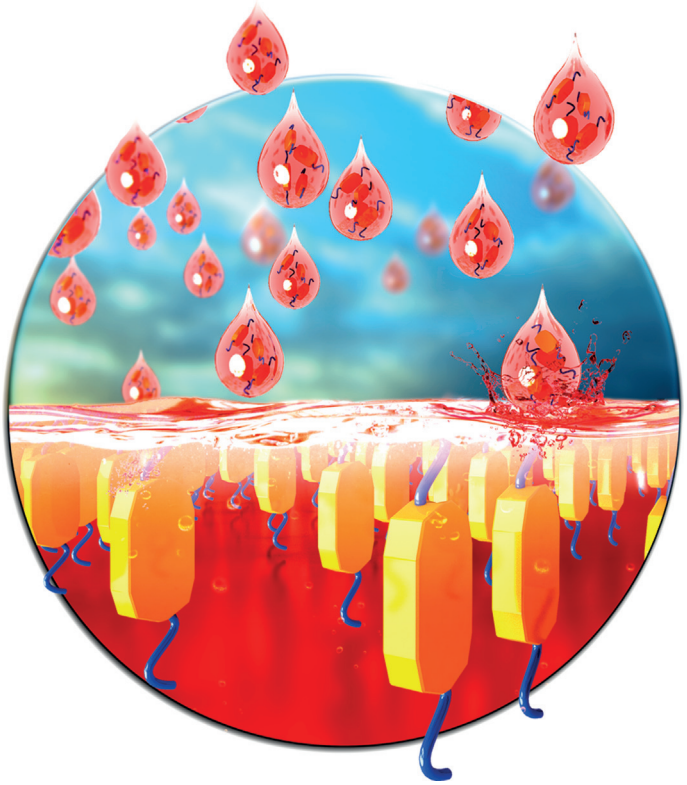
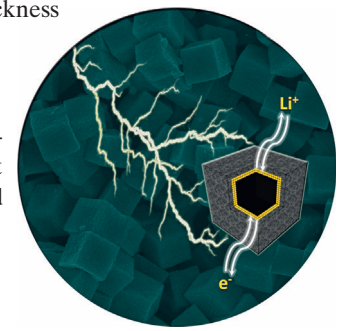


... is a method for growing high-quality two-dimensional crystals of organic semiconductors (2DCOS) up to centimeter size; these 2DCOS can be efficiently transferred to any substrate. In their Communication on page 9519 ff., W. Hu and co-workers show how this new method involves two steps: The self-assembly of micrometer-sized 2DCOS on the surface of water and their epitaxial growth into millimeter- or centimeter-sized 2DCOS with a thickness of several molecular layers.

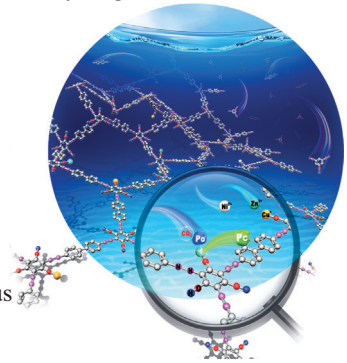
Lithium Storage

X. W. Lou and co-workers show in their Communication on page 9514 ff. how CoSe nanoboxes that have a carbon-enriched outer shell are a good anode material for lithium-ion batteries.



Photocatalysis

In their Communication on page 9580 ff., Z. R. Shen, J. C. Yu et al. report the use of fibrous-phase red phosphorus as a semiconductor photocatalyst for visible-light-driven hydrogen evolution.



CO_2 Conversion

Hierarchically mesoporous *o*-hydroxyazobenzene polymers are synthesized without any templates in water. In their Communication on page 9685 ff., Z. M. Liu and co-workers show that these porous polymers catalyze CO_2 conversion.

How to contact us:

Editorial Office:
E-mail: angewandte@wiley-vch.de
Fax: (+49) 62 01–606-331
Telephone: (+49) 62 01–606-315

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Online Open:
Margitta Schmitt
E-mail: angewandte@wiley-vch.de
Fax: (+49) 62 01–606-331
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Telephone: (+49) 62 01–606-565

Courier Services:
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Regular Mail:
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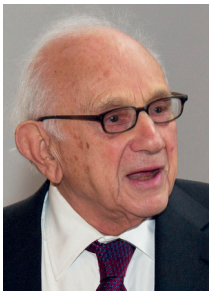
Service

Author Profile

"My favorite food is Sushi. If I were not a scientist, I would be an architect, artist, or designer of some sort. …"
This and more about Jón T. Njarðarson can be found on page 9468.

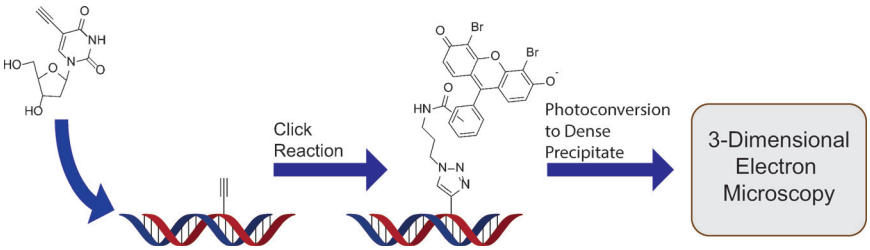
Obituaries



The German-born American historian Fritz Stern passed away on May 18, 2016 in New York. Stern dedicated his life's work to the study of European, particularly German, cultural history. Well aware that Germany's rise to preeminence during the 19th century was rooted in its scientific-technological culture, Fritz Stern created a portrait gallery of key protagonists of that "age of genius" which included Fritz Haber, his godfather.

Highlights

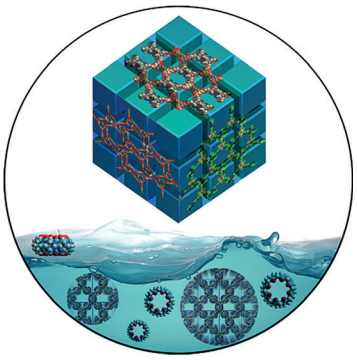
Imaging



Spitting image: Herein a recent paper on the imaging of bioorthogonal groups using three-dimensional electron microscopy is discussed. The work has demonstrated electron microscopy imaging as a technique suitable for gaining structural information on bioorthogonal groups in their cellular context.

Minireviews

Supramolecular Chemistry

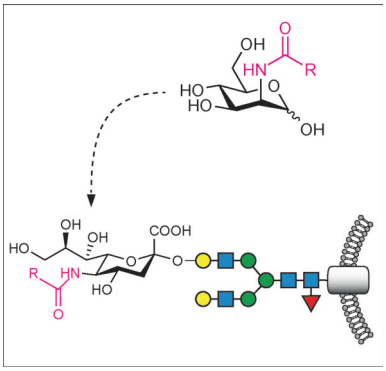


Porous organic molecular frameworks (POMFs) have recently been discovered as a new class of chemical materials for carbon storage and separation. The assembly of POMFs by means of primary hydrogen bonding interactions is reviewed based on their applications in both the solid state and solution phase.

Reviews

Metabolic Glycoengineering

Sugar for cells: Metabolic glycoengineering is a method to incorporate modified monosaccharides into cells, organs, or animals. Unnatural sialic acids can be generated by treatment with *N*-acyl side chain modified mannosamines. The application of this technique has intriguing biological consequences for treated cells. Mannosamine analogues bearing reactive groups can be used, for example, to visualize sialylated glycoconjugates.



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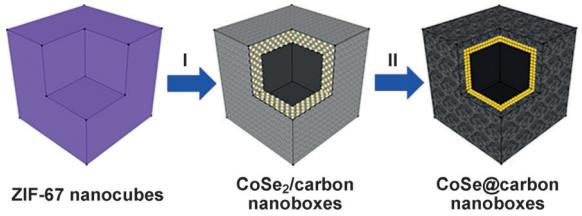
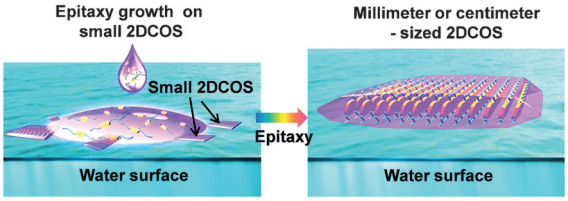
Communications



Box clever: Hybrid nanoboxes composed of a CoSe-enriched inner shell confined within a carbon-enriched outer shell are synthesized by a template-based method. With their structural and compositional advantages, these unique CoSe@carbon nanoboxes exhibit a superior electrochemical performance as an anode material for lithium-ion batteries.

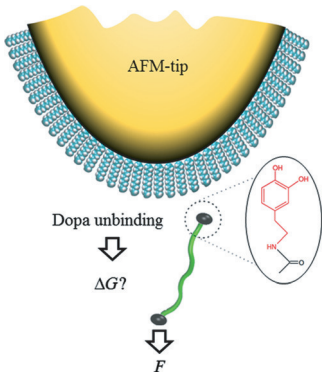
Hybrid Nanomaterials



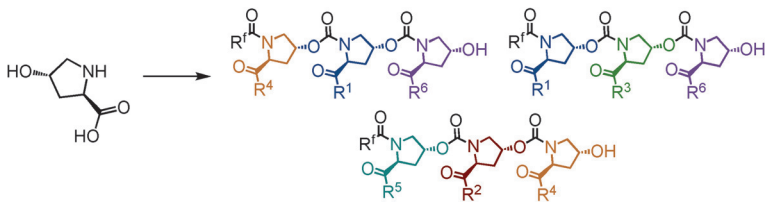
Water of crystallization: A novel method named "solution epitaxy" for the growth of 2D crystals of organic semiconductors (2DCOS) is based on the self-assembly of micrometer-sized 2DCOS on a water surface, and then epitaxial growth into centimeter sized 2DCOS with thickness of several molecular layers.

Organic Semiconductors

Biophysics: Dopa–surface interactions are very important for nanotechnology and surface modification (Dopa = 3,4-dihydroxyphenylalanine). The binding energies between Dopa and various surfaces were determined by single-molecule force spectroscopy (see picture; ΔG = free energy and F = force).



Surface Chemistry

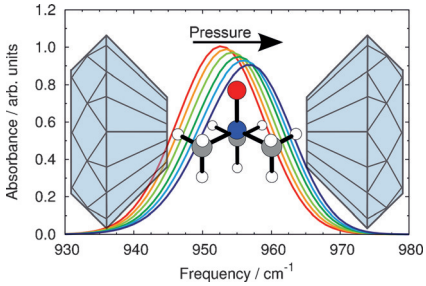


Another link in the chain: A controlled synthesis of novel oligomers from hydroxyproline-based building blocks was developed. The hydroxyproline-based monomer allows the incorporation of diverse side chains, and high-throughput purification can be achieved by fluorous solid-phase extraction. This method is adaptable to high-throughput synthesis in multiwell plates, thus allowing hundreds of novel oligomer structures to be synthesized in parallel.

Biomaterials

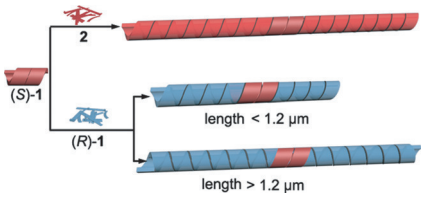
Vibrational Spectroscopy

Squeeze it out of them: Trimethylamine *N*-oxide (TMAO) is known to stabilize protein structures at extreme pressures, possibly through hydrogen bonding. A combination of FTIR spectroscopy and computer simulation has now been used to connect pressure-induced changes in the IR spectrum of TMAO (see picture) to a locally enhanced hydrogen-bonding network at high compression. The insight gained should be transferable to biomolecules in their native solvent.



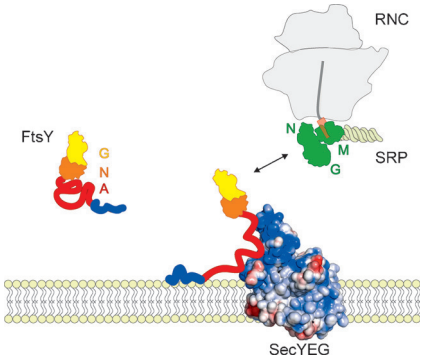
Chiral Nanotubes

A perfect role model: Chiral-selective linear nanotubule heterojunctions were achieved by living self-assembly. Chiral perylenediimide (PDI)-based nanotube seed (*S*)-**1** not only induced the supramolecular polymerization of microribbons of an achiral PDI derivative **2** at the seed ends in the same helical sense but could also polymerize the opposite enantiomer (*R*)-**1** in the same helical sense; this biasing depended on the final tube length.

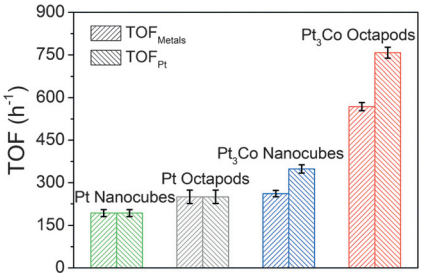


Biophysics

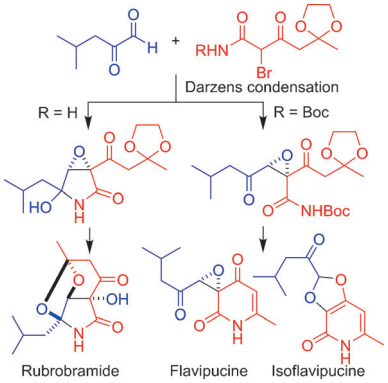
Integral membrane proteins in bacteria are co-translationally targeted to the translocon for membrane insertion via the signal recognition particle (SRP) pathway. The SRP receptor FtsY and its N-terminal A domain, which is lacking in any structural model of FtsY, were studied using NMR and fluorescence spectroscopy. Translocon targeting and binding of FtsY is driven by electrostatic interactions of the intrinsically disordered A domain.



Heterogeneous Hydrogenation

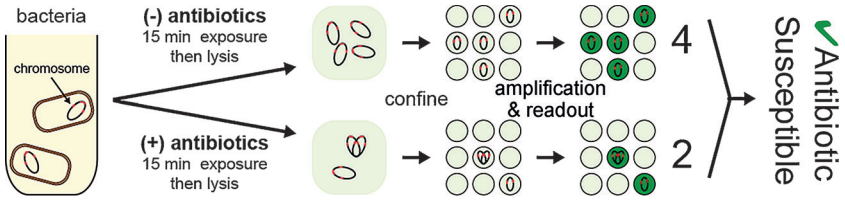


Pt_3Co octapods, Pt_3Co nanocubes, Pt octapods, and Pt nanocubes were used as catalysts for carbon dioxide hydrogenation. Owing to the multiple sharp tips and the charge transfer between Pt and Co, which leads to the accumulation of negative charge on the Pt atoms in the vertices, the Pt_3Co octapods were the most efficient catalyst of this process.



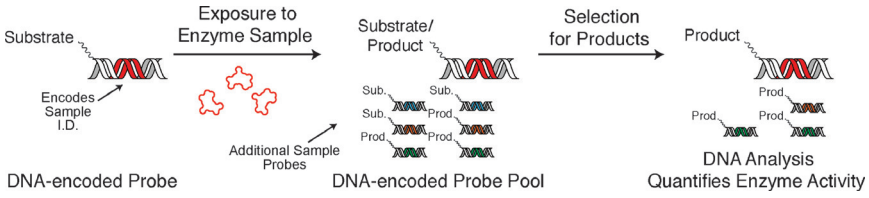
Three pieces: Total synthesis of (±)-rubrobramide, (±)-flavipucine, and (±)-isoflavipucine was achieved by a Darzens reaction between isobutyl glyoxal and α-bromo-β-ketoamides, removal of the protecting group(s), and ring formation. After optical resolution of synthetic (±)-rubrobramide, the absolute configuration of naturally occurring (+)-rubrobramide was determined by vibrational circular dichroism.

Natural Products



Single-molecule counting was used to detect subtle changes in the amount and the assembly state of bacterial chromosomes after a short exposure of live bacteria to antibiotics. The method employed digital PCR to determine the susceptibility of *E. coli* clinical isolates from urinary tract infections to four different antibiotics.

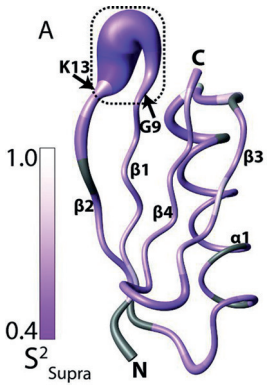
Analytical Methods



DNA-linked peptide substrates are used as probes of enzymatic activity. Encoding the sample identity in the DNA enables sensing of activity by DNA analysis. Probe phenotypes can be altered from substrate to product by enzyme exposure, affecting survival in a downstream selection. Selection-induced changes in probe frequency within a population quantify enzyme activity.

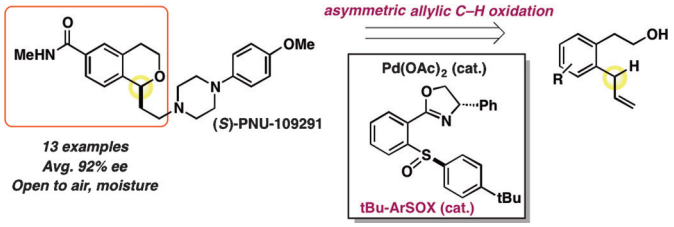
Analytical Methods

A link is made between ground-state conformational inter-conversion and molecular recognition. High-power relaxation dispersion measurements at supercooled temperatures, together with an ensemble representation of the third IgG-binding domain of protein G derived from residual dipolar couplings, point to a role for supra-τ_c dynamics in molecular recognition.



Protein Dynamics

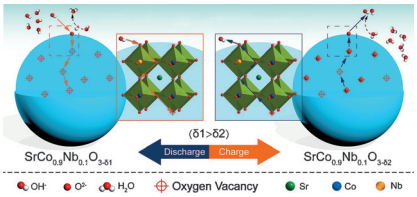
Asymmetric Catalysis



Selective and general: Palladium(II)/chiral sulfoxide catalysis affords an asymmetric allylic C–H oxidation reaction which proceeds with broad scope and high levels of enantioselectivity. The palladium(II)/ArSOX catalyst furnishes chiral isochromans from terminal olefin precursors and the reactions are run open to the air and moisture.

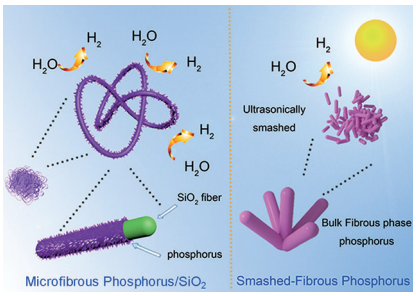
Energy Storage

Supercapacitors: The perovskite $SrCo_{0.9}Nb_{0.1}O_{3-\delta}$ (SCN) is a highly promising electrode material with an ultrahigh volumetric energy density for supercapacitors. Through an anion-insertion mechanism, this nonporous perovskite material cannot only achieve a high capacitance, but also shows an outstanding rate capability and excellent cycling stability.

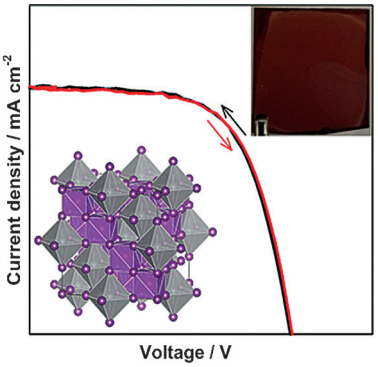


Photocatalysis

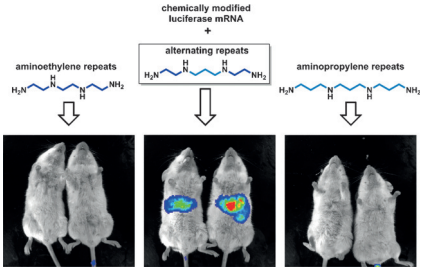
A breakthrough: Fibrous-phase red phosphorous was used as semiconductor photocatalyst for visible-light-driven hydrogen evolution. The phosphorus in micro-fibrous P/SiO_2 and smashed-fibrous phosphorus showed hydrogen evolution rates of 633 $\mu mol\,h^{-1}\,g^{-1}$ and 684 $\mu mol\,h^{-1}\,g^{-1}$, respectively. These values are records for elemental photocatalysts.



Thin-Film Photovoltaics



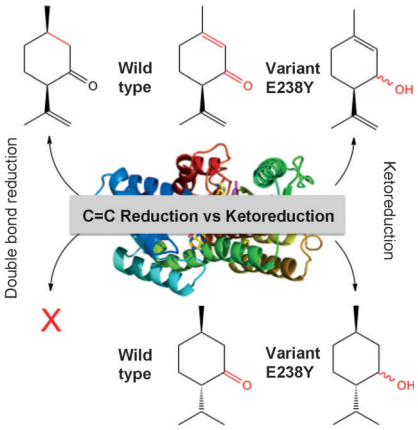
Solution-processed $AgBi_2I_7$ thin films are developed and deployed to produce air-stable and lead-free photovoltaic devices. The $AgBi_2I_7$ thin film crystallized in the cubic phase with dense and pinhole-free surface morphologies, and has an optical absorption that covers the visible spectrum and into the near-infrared region. The $AgBi_2I_7$ devices showed stable operation over 10 days under ambient conditions.



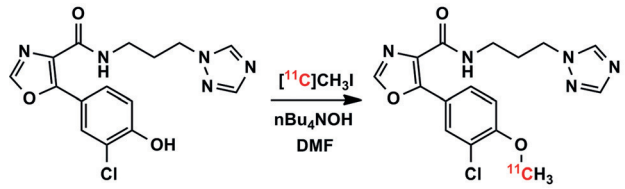
Oligoalkylamine-based lipids and polymers were generated and screened for buffering capacity and mRNA delivery. Alternating ethylene/propylene/ethylene spacers in the tetramines improve transfection efficiency (see picture, middle) over ethylene-only (left) or propylene-only repeats (right). This trend is valid regardless of delivery platform (lipids or polymers), tested cell line, targeted tissue, or application route in vivo.

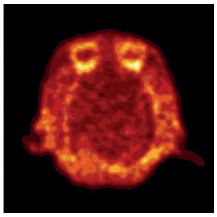
mRNA Delivery

A simple mechanistic switch: The replacement of tyrosine by glutamic acid in (−)-menthone:(+)-neomenthol reductase and of glutamic acid by tyrosine in isopiperitenone reductase enabled a switch between ene-reductase and ketoreduction activity in the short-chain dehydrogenases/reductases superfamily. This provides attractive routes to novel ene-reductase catalysts.



Enzyme Modifications

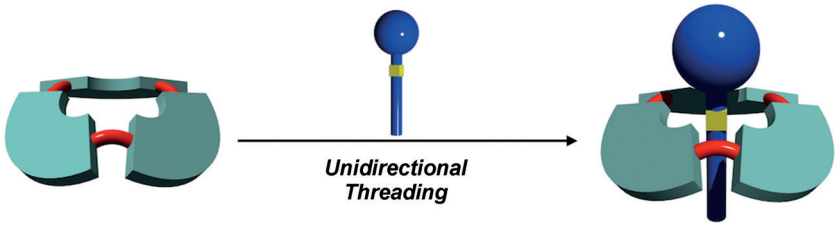




A highly potent inhibitor has been identified as a highly selective antagonist of glycogen synthase kinase-3 (GSK-3). Its efficacy in modulation of tau phosphorylation was demonstrated. A ^{11}C-isotopologue was synthesized, and preliminary PET imaging studies confirmed that the two major obstacles for imaging GSK-3 have been overcome: reasonable brain permeability and displaceable binding.

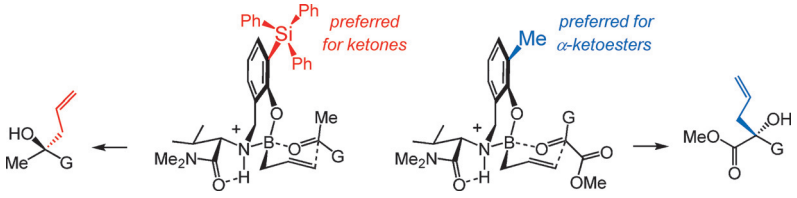
Phosphorylation

Molecular Recognition



Going in to bowl: A bowl-shaped macrocyclic trimer of a boron–dipyrrin complex has been synthesized in which six polarized B–F bonds are directed towards the center of the macrocycle for strong recognition. Cationic ammonium guests are strongly captured from the convex side of the macrocycle by hydrogen bonding, Coulomb forces, and C–H···π interactions and thread unidirectionally through the complex.

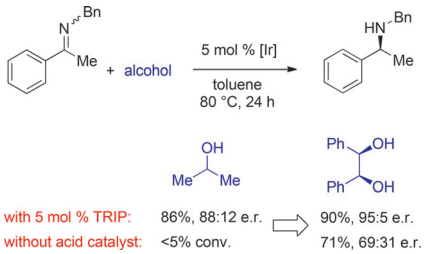
Enantioselective Synthesis



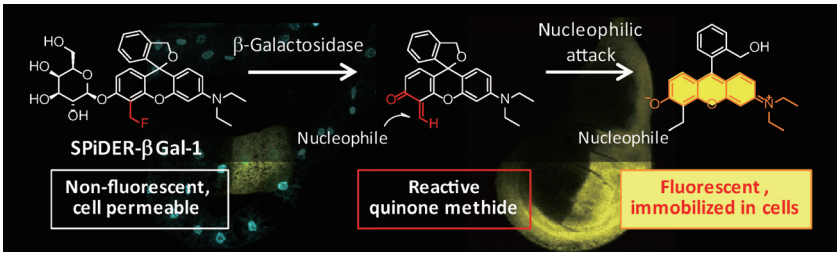
Easy to tune: A set of broadly applicable methods for efficient catalytic additions of easy-to-handle allyl-B(pin) (pin = pinacolato) compounds to ketones and acyclic α-ketoesters is introduced. Accordingly, a large array of tertiary alcohols can be obtained in up to around 98% yield and 99:1 e.r. Mechanism-based modification of the structures of aminophenol-based catalysts results in substantial improvement in enantioselectivity.

Reduction

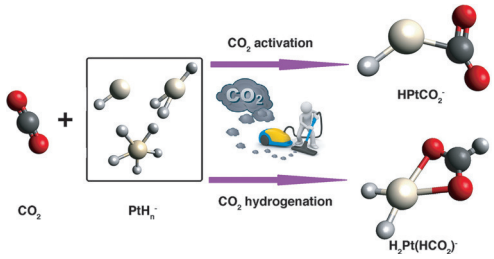
The influence of alcohol, as the hydrogen donor, on the efficiency and enantioselectivity of the asymmetric transfer hydrogenation (ATH) of imines is reported. This discovery not only leads to a highly enantioselective access to *N*-aryl as well as *N*-alkyl chiral amines, but also provides new insight into the mechanism of iridium-catalyzed ATH of imines using alcohol.



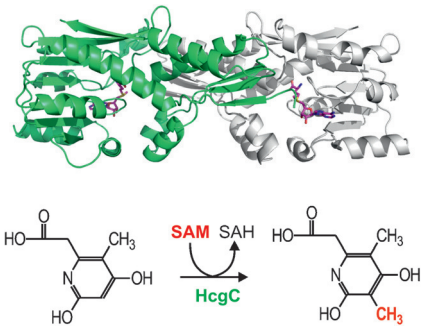
Fluorescent Probes



To detect *lacZ*-positive cells in living organisms or tissues at single-cell resolution, a fluorogenic β-galactosidase substrate, SPiDER-βGal-1, was synthesized. The substrate exhibited a dramatic increase in fluorescence upon reaction with the enzyme, remained inside cells by anchoring itself to intracellular proteins, and provided single-cell resolution.

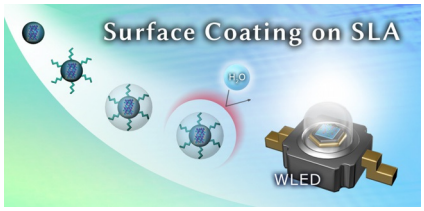


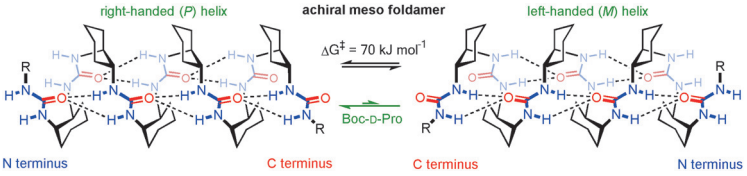
Metal hydride clusters: Naked (ligandless) transition metal hydride cluster utilization in CO_2 activation and fixation was observed for the first time. A number of PtH_n^- cluster anions were good hydrogen sources for the transformation of CO_2 into other higher value molecules, such as formate. Theoretical simulations validate the chemical reactions between the metal hydrides and CO_2.



Play it again, SAM: Biosynthesis of the iron guanylyl pyridinol cofactor of [Fe]-hydrogenase requires a challenging methyl transfer to the 3-position of the aromatic pyridinol ring. HcgC is predicted to be a novel *S*-adenosylmethionine (SAM)-dependent methyltransferase for catalyzing this reaction based on the crystal structures of HcgC with SAM and its demethylated product (SAH). The HcgC-catalyzed reaction was confirmed by enzymatic assays.

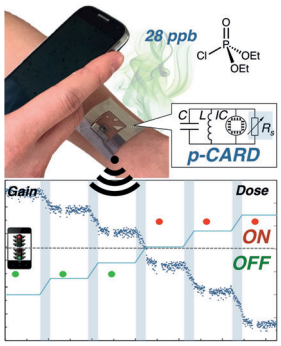
Stay dry and warm in this coat: The red-emitting phosphor $SrLiAl_3N_4:Eu^{2+}$ has superior luminescence properties but is water-sensitive and generally synthesized from metal-hydride precursors. These limitations were overcome by the use of a high-pressure synthetic method and the application of an organosilica coating. The resulting material with enhanced optical properties was used to make high-performance warm-white-light-emitting diodes.





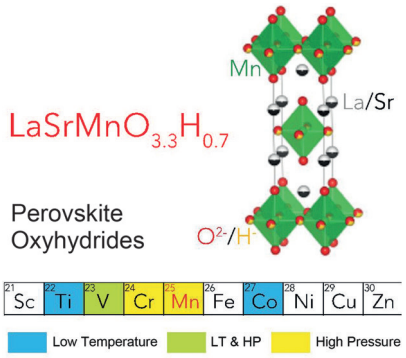
Reversible foldamers: Urea oligomers of *meso*-cyclohexane-1,2-diamine form a dynamic racemic mixture of $2.5_{12/14}$ helices in which screw-sense inversion is coupled with reversal of hydrogen-bond directionality. Desymmetrization induces a preferred directionality and screw sense.

Invisible dangers: A single-use, wirelessly addressable, highly sensitive, and selective chemical hazard badge that dosimetrically detects diethyl chlorophosphate, a nerve agent simulant, down to 28 ppb has been developed. The device comprises a wireless sensor platform and a chemidosimeter system incorporating single walled carbon nanotubes and ionic liquids. Exposure to 10 ppb DCP effects an irreversible switch in smartphone readout.



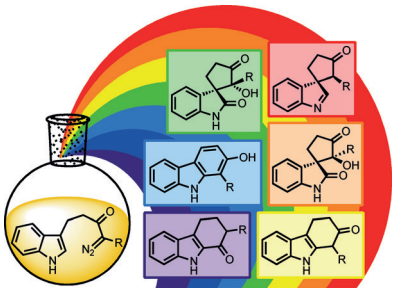
Sensors

A high-pressure reaction provides access to a new manganese oxyhydride, $LaSrMnO_{3.3}H_{0.7}$, as opposed to topochemical reactions that yield only reduced oxides. $LaSrMnO_{3.3}H_{0.7}$ crystallizes in a single-layer perovskite structure, where H^- ions are selectively located at the equatorial site. It exhibits a spin glass transition at 22 K owing to competing ferro- and antiferromagnetic interactions.

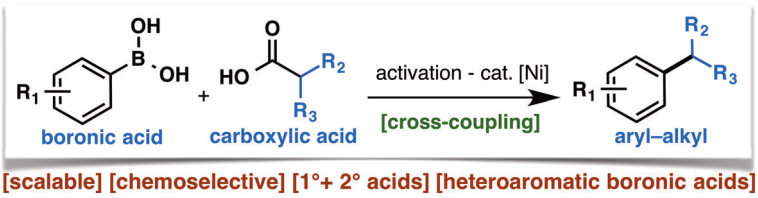


Solid-State Structures

Pick a catalyst: Indolyl α-diazocarbonyls can be selectively cyclized to give six distinct products through the careful choice of the catalyst and reaction conditions. A range of catalysts were used, including complexes of Rh^{II}, Pd^{II}, and Cu^{II} as well as SiO_2, to promote diazo decomposition and subsequent cyclization/rearrangement through a range of mechanistic pathways.

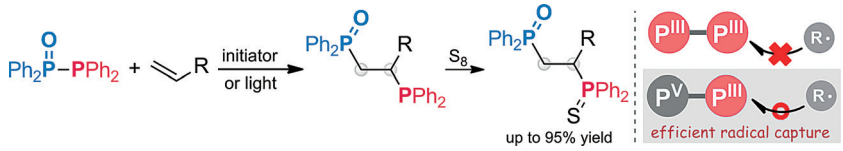


Diazo Compounds



A cross-coupling between alkyl carboxylic acids and aryl boronic acids is enabled by a new activation/cross-coupling strategy under Nickel catalysis. The operational simplicity and the wide range of heterocyclic compounds make a convenient strategy for obtaining aryl–alkyl cross-coupling products.

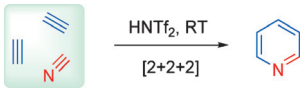
Cross-Coupling Reactions



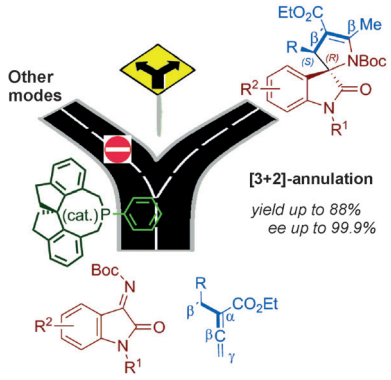
P(O)P by: $Ph_2P(O)PPh_2$ can engage in radical addition to various alkenes, thus affording the corresponding 1-phosphinyl-2-phosphinoalkanes regioselectively, and they can be converted into their sulfides by treatment with S_8. The reaction proceeds by homolytic cleavage of the $P^V(O)-P^{III}$ bond of $Ph_2P(O)PPh_2$, selective attack of the phosphinyl radical at the terminal position of the alkene, and selective trapping of the resulting carbon radical by the phosphino group.

Phosphanes

Triple two: A metal-free [2+2+2] cycloaddition of internal ynamides and nitriles was developed for the de novo synthesis of fully substituted pyridines. In contrast to metal catalysis, the versatile Brønsted acid $HNTf_2$ smoothly catalyzes the mild intermolecular cyclotrimerization of a range of ynamides and unactivated nitriles with complementary chemoselectivity.

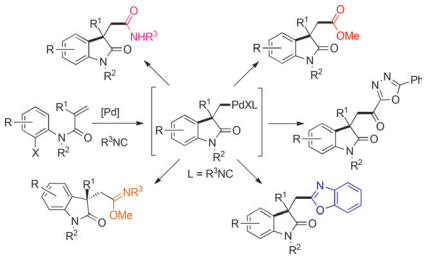


Cyclotrimerization



Taming a zwitterion: Chiral phosphine (*R*)- or (*S*)-SITCP generates a zwitterion from α-substituted allene ester that can engage the isatine derived ketimines in an unprecedented mode of [3+2] annulation reaction. This provides a facile asymmetric access to 3,2′-pyrrolidinyl spirooxindoles.

Asymmetric Phosphine Catalysis

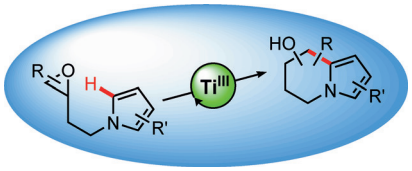


Setting a trap: The palladium-catalyzed reaction of N-aryl acrylamides with isocyanides affords diversely functionalized 3,3-disubstituted oxindoles by carbopalladation, migratory insertion of isocyanide, and nucleophilic trapping of the resulting alkylimidoyl–Pd^{II} complex. An enantioselective protocol has also been developed using 2,6-diisopropylphenyl isocyanide as a reaction partner and a chiral ligand.

Heterocycles

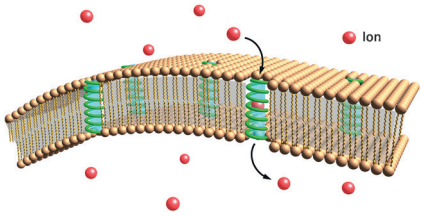
Catalysis

Bicycles Ti-cycles: The catalytic formation of dihydropyrrolizines and tetrahydroindolizines through titanocene(III) catalysis is described. The radical reaction is atom-economical, tolerates a large variety of functional groups, and proceeds with regioselectivity. It is therefore of interest for applications in the synthesis of biologically active compounds.

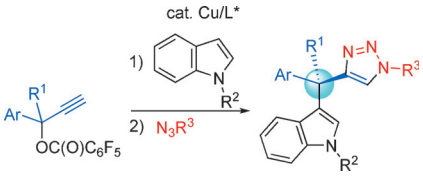


Helical Macromolecules

Opening the door: An artificial unimolecular transmembrane channel using pore-containing helical macromolecules is presented. The self-folding, shape-persistent helical macromolecules span the lipid bilayer, resulting in extraordinary channel stability and high transporting efficiency for protons and cations. The lifetime of this channel in the lipid bilayer membrane is impressively long, rivaling those of natural protein channels.



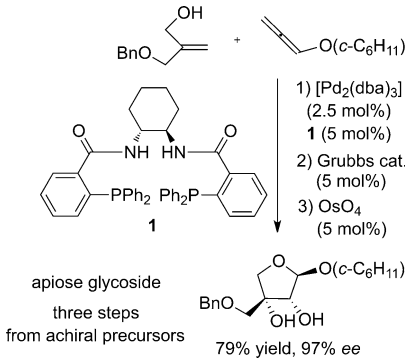
Enantioselectivity

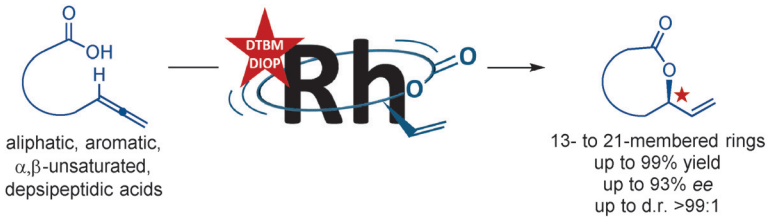


All-star all-carbon: The title reaction leads to the enantioselective construction of an all-carbon quaternary stereocenter. The subsequent derivatization of terminal alkyne moiety, into aromatic rings, is useful for the construction of chiral all-carbon substituted tri- and tetra-arylmethanes. The one-pot procedure demonstrated herein provides a successful example of the enantioselective preparation of a tetraarylmethane.

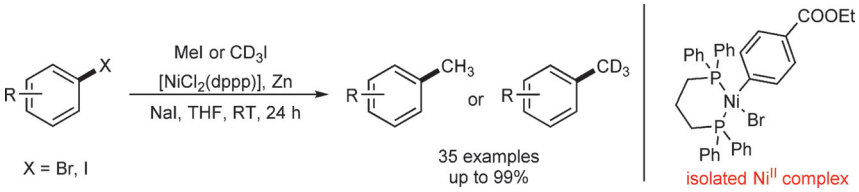
Carbohydrate Synthesis

Special sugars call for special measures: In a concise synthetic approach to apiose, a structurally unusual furanose, the palladium-catalyzed asymmetric intermolecular hydroalkoxylation of an alkoxyallene was followed by ring-closing metathesis and dihydroxylation (see scheme). This strategy enabled the efficient synthesis of various apiose-containing disaccharides and was used in a unique convergent synthesis of trisaccharides.

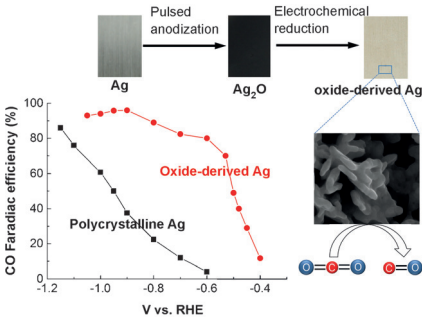




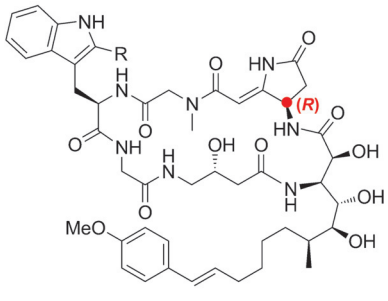
Reaching for stars: An intramolecular rhodium-catalyzed hydrooxycarbonylation of ω-allenyl-substituted carboxylic acids in the presence of a chiral phosphine ligand permitted the direct asymmetric formation of macrolactones (see scheme). The macrolactonization proceeded with good to high enantio- and diastereoselectivity.



Introducing CD_3: A nickel-catalyzed reaction suitable for the cross-coupling of aryl halides with cheap and readily available CH_3I or CD_3I is reported. The reaction is applicable to a wide range of substrates and produces the desired product in typically excellent yield. In particular, the reaction allows installation of a CD_3 group under the mild reaction conditions, without deuterium scrambling to other carbon atoms.

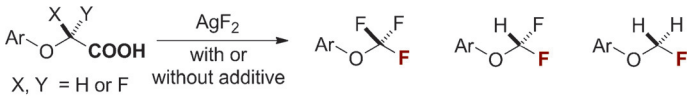


Nanostructured surfaces: An oxide-derived silver electrocatalyst was synthesized for the conversion of carbon dioxide to carbon monoxide with a high Faraday efficiency at low overpotentials. The nanostructure of the catalyst lowered the overpotentials as compared to polycrystalline silver by creating a high local pH value near the surface of the catalyst.



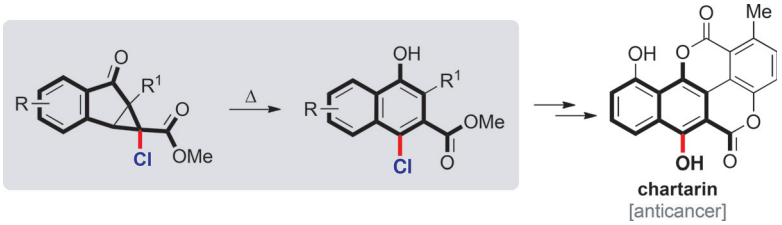
Not your everyday AAs: Efficient approaches to the unusual amino acids in the title compounds were developed on the basis of the Negishi coupling (for the Trp-2-CO_2H) and the Blaise reaction (for the pyrrolidinone unit). The original assignment of the pyrrolidinone stereostructure was confirmed to be incorrect by the synthesis of epimers, whose spectroscopic data were in complete agreement with those for the natural isolates (see structure).

Fluorination



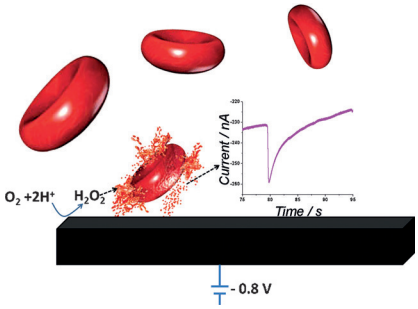
Radical F: AgF_2 induces fluorodecarboxylation of phenoxy difluoroacetic acids to afford trifluoromethyl aryl ethers. Pyridine additives modulate the reactivity of AgF_2, and enable the reaction to proceed with a variety of substrates.

Total Synthesis



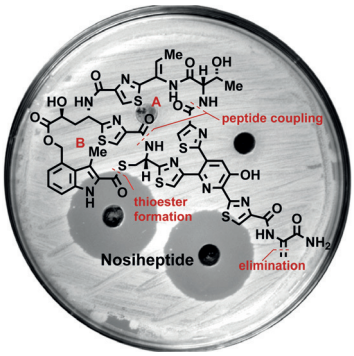
Substituted naphthalenes: Orthogonally functionalized naphthalenes were synthesized from commercially available indanones in four steps. The developed method proceeds through the thermally induced fragmentation of cyclopropane indanones and involves a regioselective 1,2-chloride shift. This transformation enables the synthesis of the anticancer agent chartarin.

Electrochemistry

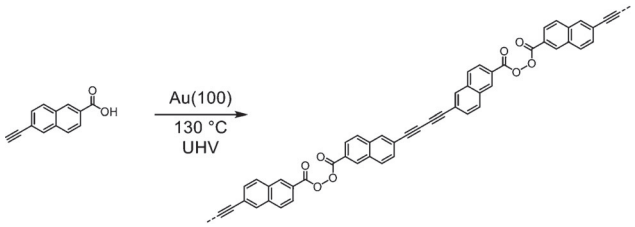


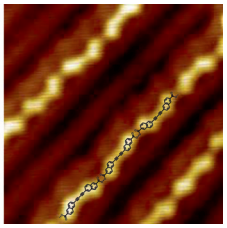
Catalytically active red blood cells: With the use of a nano-electrochemical technique fast and accurate red blood cell counting can be achieved together with complementary informations regarding the cell activity from each single cell. The method relies on the catalytic activity of red blood cells towards hydrogen peroxide and on surface-induced haemolysis.

Natural Product Synthesis



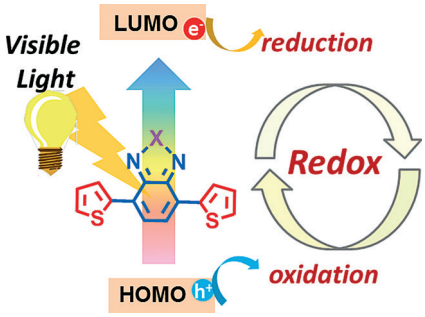
Each ring is different: A total synthesis of the bismacrocyclic thiopeptide antibiotic nosiheptide was achieved through the assembly of a fully functionalized linear precursor, followed by consecutive macrocyclizations. Key to success were the late-stage formation of a dehydroalanine residue and finely tuned deprotection of the hydroxypyridine core. The synthesis product was identical to the natural material in terms of structure and function.





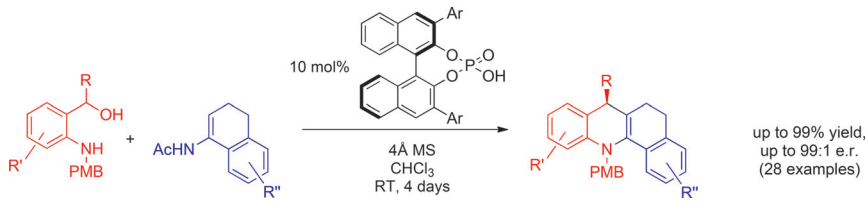
Surface Reactions

Playing dominos on gold: 6-Ethynyl-2-naphthoic acid (ENA) undergoes on-surface Glaser coupling on gold to form 6,6′-(1,4-buta-1,3-diynyl)bis(2-naphthoic acid) (BDNA) which in turn reacts in an unprecedented dehydrogenative coupling of the acid functionalities to give poly-BDNA. Polymeric chains with a length of over 100 nm can be obtained and the reaction outcome is influenced by the Au-surface topography and the surface coverage.



Photoredox Catalysis

Give and take: Small-molecule organic semiconductor donor–acceptor systems with a tunable absorption range and defined energy-band structure are designed as metal-free, pure organic, visible-light-active photocatalysts to meet crucial requirements, such as absorption in the visible region, sufficient light-generated redox potential, and long exciton lifetime.



Organocatalysis

Complex N-heterocycles in one shot: *ortho*-Quinone methide imines generated in situ react with enamides with high enantioselectivity under phosphoric acid catalysis giving valuable chiral tetrahydroacridines in one step. Subsequent diastereoselective hydrogenation provides saturated N-heterocycles with a total of three new stereocenters. PMB = *p*-methoxybenzyl.

Supporting information is available on www.angewandte.org (see article for access details).

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