitchell, R. L. Webster  
**Mild C–H Halogenation of Anilides and the Isolation of an Unusual Pd<sup>I</sup>–Pd<sup>II</sup> Species**

W. Gan, B. Xu, H.-L. Dai\*

**Activation of Reactions of Thiols at the Silver-Nanoparticle Surface**

M. Sasaki, Y. Kondo, M. Kawahata, K. Yamaguchi, K. Takeda\*  
**Enantioselective Synthesis of Siloxyallenes from Alkynoyl Silanes by Reduction and a Brook Rearrangement and Their Subsequent Trapping in a [4+2] Cycloaddition with Unusual Facial Selectivity**

H. S. Choi, K. Nasr, S. Alyabyev, D. Feith, J. H. Lee, S. H. Kim, Y. Ashitate, H. Hyun, G. Patonay, L. Strekowski, M. Henary,\* J. V. Frangioni\*

**Zwitterionic Near-Infrared Fluorophores and Their Fate In Vivo**

## Author Profile



*“When I was eighteen I wanted to be a professional tennis player.*

*When I wake up I wake up my 8-year-old son ...”*

This and more about Guy Bertrand can be found on page 5248.

Guy Bertrand \_\_\_\_\_ 5248 – 5250

## Obituaries

Gernot Boche (1938–2011)

R. W. Hoffmann,  
M. Famulok\* \_\_\_\_\_ 5251 – 5252

## Books

From Non-Covalent Assemblies to Molecular Machines

Jean-Pierre Sauvage, Pierre Gaspard

reviewed by J. Nitschke \_\_\_\_\_ 5254

High Energy Density Lithium Batteries

Katerina E. Aifantis, Stephen A. Hackney,  
R. Vasant Kumar

reviewed by B. Scrosati \_\_\_\_\_ 5254

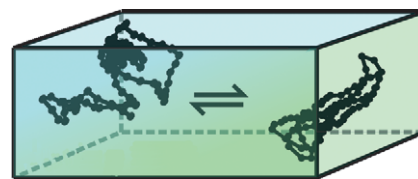
## Highlights

### Polymer Processing

F. A. Feist, T. Basché\* — 5256 – 5257

The Folding of Individual Conjugated Polymer Chains during Annealing

**Chain reaction:** Single-molecule spectroscopy can be used for the real-time observation of the translational and conformational dynamics of individual conjugated polymer chains during the annealing of solid films. Thus, a detailed molecular understanding of annealing mechanisms is at hand.

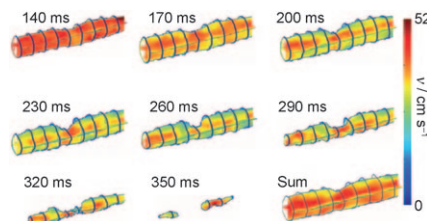


### Remote Detection

E. Paciok, B. Blümich\* — 5258 – 5260

Ultrafast Microscopy of Microfluidics: Compressed Sensing and Remote Detection

**Zooming in on microfluidics:** The potential of conventional NMR microscopy is limited by poor sensitivity and long measurement times. Recent advances in remote-detection NMR spectroscopy overcome these limitations and give unique insight into microfluidic processes with unprecedented spatial and temporal resolution (picture: high-resolution three-dimensional velocity maps of fast flow in a microcapillary).



### Mass Spectrometry

F. Coelho,\* M. N. Eberlin\* — 5261 – 5263

The Bridge Connecting Gas-Phase and Solution Chemistries



**Charged wings for flying fish:** Mass spectrometry allows the investigation of chemical reactions at the molecular level with ease, speed, selectivity, sensitivity, and great flexibility. Modern mass spectrometry provides a bridge that allows chemical reactions that occur in the gas phase to be studied in solution, and vice-versa (see picture).

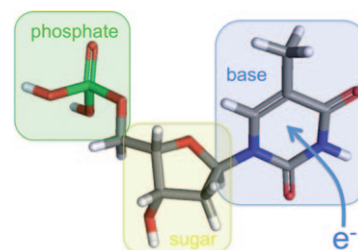
## Minireviews

### Electrons in Water

K. R. Siefertmann, B. Abel\* — 5264 – 5272

The Hydrated Electron: A Seemingly Familiar Chemical and Biological Transient

**Two recent milestones** in the research of the hydrated electron were the determination of its vertical binding energy and the discovery of a long-lived hydrated electron near the surface of liquid water. The results bear relevance for many fields, e.g., electron attachment to DNA bases (see picture).



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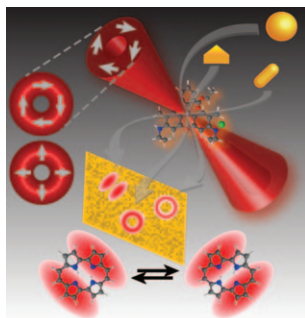
individuals who are personal members of a national chemical society prices are available on request. Postage and handling charges included. All prices are subject to local VAT/sales tax.

## Reviews

### Microscopy Techniques

T. Züchner, A. V. Failla,  
 A. J. Meixner\* \_\_\_\_\_ 5274 – 5293

Light Microscopy with Doughnut Modes:  
 A Concept to Detect, Characterize, and  
 Manipulate Individual Nanoobjects



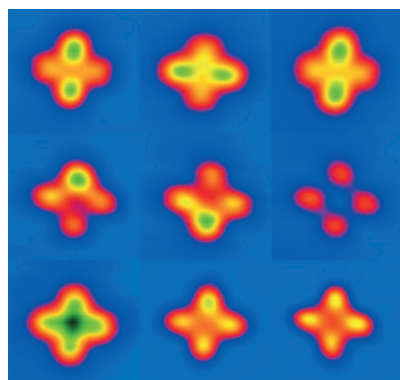
**Doughnuts for nanotechnology:** Higher-order laser modes with a doughnut-shape and a tunable polarization (see picture) have an extraordinary potential for new applications in nanotechnology. They already serve as versatile tools in microscopy and are finding use in many fields of science from pure optics to different branches of applied science including bio- and materials science.

## Communications

### Surface Chemistry

A. Sperl, J. Kröger,  
 R. Berndt\* \_\_\_\_\_ 5294 – 5297

Controlled Metalation of a Single  
 Adsorbed Phthalocyanine

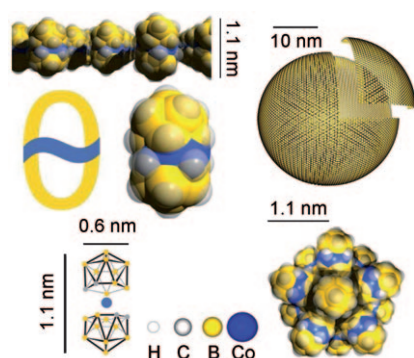


**Silvered molecules:** The controlled metalation of individual phthalocyanine ( $\text{H}_2\text{Pc}$ ) molecules to form  $\text{AgPc}$  was observed by low-temperature scanning tunneling microscopy (STM). Stepwise dehydrogenation was followed by  $\text{Ag}^+$  implantation; tautomerization and hydrogen atom hopping within the  $\text{H}_2\text{Pc}$  inner ring were also induced by electron injection from the STM tip.

### Vesicles

P. Bauduin,\* S. Prevost, P. Farràs,  
 F. Teixidor, O. Diat, T. Zemb \_\_\_\_\_ 5298 – 5300

A Theta-Shaped Amphiphilic  
 Cobaltabisdicarbollide Anion: Transition  
 From Monolayer Vesicles to Micelles



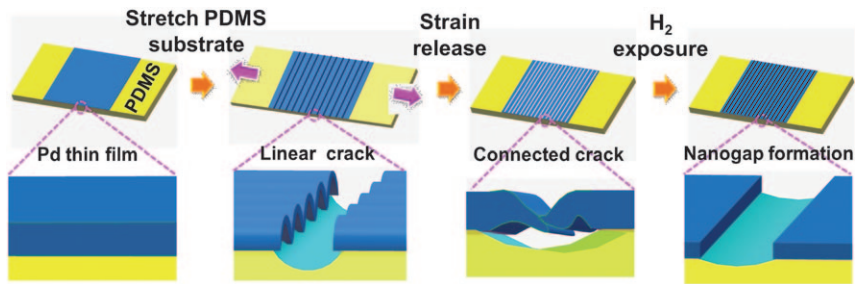
**Sandwiched:** The cobaltabisdicarbollide (mono-)anion ( $[\text{3,3'}\text{-Co}(\text{1,2-C}_2\text{B}_9\text{H}_{11})_2]^-$ ,  $\text{COSAN}^-$ ) forms monolayer vesicles at low concentrations in water (see picture). An increase in concentration leads to a Coulomb explosion of the closely packed vesicles into small micelles, which results in the coexistence of both aggregation states at higher concentrations.



J. Lee, W. Shim, E. Lee, J.-S. Noh,  
W. Lee\* 5301–5305



# Highly Mobile Palladium Thin Films on an Elastomeric Substrate: Nanogap-Based Hydrogen Gas Sensors

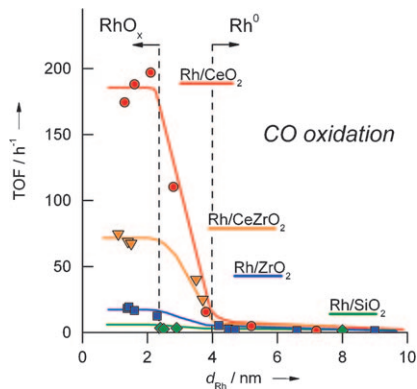


**MOTIFE chemical sensors:** A novel, low-cost, scalable, and lithography-free but nanogap-based chemical sensing method is presented. This method, termed highly-mobile thin film on elastomer (MOTIFE),

utilizes crack formation in a Pd and PdNi thin film generated by stretching the film on an elastomeric substrate to reliably and reproducibly provide highly sensitive H<sub>2</sub> sensors.

## CO Oxidation

D. A. J. M. Ligthart, R. A. van Santen,  
E. J. M. Hensen\* \_\_\_\_\_ **5306–5310**

Supported Rhodium Oxide Nanoparticles  
as Highly Active CO Oxidation Catalysts

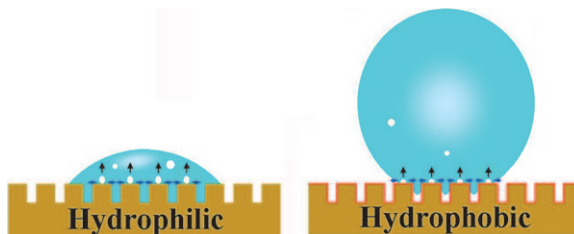
**Bigger is not always better:** Rhodium metal particles smaller than 2.5 nm are oxidized and stabilized by reducible supports such as ceria under CO oxidation conditions, whereas metal particles larger than 4 nm remain metallic. The very small Rh oxide particles are more active by two orders of magnitude in CO oxidation than Rh metal particles (see picture; TOF = turnover frequency).



T. Zhang, J. Wang,\* L. Chen, J. Zhai,  
Y. Song, L. Jiang\* \_\_\_\_\_ 5311–5314



# High-Temperature Wetting Transition on Micro- and Nanostructured Surfaces



**Wet, wet, wet:** Typical wetting transition (from spreading to bouncing) of water droplets on micro- and nanostructured surfaces at high temperatures has been investigated. The spreading and bouncing behavior varies on substrates with differ-

ent wettabilities (see picture; substrate brown, water droplet blue, water vapor white), and the bouncing temperature changes when the microstructure of the surface changes.

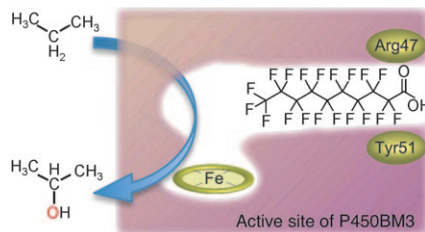
## Alkane Activation

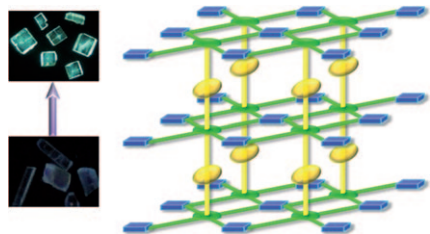
N. Kawakami, O. Shoji,  
Y. Watanabe\* \_\_\_\_\_ **5315–5318**



## Use of Perfluorocarboxylic Acids To Trick Cytochrome P450BM3 into Initiating the Hydroxylation of Gaseous Alkanes

**It has long been believed** that the fatty acid hydroxylase wild-type P450BM3 is unable to oxidize gaseous alkanes. However, the simple addition of a perfluorocarboxylic acid as a dummy substrate to initiate the P450BM3 catalytic cycle enabled the efficient hydroxylation of butane and propane (see picture).





**The same but different:** Four samples of an organic zinc phosphate hybrid material prepared at four different temperatures have the same structure but show distinct luminescence (see pictures of two of the samples).

## Luminescent Materials

S. H. Huang, S. L. Wang\* — 5319 – 5322

Variant Luminescence from an Organic–Inorganic Hybrid Structure with an Isolated 4-Ring Zinc Phosphate Tecton



**Mothball macrocycles:** Naphthalene has been coupled to form macrocyclic oligomers composed of five, six, and seven naphthalene units (see picture). Thermally stable macrocycles bearing 50, 60,

or 70  $\pi$  electrons within the hydrocarbon structure form columnar assemblies in crystals and serve as bipolar carrier transport materials in organic light-emitting diode devices.

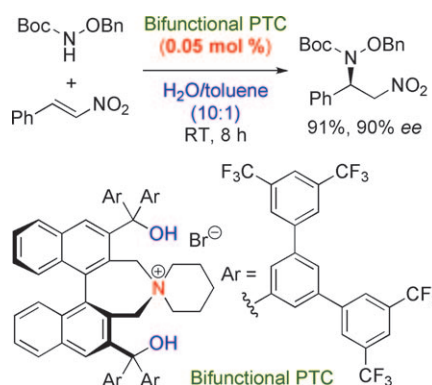
## Organic Materials

W. Nakanishi, T. Yoshioka, H. Taka, J. Y. Xue, H. Kita, H. Isobe\* — 5323 – 5326

[*n*]Cyclo-2,7-naphthylenes: Synthesis and Isolation of Macrocyclic Aromatic Hydrocarbons having Bipolar Carrier Transport Ability



**It's just a phase:** Environmentally benign title reaction was achieved under neutral phase-transfer conditions in the presence of 0.05 mol % of a chiral bifunctional ammonium bromide. The importance of bifunctional design of the chiral phase-transfer catalysts (PTC) was clearly shown in the transition-state model of the reaction based on the single-crystal X-ray structure. Bn = benzyl, Boc = *tert*-butoxy-carbonyl.



## Phase-Transfer Catalysis

L. Wang, S. Shirakawa, K. Maruoka\* — 5327 – 5330

Asymmetric Neutral Amination of Nitroolefins Catalyzed by Chiral Bifunctional Ammonium Salts in Water-Rich Biphasic Solvent



**It's the iodine:** The intra- and intermolecular title reaction is catalyzed by an in situ generated ammonium (hypo)iodite species. Either  $\text{H}_2\text{O}_2$  or *tert*-butyl hydroperoxide (TBHP) can be used as an environ-

mentally benign oxidant and a wide range of substrates react to give the corresponding  $\alpha$ -acyloxycarbonyl compounds in good to excellent yields.

## Oxidation

M. Uyanik, D. Suzuki, T. Yasui, K. Ishihara\* — 5331 – 5334

In Situ Generated (Hypo)Iodite Catalysts for the Direct  $\alpha$ -Oxyacylation of Carbonyl Compounds with Carboxylic Acids





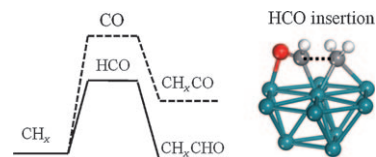
## Syngas Chemistry

Y. H. Zhao, K. J. Sun, X. F. Ma, J. X. Liu,  
D. P. Sun, H. Y. Su, W. X. Li\* **5335–5338**



Carbon Chain Growth by Formyl Insertion on Rhodium and Cobalt Catalysts in Syngas Conversion

**HCO insertion into  $\text{CH}_x$**  exhibits superior or similar activity to CO insertion and carbene coupling according to DFT calculations, which thus reveal a new reaction channel for chain growth in syngas conversion. The picture shows schematically the lower reaction barrier for HCO versus CO insertion and the optimized transition state for insertion of HCO into  $\text{CH}_2$ .

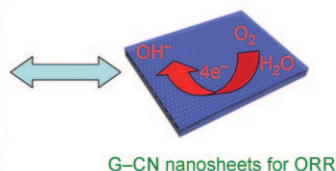
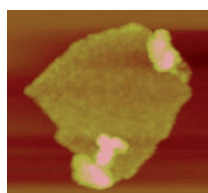


## Metal-Free Electrocatalysts

S. Yang, X. Feng,\* X. Wang,  
K. Müllen\* **5339–5343**



Graphene-Based Carbon Nitride Nanosheets as Efficient Metal-Free Electrocatalysts for Oxygen Reduction Reactions



**Sandwich-like, graphene-based** carbon nitride nanosheets (G-CN), among many other advantages, show an enhanced electrical conductivity. Oxygen atoms can thus access the catalyst surface easily and the rapid diffusion of electrons in the

electrode during the oxygen reduction process is facilitated. G-CN nanosheets can hence serve as metal-free electrocatalysts for oxygen reduction reactions (ORR) with excellent performance.

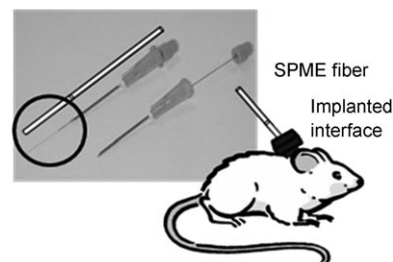
## Analytical Methods

D. Vuckovic, I. de Lannoy, B. Gien,  
R. E. Shirey, L. M. Sidisky, S. Dutta,  
J. Pawliszyn\* **5344–5348**



In Vivo Solid-Phase Microextraction: Capturing the Elusive Portion of Metabolome

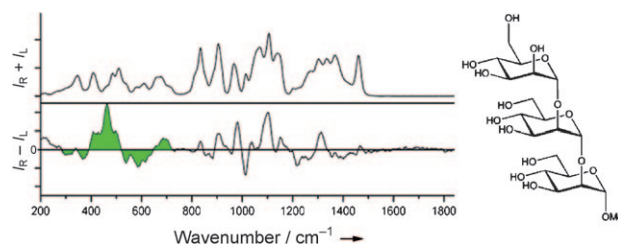
**Metabolites with fast turnover rates** and/or unstable metabolites can be captured directly from circulating blood using a new solid-phase microextraction (SPME) device based on a hypodermic needle. Such metabolites are not observed using solvent precipitation and ultrafiltration methods, indicating traditional approaches based on blood withdrawal are not adequately capturing complete metabolome at the time of sampling.



## Glycoproteins

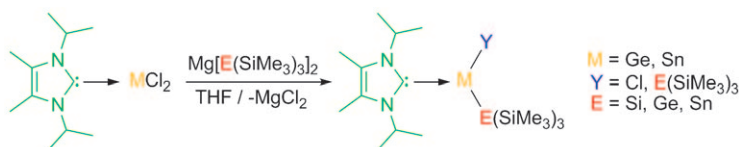
C. Johannessen, R. Pendrill, G. Widmalm,  
L. Hecht, L. D. Barron\* **5349–5351**

Glycan Structure of a High-Mannose Glycoprotein from Raman Optical Activity



**A revealing signature:** The glycan structure of intact yeast external invertase, a high-mannose glycoprotein used as biocatalyst, was investigated by using Raman optical activity (ROA) spectroscopy. The

conformational preferences present in mannose-containing di- and trisaccharides were found to be preserved in the glycan chains, with secondary polypeptide backbone structure suppressed.



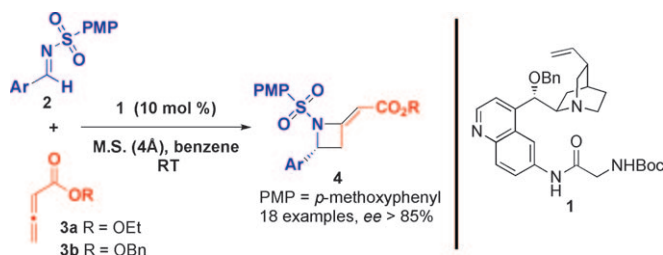
**A selective route:** Hypermetallyl germanium(II) and tin(II) compounds (see scheme) were prepared using dimetallyl magnesium reagents and were fully char-

acterized. These derivatives, which contain good leaving substituents, might be suitable candidates for the preparation of nanomaterial alloys.

## Group 14 Compounds

N. Katir, D. Matioszek, S. Ladeira, J. Escudié,\* A. Castel\* — 5352 – 5355

Stable N-Heterocyclic Carbene Complexes of Hypermetallyl Germanium(II) and Tin(II) Compounds



**Mix and go:** The quinidine amide **1** catalyzed [2+2] cycloaddition between *N*-sulfonylimines **2** and alkyl 2,3-butadienoates **3** afforded the *R*-configured azetidines **4** in excellent yields and enan-

tioselectivities (M.S. = molecular sieve). The *S* enantiomer was obtained when a quinine amide catalyst, the pseudoenantiomer of **1**, was used.

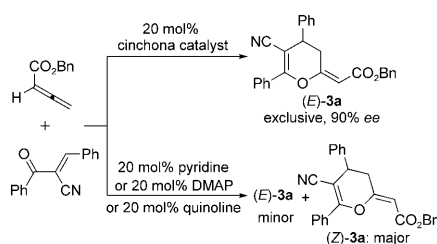
## Organocatalysis

J.-B. Denis, G. Masson,\* P. Retailleau, J. Zhu\* — 5356 – 5360

Cinchona Alkaloid Amide Catalyzed Enantioselective Formal [2+2] Cycloadditions of Allenates and Imines: Synthesis of 2,4-Disubstituted Azetidines



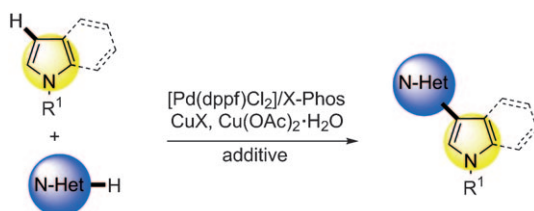
**Biologically significant** polysubstituted dihydropyrans have been prepared in high to excellent yields and enantioselectivities (see scheme). The interaction between functional groups in the zwitterionic intermediate, which is generated by addition of the amine catalyst to the allenolate substrate, is thought to play a crucial role in the stereochemical outcome. Bn = benzyl, DMAP = 4-dimethylamino-pyridine.



## Organocatalysis

X. Wang, T. Fang, X. Tong\* — 5361 – 5364

Enantioselective Amine-Catalyzed [4+2] Annulations of Allenates and Oxodienes: An Asymmetric Synthesis of Dihydropyrans



**Doubling up:** The highly regioselective C3 heteroarylation of either an indole or a pyrrole with an array of electron-rich and electron-poor *N*-heteroarenes has been carried out using a palladium/copper co-

catalytic system. The double C–H activation selectively delivers the unsymmetrical bi-heteroaryl product instead of the homocoupled products.

## Cross-Coupling

Z. Wang, K. Li, D. Zhao, J. Lan, J. You\* — 5365 – 5369

Palladium-Catalyzed Oxidative C–H/C–H Cross-Coupling of Indoles and Pyrroles with Heteroarenes

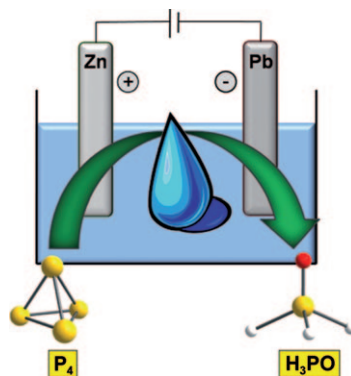


## Phosphorus Chemistry

D. Yakhvarov,\* M. Caporali, L. Gonsalvi,  
S. Latypov, V. Mirabello, I. Rizvanov,  
O. Sinyashin, P. Stoppioni,  
M. Peruzzini\* ————— 5370–5373



Experimental Evidence of Phosphine  
Oxide Generation in Solution and  
Trapping by Ruthenium Complexes



**Phosphine oxide** ( $\text{H}_3\text{PO}$ ), the first defined compound of phosphorus in the oxidation state  $-1$ , was obtained in solution by electrochemical methods starting from white phosphorus (see picture).  $\text{H}_3\text{PO}$  was characterized by NMR solution spectroscopy as a free molecule and isolated as a ligand in ruthenium(II) complexes following tautomerization to phosphinous acid,  $\text{H}_2\text{P}(\text{OH})$ .

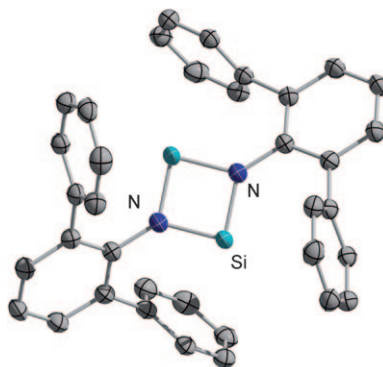


## Silicon Chemistry

R. S. Ghadwal, H. W. Roesky,\* K. Pröpper,  
B. Dittrich, S. Klein,  
G. Frenking\* ————— 5374–5378



A Dimer of Silaisonitrile with Two-  
Coordinate Silicon Atoms



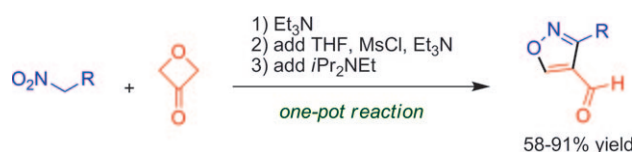
**A specialty of silicon:** A stable dimeric silaisonitrile ( $\text{ArNSi}:$ )<sub>2</sub> (see picture;  $\text{Ar} = 2,6\text{-bis}(2,4,6\text{-triisopropylphenyl})\text{-phenyl}$ ) was prepared by the reduction of dichlorosilaimine  $\text{IPr} \cdot \text{Cl}_2\text{Si}=\text{NAr}$  with  $\text{KC}_8$ . The dimer is the first base-free disilylene with two-coordinate silicon atoms; reaction with trimethylsilyl azide affords the first bis(silaimine) ( $\text{ArNSi}=\text{NSiMe}_3$ )<sub>2</sub> with three-coordinate silicon atoms.

## Synthetic Methods

J. A. Burkhard, B. H. Tchitchanov,  
E. M. Carreira\* ————— 5379–5382



Cascade Formation of Isoxazoles: Facile  
Base-Mediated Rearrangement of  
Substituted Oxetanes



**Give me five!** Nitro compounds and oxetan-3-one react through an intriguing cascade sequence to give isoxazole-4-carbaldehydes using inexpensive reagents

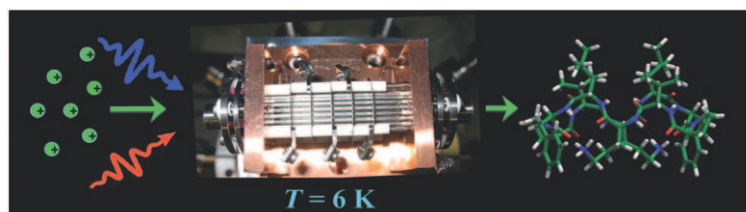
in a one-pot procedure (see scheme;  $\text{Ms} = \text{methanesulfonyl}$ ). A variety of 3-substituted isoxazole-4-carbaldehydes were obtained in high overall yields.

## Polypeptide Structures

N. S. Nagornova, M. Guglielmi,  
M. Doemer, I. Tavernelli, U. Rothlisberger,  
T. R. Rizzo, O. V. Boyarkin\* — 5383–5386



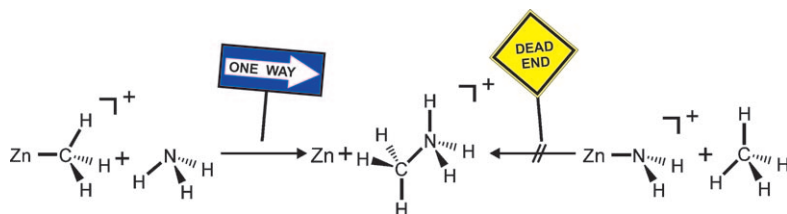
Cold-Ion Spectroscopy Reveals the  
Intrinsic Structure of a Decapeptide



**Trapped, cooled, solved:** Cold-ion spectroscopy was used to solve the three-dimensional gas-phase structure of the natural decapeptide gramicidin S. Experiments provide a detailed set of spectroscopic and structural constraints that

unambiguously identify the most stable calculated structure of the isolated peptide. These results provide new information for modeling the biological activity of this antibiotic.





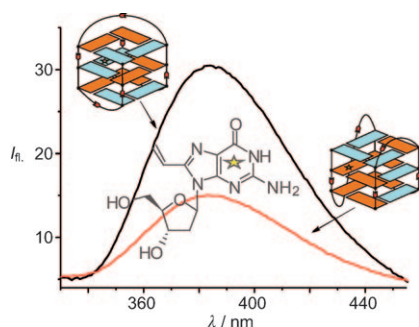
**Rally from C to N:** The goal of the race concerns connecting carbon and nitrogen. While the  $S_N2$ -type mechanism constitutes a one-way road to produce  $\text{CH}_3\text{NH}_3^+$

from  $\text{Zn}(\text{CH}_3)^+$  and  $\text{NH}_3$ , activation of the C–H bond of methane in the  $\text{Zn}(\text{NH}_2)^+/\text{CH}_4$  system is a blind alley (see scheme).

## Gas-Phase Chemistry

R. Kretschmer, M. Schlangen,  
 H. Schwarz\* 5387 – 5391

Efficient and Selective Gas-Phase Monomethylation versus N–H Bond Activation of Ammonia by “Bare”  $\text{Zn}(\text{CH}_3)^+$ : Atomic Zinc as a Leaving Group in an  $S_N2$  Reaction

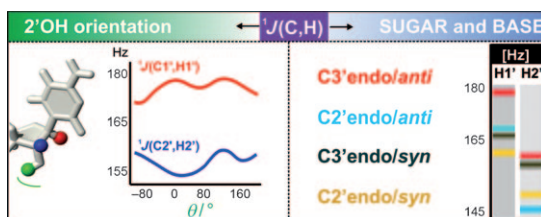


**A one light strand:** 2'-Deoxyguanosine was made fluorescent by attaching a vinyl group at the C8 position (see structure). The resulting fluorophore is highly sensitive towards DNA double-strand formation and alterations in the secondary structure of its parent oligonucleotide, such as formation of different quadruplex structures. These differences can be readily detected in the emission spectrum (see picture).

## Fluorescent Guanine

A. Nadler, J. Strohmeier,  
 U. Diederichsen\* 5392 – 5396

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## RNA Conformations

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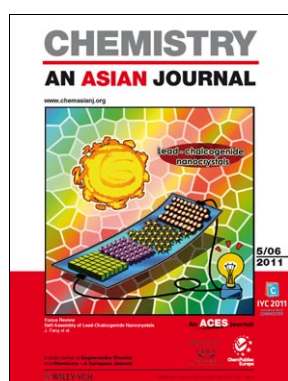
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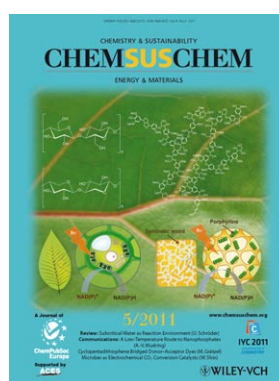
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