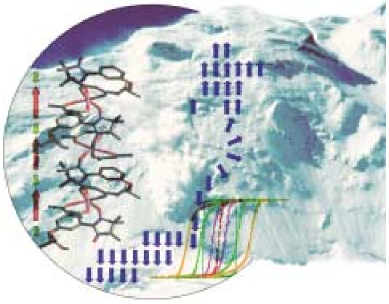
ANGEWANDTE
CHEMIE
A Journal of the
Gesellschaft
Deutscher Chemiker
INTERNATIONAL EDITION
2002
41/4
Pages 525–664

COVER PICTURE

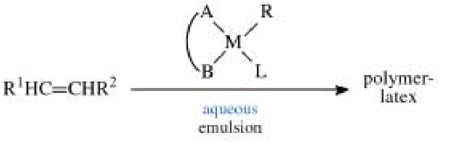
The cover picture shows a snow avalanche on the north face of Everest behind the structure of an enantiopure molecular ferromagnet which displays a rare phenomenon: a “magnetic avalanche”. The chiral complex [Mn(hfac)$_2$] ($S=\tfrac{5}{2}$, spins represented by the red arrows) with an enantiopure nitronyl nitroxide ligand ($S=\tfrac{1}{2}$, green arrows) exists as a coordination polymer (hfac = hexafluoroacetylacetonate). These chains are ferromagnetic (and are represented by the blue arrows), since the antiferromagnetically coupled organic- and metallic-centered spins do not compensate. At temperatures below 3 K these chains order magnetically and hysteresis loops open up. However, at 0.13 K (green curve) the width of the loop is smaller than at 0.3 K (orange curve). Thus, at a certain value of applied field there is an abrupt switching of some spins, which releases energy as a local heating. This heat initiates a switching of neighboring spins, which creates more heat and results in the magnetic equivalent of an avalanche. Further details about this optically active material are described by Veciana et al. on p. 586 ff. (Snow avalanche picture courtesy of Albert Castellet©, and thanks to Pere Oller of the Institut Cartográfic de Catalunya)

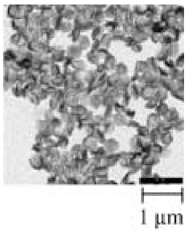


REVIEW

Contents

Water—a highly attractive, environmentally friendly reaction medium particularly for polymerization reactions. Whereas free-radical routes are well established, aqueous catalytic polymerization has seen major advances only recently. Numerous olefinic monomers can be transformed, for example, to aqueous polymer latices (see schematic reaction and picture).





Angew. Chem. **2002**, *114*, 564–582

S. Mecking,* A. Held,
F. M. Bauers 544–561

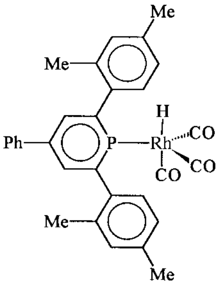
Aqueous Catalytic Polymerization of Olefins

Keywords: colloids · homogeneous catalysis · polymerizations · polymer latex · water chemistry

MINIREVIEW

Molecules with low-coordinate phosphorus atoms were considered as a domain for basic researchers for three decades. They were studied as building blocks in element–organic synthesis or as ligands in coordination chemistry. In the last few years transition metal complexes with phosphabenzene and phospholyl ligands have attracted attention as components in efficient catalysts. Thus, **1** is an efficient hydroformylation catalyst. This account also covers the use of such complexes for C–C coupling reactions, polymerization, and asymmetric synthesis.

Angew. Chem. **2002**, *114*, 583–592



1

L. Weber* 563–572

Phosphorus Heterocycles: From Laboratory Curiosities to Ligands in Highly Efficient Catalysts

Keywords: asymmetric catalysis · homogeneous catalysis · hydroformylation · phosphaheterocycles · P ligands

HIGHLIGHT

O_2, O_3, O_4, … O_n?

More than 200 years after the discovery of the element oxygen, recent experimental studies at very large as well as extremely low pressures suggest the existence of molecular tetraoxygen O_4 as a possible new modification of oxygen.

Angew. Chem. **2002**, *114*, 593–594

D. Schröder* 573–574

News about Oxygen

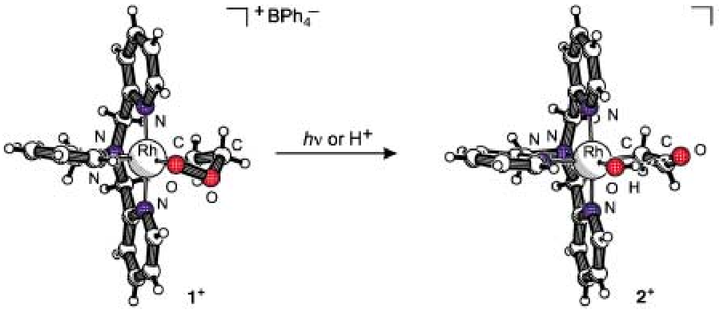
Keywords: allotropes · atmospheric chemistry · mass spectrometry · oxygen

VIPs

The following communications are “Very Important Papers” in the opinion of two referees. They will be published shortly (those marked with a diamond will be published in the next issue). Short summaries of these articles can be found on the *Angewandte Chemie* homepage at the address http://www.angewandte.com

A Nanoporous Metal-Organic Framework Based on Bulky Phosphane Ligands	X. Xu, M. Nieuwenhuyzen, S. L. James*	◆
Surface Structure and Crystal Growth in Zeolite Beta C	B. Slater,* R. A. Catlow, Z. Liu, T. Ohsuna, O. Terasaki, M. A. Camblor	
Callipeltoside A: Assignment of Its Absolute and Relative Configuration by Total Synthesis	B. M. Trost,* O. Dirat, J. L. Gunzner	◆
A New Catalyst for the Selective Oxidation of Butane and Propane	M. E. Davis,* C. J. Dillon, J. H. Holles, J. Labinger	◆
Surface Structure and Crystal Growth in Zeolite Beta C	B. Slater,* R. A. Catlow, Z. Liu, T. Ohsuna, O. Terasaki, M. A. Camblor	

Instead of direct conversion into a metal oxo complex and acetaldehyde or ethylene oxide, as was proposed earlier for 3-metalla-1,2-dioxolanes, 3-rhoda-1,2-dioxolanes **1**$^+$ have now been found to rearrange to rhodium formylmethyl hydroxy complexes **2**$^+$ upon exposure to light or protons.



Angew. Chem. **2002**, *114*, 595–599

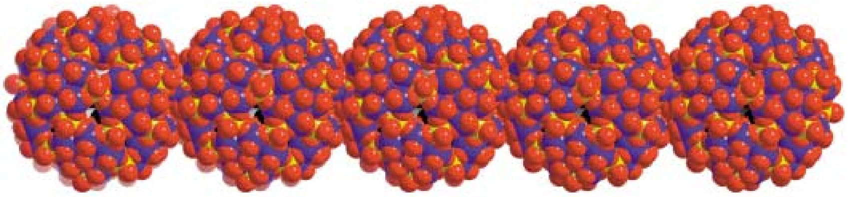
M. Krom, R. G. E. Coumans,
J. M. M. Smits, A. W. Gal* 575–579

Rearrangement of 3-Rhoda-1,2-dioxolanes to Rhodium Formylmethyl Hydroxy Complexes

Keywords: formylmethyl complexes · metallacycles · N ligands · photochemistry · rhodium

---

Crystal engineering with nanoobjects? Spherical structurally well-defined molybdenum-oxide-based giant clusters can be appropriately functionalized to a crystalline material with the remarkable property of having discrete cluster units which get covalently linked to form chains (see picture) through Fe–O–Mo bonds after having approached each other as a result of the release of crystal water upon drying.



Angew. Chem. **2002**, *114*, 599–602

A. Müller,* S. K. Das,
M. O. Talismanova, H. Bögge,
P. Kögerler, M. Schmidtmann,
S. S. Talismanov, M. Luban,
E. Krickemeyer 579–582

Paramagnetic Keplerate "Necklaces" Synthesized by a Novel Room-Temperature Solid-State Reaction: Controlled Linking of Metal-Oxide-Based Nanoparticles

Keywords: composites · magnetic properties · molybdenum · polyoxometalates · supramolecular chemistry

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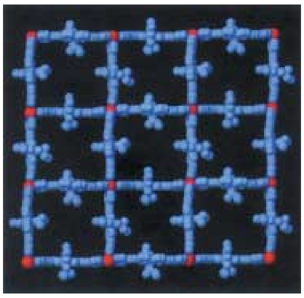
Use of a chiral, fluorene-based *N,N′*-bipyridine-type ligand (9,9-bis[(*S*)-2-methylbutyl]-2,7-bis-(4-pyridylethynyl)fluorene) with a Cu$^{\text{II}}$ salt led to the formation of the first chiral non-interpenetrating square-grid coordination polymer (see picture). The free space in the polymer can be controlled by adjusting the size of the side chains attached to the ligand. The large channels and the incorporation of chirality into the polymer make such compounds strong candidates for chiral recognition applications.



Angew. Chem. **2002**, *114*, 603–605

N. G. Pschirer, D. M. Ciurtin,
M. D. Smith, U. H. F. Bunz,*
H.-C. zur Loye* 583–585

Noninterpenetrating Square-Grid Coordination Polymers With Dimensions of 25 × 25 Å^2 Prepared by Using *N,N′*-Type Ligands: The First Chiral Square-Grid Coordination Polymer

Keywords: chirality · coordination polymers · copper · square grids

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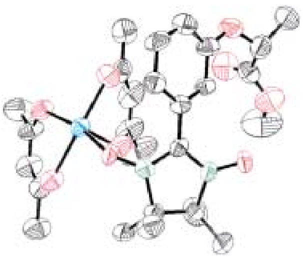
Magnetic avalanches: A chiral polymeric manganese(II) complex with an enantiopure nitronyl nitroxide ligand (see structure of repeating unit; red: oxygen, green: nitrogen, blue: manganese) shows unusual magnetic behavior. The polymer orders magnetically in the bulk at 3 K, and reveals unusual dynamic and hysteretic magnetic effects as well as a remarkable magnetic avalanche phenomenon below 0.3 K.



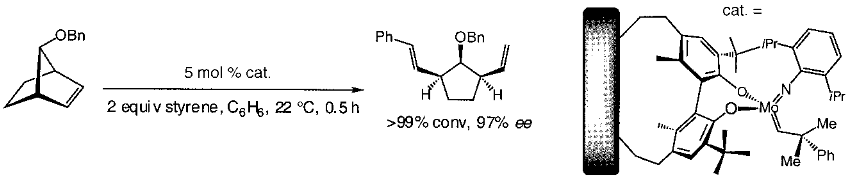
Angew. Chem. **2002**, *114*, 606–609

M. Minguet, D. Luneau,* E. Lhotel,
V. Villar, C. Paulsen, D. B. Amabilino,
J. Veciana* 586–589

An Enantiopure Molecular Ferromagnet

Keywords: chirality · ferromagnets · magnetic properties · manganese · O ligands · polymers

Substantially less toxic metal impurity than when unbound chiral complexes are used—this is achieved by the first polymer-supported chiral catalysts for olefin metathesis. These allow for efficient synthesis of various unsaturated carbo- and heterocycles in high optical purity through ring-opening (see scheme) and ring-closing reactions.



Angew. Chem. **2002**, *114*, 609 – 613

K. C. Hultzsch, J. A. Jernelius,
A. H. Hoveyda,*
R. R. Schrock* .... 589 – 593

The First Polymer-Supported and Recyclable Chiral Catalyst for Enantioselective Olefin Metathesis

Keywords: asymmetric catalysis · immobilization · metathesis · molybdenum · solid-phase synthesis

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A birefringent gel is formed when polycondensation of a strongly dipolar bis(trialkoxysilyl) compound is performed in an electric field. The birefringence is evidence for anisotropic ordering on the micrometer scale. The picture shows an image of the gel under a polarizing light microscope.

Angew. Chem. **2002**, *114*, 614 – 616

G. Cerveau, R. J. P. Corriu,* E. Framery,
S. Ghosh, M. Nobili .... 594 – 596

Nonrelaxable Anisotropic Organization of Organic – Inorganic Hybrid Materials Induced by an Electric Field

Keywords: birefringence · gels · organic – inorganic hybrid composites · self-assembly · sol – gel processes

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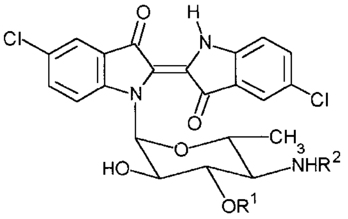
Sky blue akashins (see picture) are the first indigoglycosides and the first natural derivatives of 5,5′-dichloroindigo to be isolated from terrestrial Streptomyces.



Angew. Chem. **2002**, *114*, 623 – 625

R. P. Maskey, I. Grün-Wollny,
H. H. Fiebig, H. Laatsch* ..... 597 – 599

Akashins A, B, and C: Novel Chlorinated Indigoglycosides from *Streptomyces* sp. GW 48/1497

Keywords: antitumor agents · glycosides · indigo · natural products

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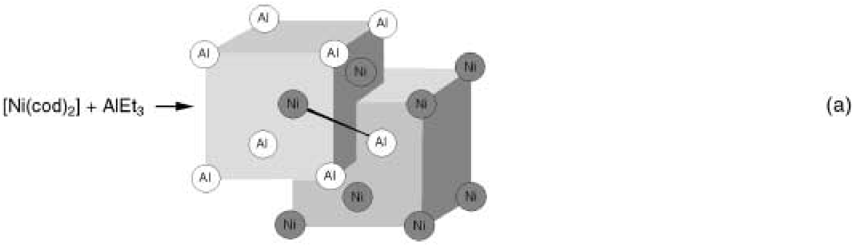
A fine powder of β-nickel aluminide NiAl is obtained by reaction of [Ni(cod)$_2$] (cod = cycloocta-1,5-diene) with $AlEt_3$ in toluene at room temperature and under a H_2 pressure of 5 – 10 MPa and subsequent distillation of the solvent, subsequent hydrogenation (0.1 – 5 MPa), and annealing (200°C) [Eq. (a)]. This novel wet chemistry synthesis of aluminides may be transferred to Ni_3Al (α-nickel aluminide) and ternary aluminide powders such as GaNiAl, FeNiAl, and CeNiAl.



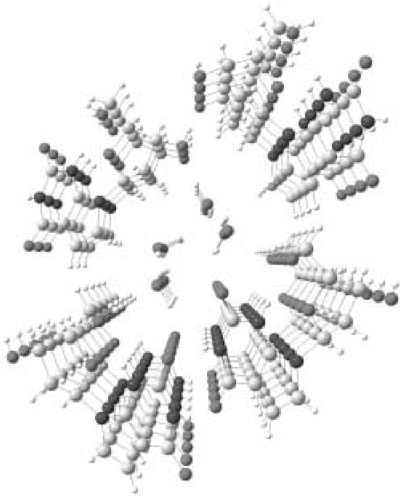
Angew. Chem. **2002**, *114*, 628 – 632

H. Bönnemann,* W. Brijoux,
H.-W. Hofstadt, T. Ould-Ely, W. Schmidt,
B. Waßmuth, C. Weidenthaler . 599 – 603

Wet Chemistry Synthesis of β-Nickel Aluminide NiAl

Keywords: aluminum · intermetallic phases · metal – metal interactions · nickel · solid-state structures

Supramolecular tetrameric aggregates of four molecules of 7-deaza-2′-deoxyxanthosine are stabilized by unconventional C–H ··· O hydrogen bonds. In the solid state the tetramers stack to form a nanotube (see picture), in the center of which water molecules are found which are also stacked.



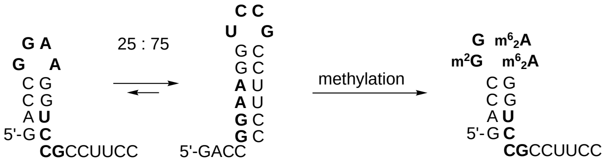
Angew. Chem. **2002**, *114*, 617–619

F. Seela,* T. Wiglenda, H. Rosemeyer, H. Eickmeier, H. Reuter* 603–605

7-Deaza-2′-deoxyxanthosine Dihydrate Forms Water-Filled Nanotubes with C–H ··· O Hydrogen Bonds

Keywords: hydrogen bonds • nanotubes • nitrogen heterocycles • nucleosides • oligonucleotides

One sequence, but two conformations! The coexistence of conformers has been ascertained for a series of RNA oligonucleotides. The monomolecular conformation equilibria are shifted significantly by methylation of selected nucleobases; the methylation of the studied sequences corresponds to that of the naturally occurring helix 45 at the 3′-end in the small subunit of ribosomal RNA.



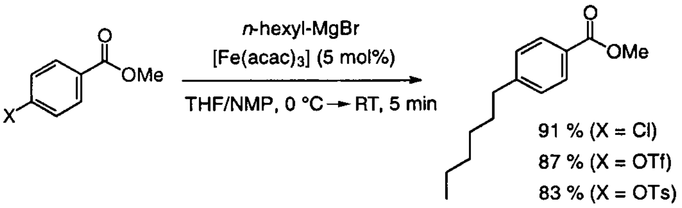
Angew. Chem. **2002**, *114*, 619–623

C. Höbartner, M.-O. Ebert, B. Jaun, R. Micura* 605–609

RNA Two-State Conformation Equilibria and the Effect of Nucleobase Methylation

Keywords: conformation analysis • nucleosides • oligonucleotides • RNA conformation equilibria • RNA structures

Aryl chlorides are better substrates than the corresponding bromides or iodides in the presented cross-coupling with alkyl Grignard reagents that is catalyzed by iron salts (see scheme); even aryl tosylates are converted efficiently. This situation is attributed to a novel catalytic cycle, which probably involves iron complexes with formally negative oxidation states (probably $\text{Fe}^{-\text{II}}$).



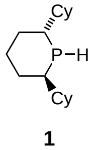
Angew. Chem. **2002**, *114*, 632–635

A. Fürstner,* A. Leitner 609–612

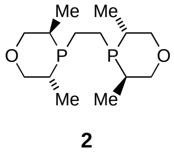
Iron-Catalyzed Cross-Coupling Reactions of Alkyl-Grignard Reagents with Aryl Chlorides, Tosylates, and Triflates

Keywords: cross-coupling • Grignard reagents • iron • magnesium • organometallic compounds

Obvious but unknown in asymmetric catalysis were chiral six-membered-ring phosphanes and secondary phosphanes. As first examples of such ligands, oxaphosphinanes were now prepared and examined in asymmetric hydrogenation. With the monodentate oxaphosphinane **1**, for example, 96% *ee* was achieved with itaconic acid as the substrate and 97.5% *ee* was achieved with the chelate ligand **2** and 2-acetamidoacrylic acid as the substrate.



1



2

Angew. Chem. **2002**, *114*, 625–628

M. Ostermeier, J. Prieß, G. Helmchen* 612–614

Mono- and Bidentate Phosphinanes—New Chiral Ligands and Their Application in Catalytic Asymmetric Hydrogenations

Keywords: asymmetric catalysis • chelate ligands • hydrogenation • phosphane ligands • P ligands

Complementary strands and redox intercalators can be self-assembled into nanoscale structures capable of charge transfer at the electrode surface from DNA oligomers synthesized directly at covalently modified semiconductor silicon surfaces (see schematic representation).

Angew. Chem. **2002**, *114*, 637–639

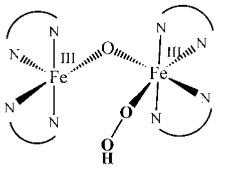
A. R. Pike, L. H. Lie, R. A. Eagling, L. C. Ryder, S. N. Patole, B. A. Connolly, B. R. Horrocks, A. Houlton* .. 615–617

DNA On Silicon Devices: On-Chip Synthesis, Hybridization, and Charge Transfer

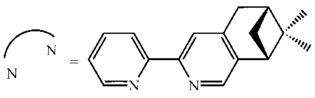
Keywords: charge transfer • DNA • molecular devices • semiconductors • silicon

---

In a spin: Transient peroxo iron complexes have been characterized by the means of Mössbauer and HF-EPR spectroscopies. Complex **1** has an unexpected $S=2$ ground state resulting from the coupling between the $S=5/2$ and $S=1/2$ iron(III) ions.



1



Angew. Chem. **2002**, *114*, 639–642

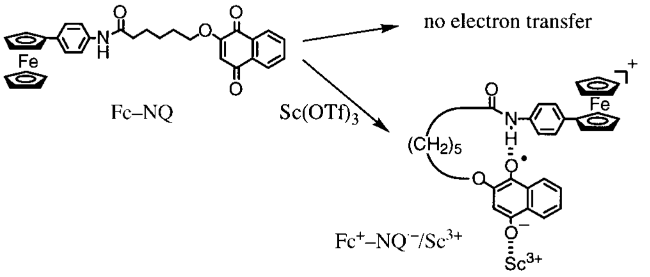
H. Hummel, Y. Mekmouche, C. Duboc-Toia, R. Y. N. Ho, L. Que, Jr., V. Schünemann, F. Thomas, A. X. Trautwein, C. Lebrun, M. Fontecave,* S. Ménage* ... 617–620

A Diferric Peroxo Complex with an Unprecedented Spin Configuration: An $S=2$ System Arising from an $S=5/2$, 1/2 Pair

Keywords: EPR spectroscopy • iron • metalloenzymes • O–O activation • peroxo ligands

---

Initiated by metal ions: Intramolecular electron transfer in a donor–acceptor linked system (a ferrocene–naphthoquinone dyad Fc–NQ; see scheme) can be started thermally by adding an appropriate metal ion (Sc^{3+}) which can promote thermal electron transfer, which would otherwise be unlikely to occur, to give a radical ion complex (Fc^+–$NQ^{\bullet-}$/Sc^{3+}).



Angew. Chem. **2002**, *114*, 642–644

S. Fukuzumi,* K. Okamoto, H. Imahori* 620–622

Thermal Intramolecular Electron Transfer in a Ferrocene–Naphthoquinone Linked Dyad Promoted by Metal Ions

Keywords: electron transfer • ESR • ferrocene • kinetics • scandium

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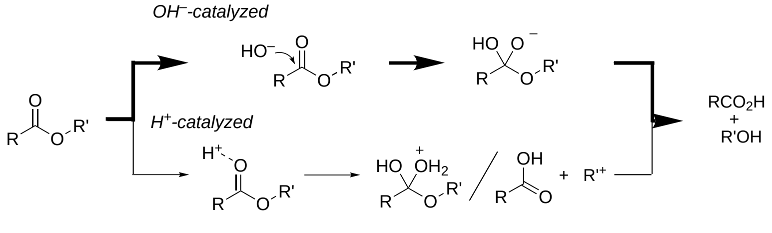
Not protons but hydroxide ions catalyze ester hydrolysis in supercritical water when the concentration of carboxylic acid generated by hydrolysis is low, as in the initial stage of the reaction. As the concentration of carboxylic acid increases with the progress of the reaction, the mechanism would become proton catalyzed (see scheme).



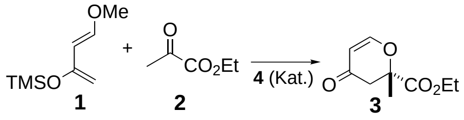
Angew. Chem. **2002**, *114*, 645–647

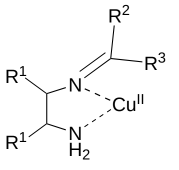
H. Oka, S. Yamago,* J. Yoshida,* O. Kajimoto* 623–625

Evidence for a Hydroxide Ion Catalyzed Pathway in Ester Hydrolysis in Supercritical Water

Keywords: esters • hydrolysis • kinetics • supercritical fluids

Only the correct order of addition of the components is required to prepare a variety of chiral Lewis acids. The complexes (for example, **4**) formed by the condensation of chiral, nonracemic 1,2-diamines with ketones and subsequent addition of $Cu(OTf)_2$ (Tf = trifluoromethanesulfonyl) catalyze the cycloaddition reaction of Danishefsky's diene (**1**) and ethyl pyruvate (**2**) to form dihydropyrone (**3**).





4

Angew. Chem. **2002**, *114*, 647–650

P. I. Dalko,* L. Moisan,
J. Cossy* 625–628

Modular Ligands for Asymmetric Synthesis: Enantioselective Catalytic Cu^{II}-Mediated Condensation Reaction of Ethyl Pyruvate with Danishefsky's Diene

Keywords: asymmetric catalysis · chiral auxiliaries · cycloaddition · Lewis acids

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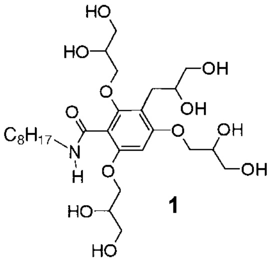
A new mesophase, composed of closed spheroidal aggregates that contain lipophilic alkyl chains within a continuum of hydrogen-bonding networks, was realized with the low molecular weight amphiphile **1** in the absence of water or any other solvent.



Angew. Chem. **2002**, *114*, 650–653

P. Fuchs, C. Tschierske,* K. Raith, K. Das,
S. Diele 628–631

A Thermotropic Mesophase Comprised of Closed Micellar Aggregates of the Normal Type

Keywords: amphiphiles · hydrogen bonds · liquid crystals · mesophases · micelles

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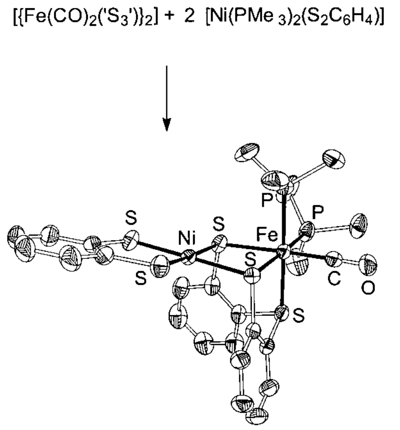
Attempts to model the active sites of [NiFe] hydrogenases have yielded numerous NiFe thiolate complexes but never a species with the characteristic $[(RS)_2Ni(\mu\text{-}SR)_2Fe(CO)(CN)_2]$ core. The Ni^{II}/Fe^{II} title complex represents the PMe_3 derivative. Its ν(CO) frequency of $1948\ cm^{-1}$ corresponds to that of $1940\ cm^{-1}$ for [NiFe] hydrogenase in the Ni–R state.



Angew. Chem. **2002**, *114*, 654–656

D. Sellmann,* F. Geipel, F. Lauderbach,
F. W. Heinemann 632–634

$[(C_6H_4S_2)Ni(\mu\text{-'}S_3\text{'})Fe(CO)(PMe_3)_2]$: A Dinuclear [NiFe] Complex Modeling the $[(RS)_2Ni(\mu\text{-}SR)_2Fe(CO)(L)_2]$ Core of [NiFe] Hydrogenase Centers

Keywords: carbonyl ligands · hydrogenases · iron · nickel · S ligands

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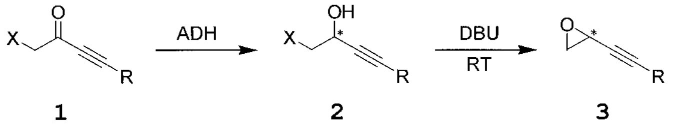
Halogenated propargylic alcohol 2a (X = Cl, R = Ph) was prepared by the enzymatic reduction of the corresponding ketone **1a** with excellent enantioselectivity and chemical yield. Both enantiomers were synthesized with high total turnover numbers of the catalysts and cofactors. Other halogenated alkynones **1** were also suitable substrates for the enzymes. Alcohols **2** were converted into propargylic epoxides **3** under mild conditions to give multifunctionalized building blocks. X = Cl, Br; R = H, Ph, trimethylsilyl, *tert*-butyldimethylsilyl; ADH = alcohol dehydrogenase; DBU = 1,8-diazabicyclo[5.4.0]undec-7-ene.



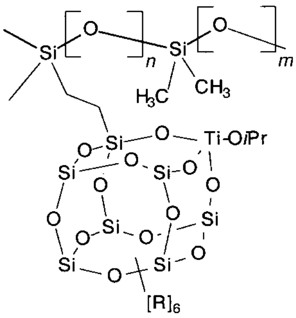
Angew. Chem. **2002**, *114*, 656–659

T. Schubert, W. Hummel,
M. Müller* 634–637

Highly Enantioselective Preparation of Multifunctionalized Propargylic Building Blocks

Keywords: asymmetric catalysis · enzyme catalysis · epoxides · ketones · oxidoreductases · reduction

Catalytic ensembles suitable for alkene epoxidation with aqueous hydrogen peroxide are now accessible. They are made by grafting robust titanium silsesquioxane sites onto dimethylsiloxane polymers (see picture) followed by cross-linking to three-dimensionally netted polymers.



Angew. Chem. **2002**, *114*, 659–661

M. D. Skowronska-Ptasinska,
M. L. W. Vorstenbosch, R. A. van Santen,
H. C. L. Abbenhuis* 637–639

Titanium Silsesquioxanes Grafted on Three-Dimensionally Netted Polysiloxanes: Catalytic Ensembles for Epoxidation of Alkenes with Aqueous Hydrogen Peroxide

Keywords: epoxidation · immobilization · silicon · silsesquioxanes · titanium

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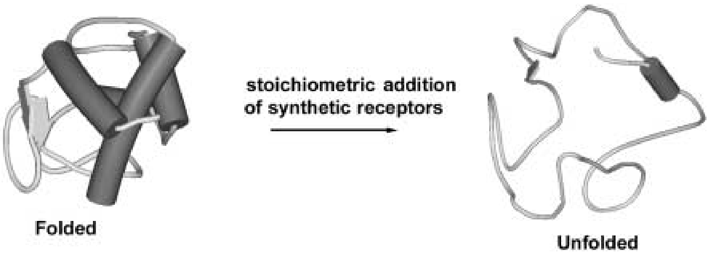
The structural diversity and marginal stability (5–15 kcal mol^{-1}) of proteins suggest the development of synthetic agents that selectively promote denaturation at low concentrations (see scheme). It is shown that an artificial receptor that binds tightly to the surface of native cytochrome *c* (cyt. *c*) lowers the melting temperature of the protein by 20°C at stoichiometric amounts; the receptor also selectively binds the native form rather than cyt. C551 and surface-modified cyt. *c*.



Angew. Chem. **2002**, *114*, 663–665

R. K. Jain, A. D. Hamilton* ... 641–643

Designing Protein Denaturants: Synthetic Agents Induce Cytochrome *c* Unfolding at Low Concentrations and Stoichiometries

Keywords: denaturation · molecular recognition · porphyrinoids · protein folding · protein surface

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A short-circuited solid-state battery: A novel silveroxonickelate with the apparently innocent composition Ag_2NiO_2 (see structure: silver: white, nickel: dark gray, fluorine: black) shows a surprising charge distribution: subvalent silver atoms exist adjacent to rather highly oxidized nickel(III) centers. The compound is thus reminiscent of a short-circuited solid-state battery.

Angew. Chem. **2002**, *114*, 665–668

M. Schreyer, M. Jansen* 643–646

Synthesis and Characterization of Ag_2NiO_2 Showing an Uncommon Charge Distribution

Keywords: high-pressure chemistry · magnetic properties · nickel · silver · subvalent compounds · transition metal oxides

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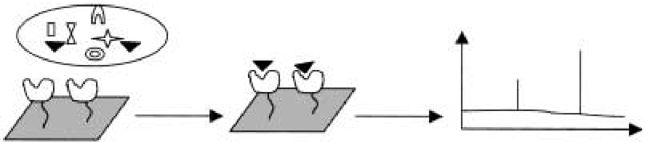
Simple and versatile: In a new assay for screening binding partners of proteins (see schematic representation), the target protein is first immobilized on a porous silicon probe, which is then incubated with a mixture of the possible binding partners. The captured binding partners are identified on-probe by means of laser desorption/ionization time-of-flight mass spectrometric analysis.



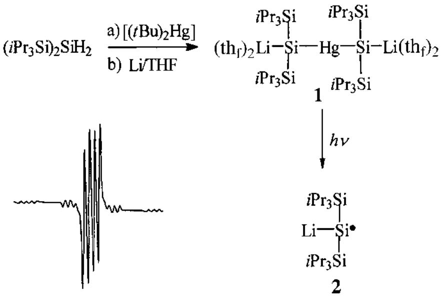
Angew. Chem. **2002**, *114*, 668–670

H. Zou,* Q. Zhang, Z. Guo, B. Guo,
Q. Zhang, X. Chen 646–648

A Mass Spectrometry Based Direct-Binding Assay for Screening Binding Partners of Proteins

Keywords: analytical methods · biosensors · drug research · immobilization · mass spectrometry · silicon

Radical developments in silyllithium chemistry: The preparation and X-ray structure of **1**, the first compound with geminal Hg-Si-Li bonding, is reported. Compound **1** is obtained by the reaction of $(i\mathrm{Pr_3Si})_2$-$\mathrm{SiH_2}$ with $[(t\mathrm{Bu})_2\mathrm{Hg}]$ followed by lithiation in THF. Irradiation of **1** produces $[\mathrm{Li}(i\mathrm{Pr_3Si})_2\mathrm{Si}]^\bullet$ (**2**), the first silyl radical with an α-Si-Li bond detected by EPR spectroscopy (see spectrum).



Angew. Chem. **2002**, *114*, 671–673

D. Bravo-Zhivotovskii,* M. Yuzefovich, N. Sigal, G. Korogodsky, K. Klinkhammer, B. Tumanskii, A. Shames, Y. Apeloig* 649–651

The Synthesis of the First Compound with Li-Si-Hg Bonding: $[\{\mathrm{Li}(i\mathrm{Pr_3Si})_2\mathrm{Si}\}_2\mathrm{Hg}]$—a Source for the $[\mathrm{Li}(i\mathrm{Pr_3Si})_2\mathrm{Si}]^\bullet$ Radical

Keywords: EPR spectroscopy · lithium · mercury · radicals · silicon

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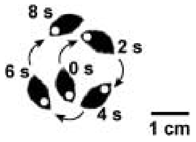
The artificial millimeter-scale "autonomous movers" glide across the surface of a liquid without an external power source. This system is based on a combination of two processes: Motion of individual objects powered by the catalytic decomposition of hydrogen peroxide, and relative motion (self-assembly) caused by capillary interactions at the fluid/air interface. The picture shows the rotational/translational motion of a single object; the motion of a pair of these object depends on their chirality.



Angew. Chem. **2002**, *114*, 674–676

R. F. Ismagilov, A. Schwartz, N. Bowden, G. M. Whitesides* 652–654

Autonomous Movement and Self-Assembly

Keywords: autonomous movement · complexity · energy-dissipating systems · self-assembly

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Supporting information on the WWW (see article for access details).

Accelerated publications

* Author to whom correspondence should be addressed

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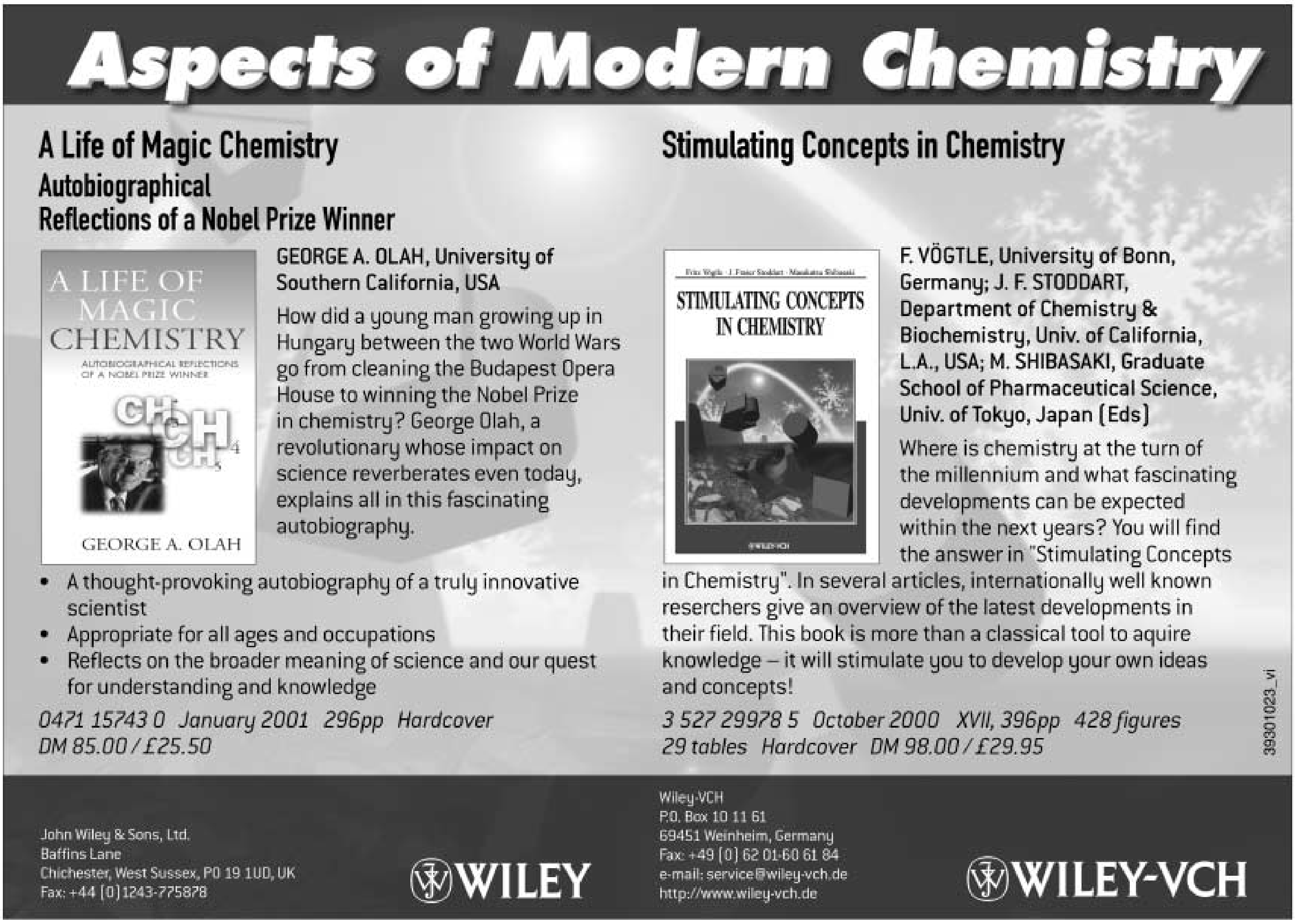

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