

## Cover

$\beta$ -Keto esters can be converted into alkynes by the nitrosative cleavage of the corresponding isoxazolin-5-ones. The story behind this reaction and other new syntheses of alkynes is presented.



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## contents

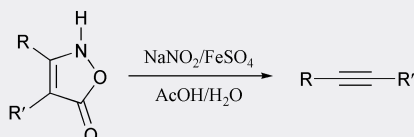
## FEATURE ARTICLE

1555

## New syntheses of alkynes: a tale of serendipity and design

Samir Z. Zard

The story behind the development of several new syntheses of alkynes is presented.

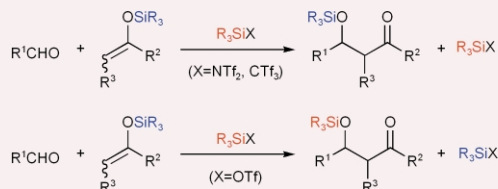


## COMMUNICATIONS

1564

## Crucial role of the ligand of silyl Lewis acid in the Mukaiyama aldol reaction

Kazuaki Ishihara, Yukihiro Hiraiwa and Hisashi Yamamoto\*

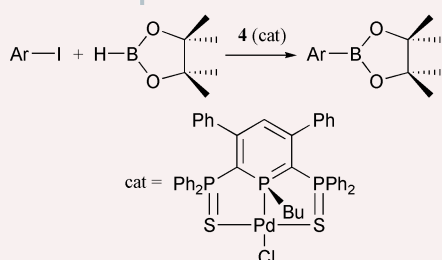


The  $\text{Me}_3\text{SiX}$ -induced Mukaiyama aldol reaction proceeds through each catalytic cycle under the influence of  $\text{X}^-$ : the silyl group of  $\text{Me}_3\text{SiNTf}_2$  does not release from  $\text{NTf}_2^-$  and that of silyl enol ether intermolecularly transfers to the product, while the silyl group of  $\text{Me}_3\text{SiOTf}$  remains in the product and that of the silyl enol ether becomes the catalyst for the next catalytic cycle.

1566

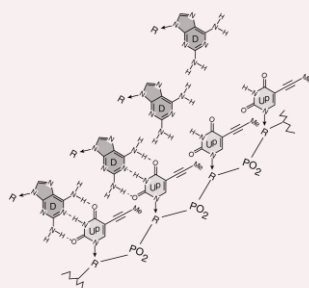
A  $\sigma^4$ ,  $\lambda^5$ -phosphinine palladium complex: a new type of phosphorus ligand and catalyst. Application to the Pd-catalyzed formation of arylboronic esters

Marjolaine Doux, Nicolas Mézailles, Mohand Melaimi, Louis Ricard and Pascal Le Floch\*



A palladium complex featuring a central pentacoordinated phosphorus atom and two pendant sulfide ligands efficiently catalyses the formation of C–B bonds from iodoarenes and pinacolborane.

1568



### 5-Propynyluracil-diaminopurine: an efficient base-pair for non-enzymatic transcription of DNA

John C. Chaput, Surajit Sinha and Christopher Switzer\*

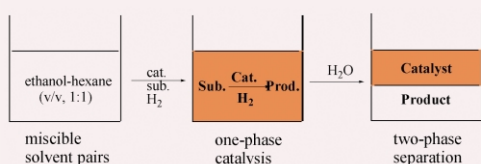
The Up·D base-pair (5-propynyl uracil-diaminopurine) is found to be more effective at non-enzymatic transcription than the corresponding natural T·A pair.

1570



### A novel system consisting of easily recyclable dendritic Ru-BINAP catalyst for asymmetric hydrogenation

Guo-Jun Deng, Qing-Hua Fan,\* Xiao-Min Chen, Dong-Sheng Liu and Albert S. C. Chan\*



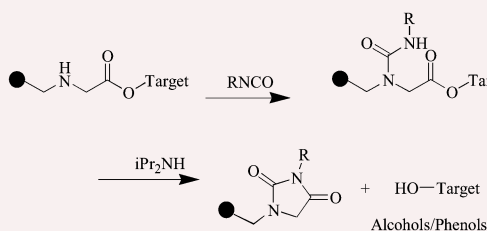
Ru(BINAP)-centered dendrimers with alkyl chains at the periphery together with organic binary solvent system have been employed for asymmetric hydrogenation, leading to high catalytic activity and enantioselectivity as well as facile catalyst recycling.

1572



### Target alcohol/phenol release by cyclative cleavage using glycine as a safety catch linker

Sadagopan Raghavan\* and A. Rajender



A new safety catch linker for alcohols/phenols that is stable under a variety of reaction conditions and cleaved by the cyclative mode under mild conditions is disclosed.

1574



### A facile structural rearrangement to unprecedented $\mu_2$ - $\eta^6$ : $\eta^6$ -bis(cyclopentadienylidene)ethene diruthenium complexes in the oxidation of 1,2-bis(ruthenocenyl)ethynes

Masaru Sato\* and Masanobu Watanabe



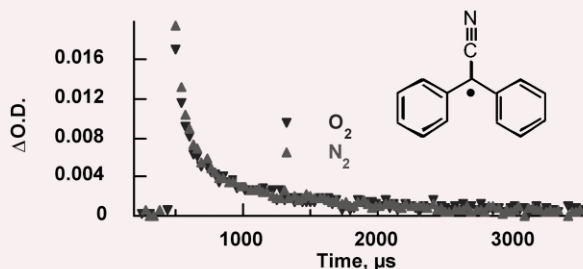
Bis(ruthenocenyl)ethynes were prepared by the alkyne metathesis of the corresponding propyne derivatives. Their two-electron oxidation gave bis(cyclopentadienylidene)ethene diruthenium complexes.

1576

### Greatly attenuated reactivity of nitrile-derived carbon-centered radicals toward oxygen

Enrique Font-Sanchis, Carolina Aliaga, K.-S. Focsaneanu and J. C. Scaiano\*

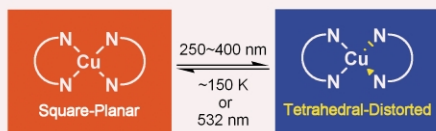
Laser photolysis studies show that several nitrile substituted radicals are unreactive towards molecular oxygen.



1578

**Unusually long-lived light-induced metastable state in a thermochromic copper(II) complex**

Kazuyuki Takahashi, Rie Nakajima, Zhong-ze Gu, Hajime Yoshiki, Akira Fujishima and Osamu Sato\*

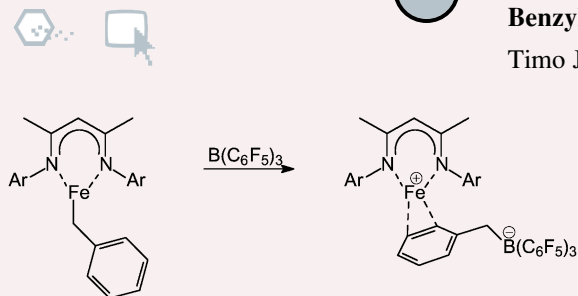


We report the first observation of a color change from red to purple for a thermochromic complex,  $[\text{Cu}(\text{dieten})_2](\text{BF}_4)_2$  (dieten = *N,N*-diethylethylenediamine), which is induced by illuminating with UV light.

1580

**Benzyl anion abstraction from a ( $\beta$ -diiminato)Fe(II) benzyl complex**

Timo J. J. Sciarone, Auke Meetsma, Bart Hesssen\* and Jan H. Teuben

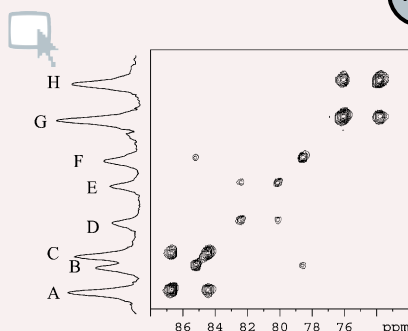


Benzyl abstraction from  $[(\text{nacnac})\text{Fe}(\text{CH}_2\text{Ph})]$  yields  $[(\text{nacnac})\text{Fe}][\text{PhCH}_2\text{B}(\text{C}_6\text{F}_5)_3]$ , which was characterised by X-ray diffraction; the Fe(II)-induced paramagnetic shifts in the  $^{19}\text{F}$  NMR spectrum of the coordinated anion enable study of cation–anion interactions in solution.

1582

**Two-dimensional and variable temperature  $^{31}\text{P}$  solid-state NMR studies of single crystals containing symmetrical/unsymmetrical bis[6-*O*,6-*O'*-(1,2:3,4-diisopropylidene- $\alpha$ -D-galactopyranosyl)thiophosphoryl] dichalcogenides**

M. J. Potrzebowski,\* J. Helinski and W. Ciesielski

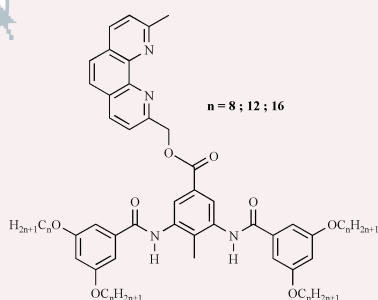


1D, 2D spin-exchange and variable temperature  $^{31}\text{P}$  NMR experiments were employed to establish the organisation and phase transition of single crystals containing three isostructural bis[6-*O*,6-*O'*-(1,2:3,4-diisopropylidene- $\alpha$ -D-galactopyranosyl)thiophosphoryl] dichalcogenide derivatives.

1584

**Mesomorphic phenanthroline derivatives: novel architectures based on hydrogen bonding**

Guillaume Pickaert, Laurent Douce, Raymond Ziessel\* and Daniel Guillon

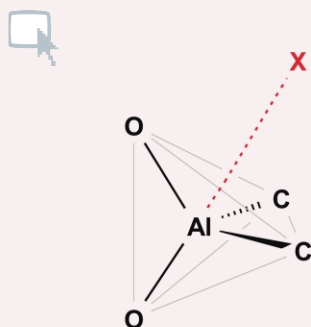


Columnar mesophases were provided by constructing phenanthroline templates grafted with diacylaminobenzene derivatives. Palladium coordination gives a smectic arrangement of the mesophase.

1586

**Directionality and borderline distance of secondary bonding on the fifth coordinate site in aluminium alkoxides**

Janusz Lewiński,\* Janusz Zachara\* and Iwona Justyniak

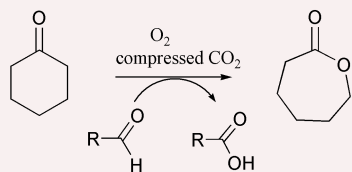


A survey of the Cambridge Structural Database for alkylaluminium alkoxides has been used to determine the borderline distances and angular distributions of secondary interactions on the fifth coordinate site, and map out the minimum-energy pathways in associative reactions.

1588

**Baeyer–Villiger oxidation in compressed CO<sub>2</sub>**

Carsten Bolm,\* Chiara Palazzi, Giancarlo Franciò and Walter Leitner\*

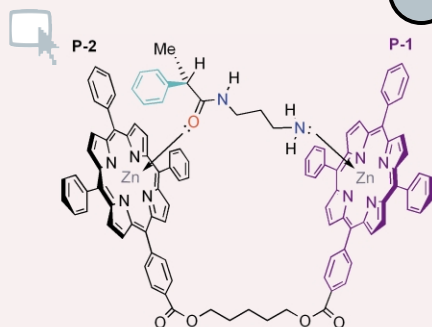


Highly efficient Bayer–Villiger oxidation of cyclic and acyclic substrates can be achieved in compressed carbon dioxide as reaction medium without the need for additional catalysts using oxygen as the primary oxidant and aldehydes as sacrificial co-reductant.

1590

**Configurational assignment of  $\alpha$ -chiral carboxylic acids by complexation to dimeric Zn–porphyrin: host–guest structure, chiral recognition and circular dichroism**

Gloria Proni, Gennaro Pescitelli, Xuefei Huang, Nazia Q. Quraishi, Koji Nakanishi\* and Nina Berova\*

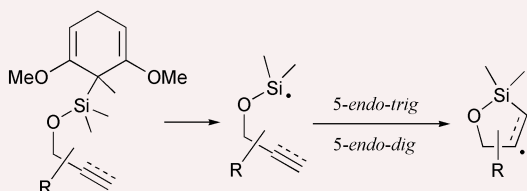


A circular dichroic exciton chirality method based on host–guest chiral recognition has been developed to determine the absolute configuration of carboxylic acids with an  $\alpha$ -stereogenic center; an amide C=O $\rightarrow$ Zn coordination is involved in this complexation.

1592

**Intramolecular radical hydrosilylation — the first radical 5-endo-dig cyclisation**

Stephan Amrein and Armido Studer\*

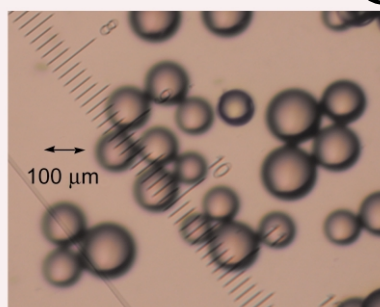


Intramolecular radical hydrosilylations using allyloxy- and propargyloxycyclohexadienylsilanes comprising 5-endo-trig reactions are discussed; furthermore the first example of a radical 5-endo-dig cyclisation is presented.

1594

**Sustainable photochemistry: solvent-free singlet oxygen-photooxygenation of organic substrates embedded in porphyrin-loaded polystyrene beads**

Axel G. Griesbeck\* and Anna Bartoschek

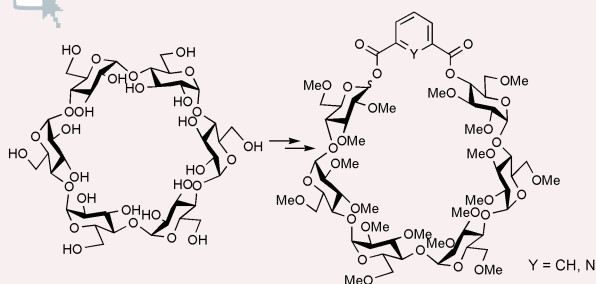


A solvent-free photooxygenation process that uses organic substrates embedded in porphyrin-loaded polystyrene beads (crosslinked with 1% of divinylbenzene) as solid support is described.

1596

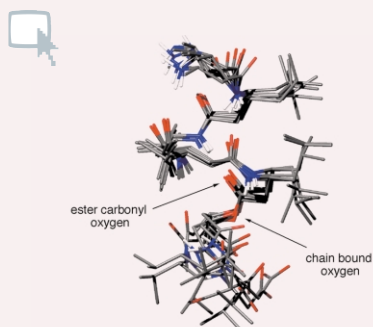
**A facile synthesis of novel types of cyclodextrin derivatives by insertion of an aromatic dicarbonyl spacer into a permethylated  $\alpha$ -cyclodextrin skeleton**

Toshiyuki Kida,\* Takao Michinobu, Wanbin Zhang, Yohji Nakatsuji and Isao Ikeda\*



Novel types of cyclodextrin derivatives were easily synthesized by the insertion of an aromatic dicarbonyl spacer into the skeleton of permethylated  $\alpha$ -cyclodextrin.

1598



### $\beta$ -Depsipeptides—the effect of a missing and a weakened hydrogen bond on the stability of the $\beta$ -peptidic $3_{14}$ -helix

Dieter Seebach,\* Yogesh R. Mahajan, Ramanathan Senthilkumar, Magnus Rueping and Bernhard Jaun\*

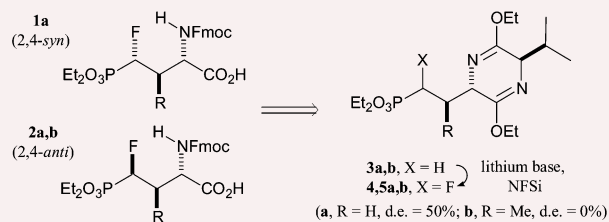
The importance of hydrogen bonding in  $\beta$ -peptide  $3_{14}$ -helices is demonstrated by an NMR analysis of three  $\beta$ -heptadepsipeptides containing a 3-hydroxybutanoic residue in different positions.

1600



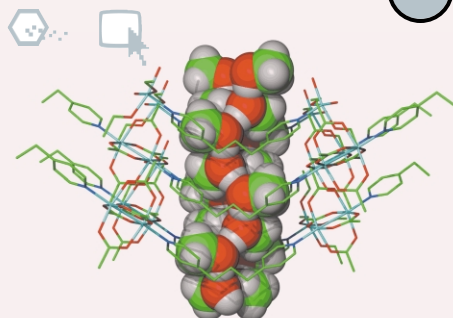
### Stereoselective synthesis of $\alpha$ -monofluorinated phosphonate mimetics of naturally occurring phosphoserine and phosphothreonine, via electrophilic fluorination of lithiated bis-lactim ethers

María Ruiz,\* Vicente Ojea,\* José M. Quintela and Juan J. Guillín



Electrophilic fluorinations of lithiated bis-lactim ethers allow a direct access to  $\alpha$ -monofluorinated phosphonate mimetics of phosphoserine and phosphothreonine in enantiomerically pure form and suitably protected for solid-phase peptide synthesis.

1602



### Channel-containing 1D coordination polymers based on a linear dimetallic spacer

Warwick J. Belcher, Christopher A. Longstaff, Marcus R. Neckenig and Jonathan W. Steed\*

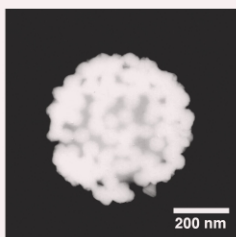
A predictable 1D coordination polymer based on a dimetallic copper(II) spacer forms a porous channel-containing architecture.

1604

### Spherical ensembles of gold nanoparticles on silica: electrostatic and size effects

Bryce Sadtler and Alexander Wei\*

Core-shell ensembles of citrate-stabilized gold nanoparticles (20–80 nm) on submicron silica cores (330–550 nm) have been prepared by electrostatic self-assembly with shell packing densities as high as  $\phi = 0.55$ .

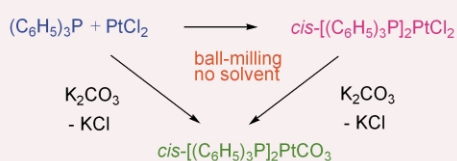


1606



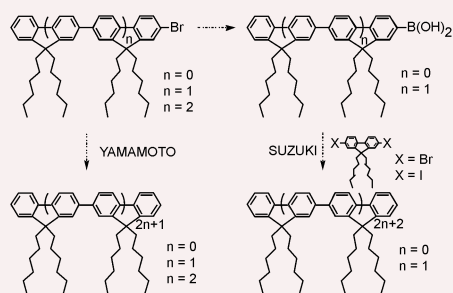
### Solvent-free mechanochemical synthesis of two Pt complexes: *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCl<sub>2</sub> and *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCO<sub>3</sub>

Viktor P. Balema,\* Jerzy W. Wiench, Marek Pruski and Vitalij K. Pecharsky\*



*Cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCl<sub>2</sub> and *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCO<sub>3</sub> complexes were prepared mechanochemically from solid reactants in the absence of a solvent; their formation during mechanical treatment was confirmed directly using solid-state <sup>31</sup>P MAS NMR spectroscopy, X-ray powder diffraction and differential thermal analysis.

1608



### Monodisperse fluorene oligomers exhibiting strong dipolar coupling interactions

Rémi Anémian, Jean-Christophe Mulatier, Chantal Andraud,\* Olivier Stéphan and Jean-Claude Vial

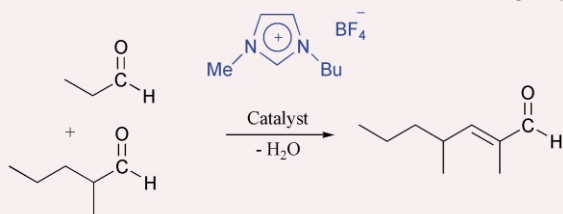
Well-defined fluorenes ( $n = 1$  to 6), in which physical properties are interpreted by large excitonic interactions, were synthesized.

1610

### Preparation of $C_9$ -aldehyde *via* aldol condensation reactions in ionic liquid media

Christian P. Mehnert,\* Nicholas C. Dispenziere and Raymond A. Cook

$C_9$ -aldehyde has been prepared *via* aldol condensation reactions in ionic liquid media.

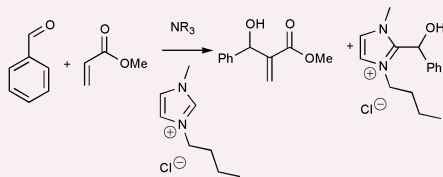


1612

### Unexpected side reactions of imidazolium-based ionic liquids in the base-catalysed Baylis–Hillman reaction

Varinder K. Aggarwal,\* Ingo Emme and Andrea Mereu

Low yields are obtained when the Baylis–Hillman reaction is conducted in the presence of an imidazolium-based ionic liquid due to direct addition of the deprotonated imidazolium salt to the aldehyde. Ionic liquids are evidently not inert.

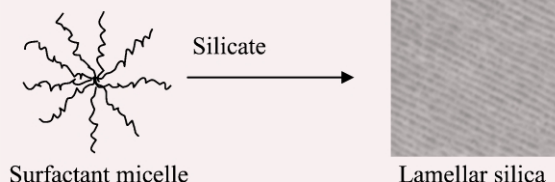


1614

### The preparation of a highly ordered long-range lamellar silica structure with large interlayer spacings

An-Wu Xu,\* Jimmy. C. Yu, Yue-Peng Cai, Hua-Xin Zhang and Li-Zhi Zhang

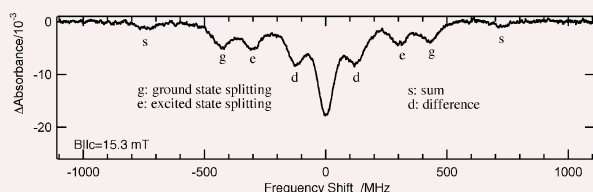
Highly ordered long-range lamellar silicas have been prepared using a novel silicone surfactant as a template. These lamellar silicas have the largest lattice constant to date reported. The coassembly of inorganic and organic species produces unusual lamellar silicas with an interlayer spacing of  $>100$  nm.



1616

### Multiple transient spectral holes in the $R_1$ -line of $NaMgAl(oxalate)_3 \cdot 9H_2O : Cr(III)$ . Probing excited and ground state $g$ -factors in an external magnetic field of 15 mT

Joseph L. Hughes and Hans Riesen\*

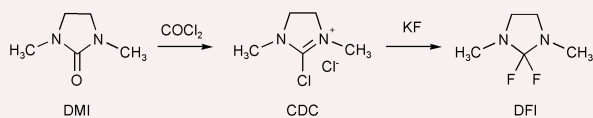


The  $g_z$ -factors of the  $^4A_2$  ground state and the  $^2E$  excited state of chromium(III) doped into  $NaMgAl(oxalate)_3 \cdot 9H_2O$  are accurately determined at 2.5 K by the observation of *multiple* transient spectral holes in the  $R_1$ -line ( $^2E \leftarrow ^4A_2$  transition) in *minute* magnetic fields of  $\approx 15$  mT.

1618

**2,2-Difluoro-1,3-dimethylimidazolidine (DFI). A new fluorinating agent**

Hidetoshi Hayashi,\* Hiroshi Sonoda, Kouki Fukumura and Teruyuki Nagata

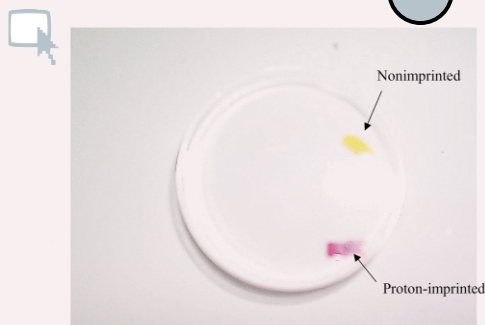


2,2-Difluoro-1,3-dimethylimidazolidine (DFI) is a new deoxy-fluorinating agent that is useful for the conversion of alcohols to monofluorides, and aldehydes/ketones to *gem*-difluorides under mild conditions.

1620

**Change of pH indicator's  $pK_a$  value via molecular imprinting**

Chengdu Liang, Michael J. Weaver and Sheng Dai\*

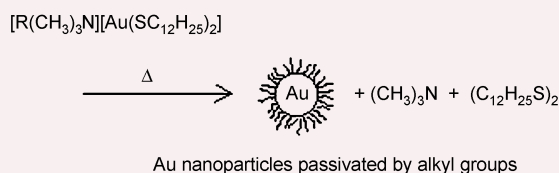


This communication describes a technique for the successful imprinting of protons (**the smallest chemical species**) in a hybrid sol-gel matrix; the  $pK_a$  value of the indicator is significantly changed up to several orders of magnitude by the imprinting synthesis.

1622

**Thermolysis of gold(I) thiolate complexes producing novel gold nanoparticles passivated by alkyl groups**

Masami Nakamoto,\* Mari Yamamoto and Masao Fukusumi

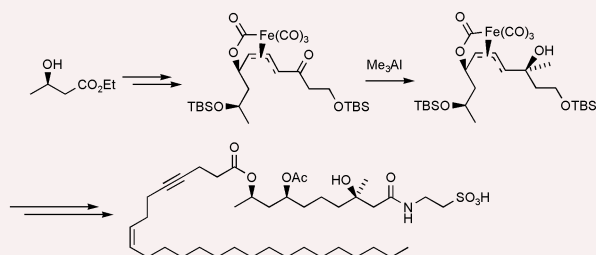


Thermolysis of gold(I) thiolate complexes,  $[\text{R}(\text{CH}_3)_3\text{N}][\text{Au}(\text{SC}_{12}\text{H}_{25})_2]$  ( $\text{R} = \text{C}_{14}\text{H}_{29}, \text{C}_{12}\text{H}_{25}$ ), produces novel gold nanoparticles passivated by alkyl groups, the average diameter of which is 26 nm.

1624

**Synthesis of taurospingin A: a potent inhibitor of DNA polymerase and HIV reverse transcriptase, using  $\pi$ -allyltricarbyliron lactone complexes**

Christopher J. Hollowood, Steven V. Ley\* and Shigeo Yamanoi

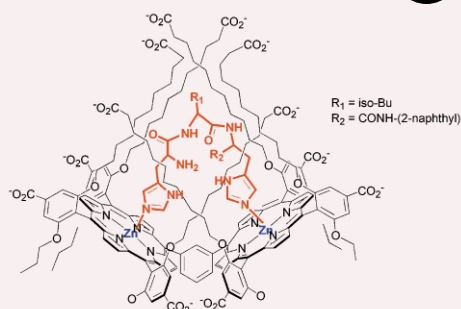


The synthesis of taurospingin A has been achieved using, as a key step, a  $\pi$ -allyltricarbyliron lactone complex to control a highly stereoselective addition of a methyl group to a carbonyl unit located in the side chain of the complex.

1626

**Hydrophobic environment of gable-type bisporphyrin receptors in water promotes binding of amines and oligopeptides**

Tadashi Mizutani,\* Kenji Wada and Susumu Kitagawa

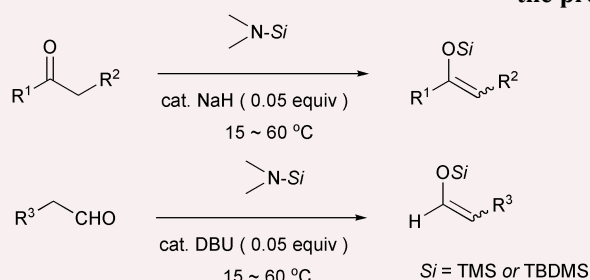


Amines and oligopeptides are bound to bisporphyrin receptors having the Lewis acidic site in a hydrophobic environment with a binding constant up to  $1 \times 10^6 \text{ M}^{-1}$  in water.

1628

**Silazanes/catalytic bases: mild, powerful and chemoselective agents for the preparation of enol silyl ethers from ketones and aldehydes**

Yoo Tanabe,\* Tomonori Misaki, Minoru Kurihara, Akira Iida and Yoshinori Nishii

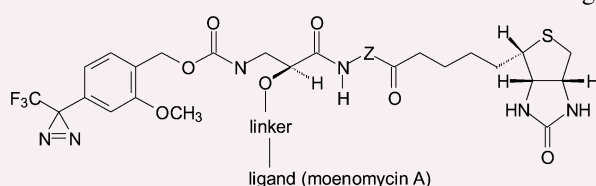


We have developed an efficient method for the preparation of enol silyl ethers using novel agents, silazanes together with NaH or DBU catalyst, wherein TMS and TBDMS groups were smoothly and chemoselectively introduced into ketones and aldehydes under mild conditions.

1630

**Isoerine-based biotinylated photoaffinity probes that interact with penicillin-binding protein 1b**

Thomas Rühl, Daniela Volke, Katherina Stembera, Yasumaru Hatanaka, Horst Hennig, Frank Schumer and Peter Welzel\*

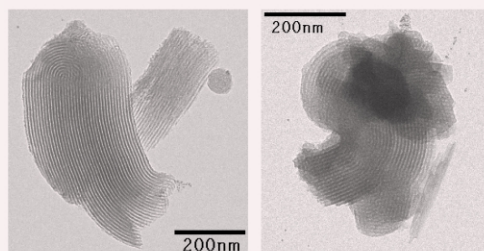


Photolytic decomposition of trifunctional carbene generating photoaffinity probes in methanolic solution.

1632

**A 3D-TEM study of the shape of mesopores in SBA-15 and modified SBA-15 materials**

Andries H. Janssen, Pascal Van Der Voort, Abraham J. Koster and Krijn P. de Jong\*

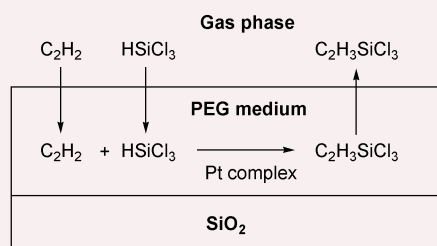


3D-TEM of SBA-15 type materials shows that the mesopores in these materials are highly curved on a mesoscopic length scale, which has a large influence on the diffusion path length through the particle.

1634

**A novel catalyst containing a platinum complex in polyethylene glycol medium supported on silica gel for vapor-phase hydrosilylation of acetylene with trichlorosilane or trimethoxysilane**

Masaki Okamoto,\* Hironari Kiya, Hiromi Yamashita and Eiichi Suzuki\*

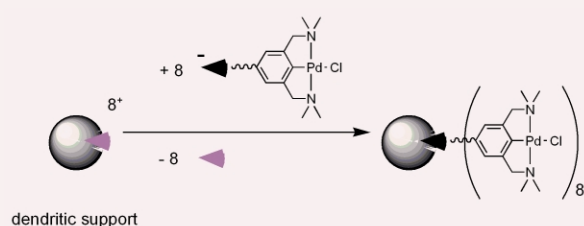


The title catalyst is very active for vapor-phase hydrosilylation of acetylene with trichlorosilane or trimethoxysilane, stable and readily prepared from easily available materials, tetraammineplatinum(II) chloride, polyethylene glycol and silica gel.

1636

**A polycationic dendrimer as noncovalent support for anionic organometallic complexes**

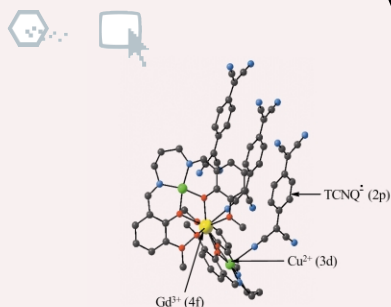
Rob van de Coevering, Mark Kuil, Robertus J. M. Klein Gebbink and Gerard van Koten\*



Polycationic dendrimer **1** forms a well-defined, stoichiometric assembly with eight anionic metal complexes; this assembly is successfully applied as a Lewis acidic catalyst.



1638

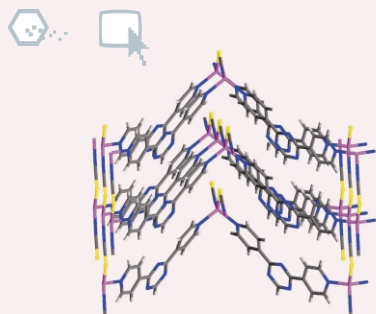


### The first coordination compound containing three different types of spin carriers: 2p–3d–4f (TCNQ<sup>•-</sup>, Cu<sup>2+</sup> and Gd<sup>3+</sup>)

Augustin M. Madalan, Herbert W. Roesky,\* Marius Andruh,\* Mathias Noltemeyer and Nicolae Stanica

The paper reports on a straightforward synthetic route towards heterospin systems by using cationic heteronuclear 3d–4f complexes as building blocks, and anionic radicals bearing the 2p unpaired electrons.

1640

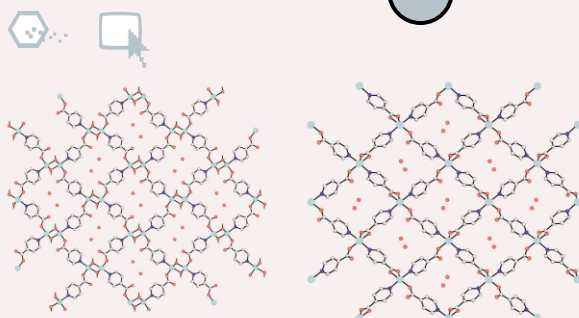


### Structural isomerism in CuSCN coordination polymers

Sarah A. Barnett, Alexander J. Blake, Neil R. Champness\* and Claire Wilson

CuSCN reacts with the angular ligand 2,4-bis(4-pyridyl)-1,3,5-triazine (dpt) to afford rare examples of coordination polymer structural isomers including a non-centrosymmetric three-dimensional framework with Cd(SO<sub>4</sub>) topology constructed from tetrahedral metal cations.

1642

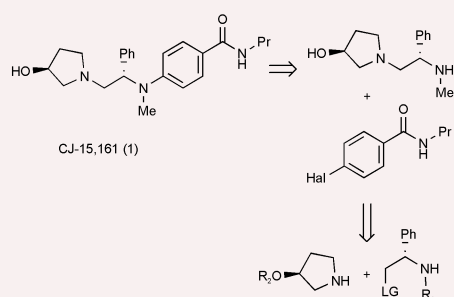


### Physical stability vs. chemical lability in microporous metal coordination polymers: a comparison of [Cu(OH)(INA)]<sub>n</sub> and [Cu(INA)<sub>2</sub>]<sub>n</sub>: INA = 1,4-(NC<sub>5</sub>H<sub>4</sub>CO<sub>2</sub>)

Candy Z-J. Lin, Stephen S-Y. Chui, Samuel M-F. Lo, Fanny L-Y. Shek, Mingmei Wu, Kinga Suwinska, Janusz Lipkowski and Ian D. Williams\*

Frameworks of [Cu(OH)(INA)] and [Cu(INA)<sub>2</sub>]: both exhibit thermal stabilities in excess of 200 °C, yet the latter is highly labile to chemical transformation.

1644

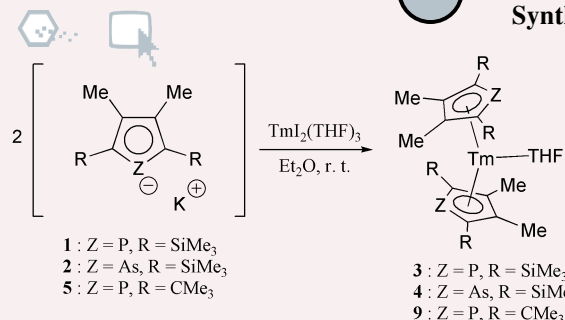


### Synthesis of the kappa-agonist CJ-15,161 via a palladium-catalyzed cross-coupling reaction

Arun Ghosh,\* Janice E. Sieser, Stéphane Caron and Timothy J. N. Watson

Syntheses of CJ-15,161 (**1**) involving intermolecular N-arylation of an appropriately functionalized diamine, obtained from the precursor  $\alpha$ -amino acids or, more conveniently, from the corresponding 1,2-amino alcohols *via* 1,2,3-oxathiazolidine-2,2-dioxide **22**, are reported.

1646



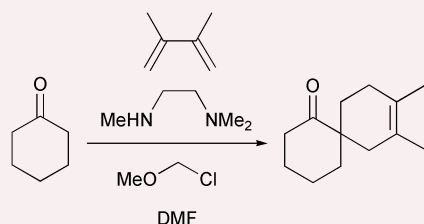
### Synthesis and structure of phospholyl- and arsolythulium(II) complexes

François Nief,\* Daniela Turcitu and Louis Ricard

The use of sterically crowded phospholyl and arsolyl ligands has permitted the preparation of new 'non-classical' organothulium(II) complexes with improved stability in solution at room temperature.

COMMUNICATIONS

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**$\alpha$ -Methylenation/Diels–Alder tandem reaction promoted by ammonium salts generated *in situ* from secondary–tertiary diamines and alkoxymethyl chlorides**

Hiroko Nakamura and Hisashi Yamamoto\*

A simple one-pot synthesis of spiranones from cycloketones and dienes promoted by an ammonium salt generated *in situ* from diamine and alkoxymethyl chloride through a tandem  $\alpha$ -methylenation/Diels–Alder reaction is described.

ADDITIONS AND CORRECTIONS

1650

K. N. Jayaprakash, Aaron M. Gillepsie, T. Brent Gunnoe and David P. White

**Synthesis of the dicationic Ru<sup>IV</sup> amido complex [TpRu(CO)(PPh<sub>3</sub>)(NHP)]<sub>2</sub>[OTf]<sub>2</sub> (Tp = hydridotris(pyrazolyl)borate; OTf = trifluoromethanesulfonate) and deprotonation to form an octahedral and d<sup>4</sup> imido complex: computational study of Ru<sup>IV</sup>–imido bonding**

1650

YooJin Kim and Duk-Young Jung

**Conformation change of the cyclohexanedicarboxylate ligand toward 2D and 3D La(III)-organic coordination networks**

1651

David S. Boyle, Kuveshni Govender and Paul O'Brien

**Novel low temperature solution deposition of perpendicularly oriented rods of ZnO: substrate effects and evidence of the importance of counterions in the control of crystallite growth**

1651

Xiaqin Wang, Kensuke Naka, Hideaki Itoh, Sooyun Park and Yoshiki Chujo

**Synthesis of silver dendritic nanostructures protected by tetrathiafulvalene**

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Dates, venues and contact details of forthcoming events.

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Supplementary crystallographic data are available: see article for further information.

Electronic supplementary information is available on <http://www.rsc.org/esi>: see article for further information.

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