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Cover

A crystal of magnetite (on an assembly of crystals of orthose) from Fiesch, Valais, Switzerland. Courtesy of Musée ENSMP. Photo copyright: J. M. Le Cléach, Musée de Minéralogie ENSMP, Paris, France (pp. 481–487).

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OCUS ARTICLE

Medicinal chemistry in academia: molecular recognition with biological receptors

tents

Fraser Hof and François Diederich*

Medicinal chemistry has traditionally been the realm of industry, but this rich field also provides ample opportunities for academic researchers. Academic medicinal chemistry investigations serve both as fundamental learning tools and as a complement to the endeavours of the private healthcare industry.

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Iron oxide chemistry. From molecular clusters to extended solid networks

Jean-Pierre Jolivet, Corinne Chanéac and Elisabeth Tronc

This overview features the chemical background on the formation of molecular clusters or nanosized solid phases in aqueous solution. Ferrous, ferric and mixed valent compounds are considered showing the versatility of the iron chemistry in solution.



COMMUNICATIONS

A monomolecularly imprinted dendrimer (MID) capable of selective binding with a tris(2-aminoethyl)amine guest through multiple functional group interactions

James B. Beil and Steven C. Zimmerman*

A molecularly imprinted dendrimer (MID) with a colorimetric reporter group exhibits three-point binding of tris(aminoethyl)amine in THF with a $K_{\text{assoc}} = 3.3 \times 10^6 \text{ M}^{-1}$.

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Chemical Science

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Self-assembly of heteroleptic $[2 \times 2]$ and $[2 \times 3]$ nanogrids

Michael Schmittel,* Venkateshwarlu Kalsani, Dieter Fenske and Andreas

Using the HETPHEN concept a general approach to heteroleptic nanogrids is illustrated. Mechanistic investigations propose a three-step pathway.

A host-guest complex between a metal-organic cyclotriveratrylene analog and a polyoxometalate: [Cu₆(4,7-phenanthroline)₈(MeCN)₄]2PM₁₂O₄₀

Jacqueline M. Knaust, Chad Inman and Steven W. Keller*

A novel Cu(I)-molecular hexamer is described in which the metal cations and phenanthroline molecules self-assemble into a dimer of shallow triangular-shaped bowls, within which are located large spherical polyoxometalate anions $PM_{12}O_{40}^{3-}$

Synthesis and X-ray single crystal structure of a bivalent glycocluster

Manuela Tosin, Helge Müller-Bunz and Paul V. Murphy*

The X-ray single crystal structure of a bivalent glycocluster shows that amide alkylation alters carbohydrate presentation and facilitates non covalent interactions

A ratiometric fluorescent probe for imaging hydroxyl radicals in living

Nobuaki Soh, Koji Makihara, Emino Sakoda and Toshihiko

A novel fluorescent probe, the detection mechanism of which is based on the 'on-off' switching of a FRET triggered by the 'OHinduced cleavage of a DNA strand, has been developed for the

The influence of solvation on short strong hydrogen bonds: a density functional theory study of the Asp-His interaction in

A high-level DFT study reveals that a structural water molecule favors a short strong hydrogen bond in the catalytic triad of subtilisins in sharp contrast to some current beliefs.

500 Confined organization of Au nanocrystals in glycolipid nanotube hollow cylinders Bo Yang, Shoko Kamiya, Kaname Yoshida and Toshimi Shimizu* 50 nm Mild fabrication of anisotropic metal-lipid nanotube (LNT)

nanocomposites, in which Au nanocrystals of 3-10 nm wide are organized in a glycolipid nanotube hollow cylinder, has been achieved by filling the internal channel of the LNT with HAuCl₄ aqueous solution by capillary force and subsequent photochemical reduction of $[AuCl_4]^-$.

Self-indicating amine scavenger resins

Jin Ku Cho, Peter D. White, Wolfgang Klute, Tony W. Dean and Mark Bradley*

> Self-indicating methylisocyanate resin, which functions as both a scavenger and an indicator for amines, was used for in situ reaction monitoring and purification of a urea based library.

From metal to ligand electroactivity in nickel(II) oxamato complexes

Xavier Ottenwaelder, Rafael Ruiz-García, Geneviève Blondin, Rosa Carasco, Joan Cano, Doris Lexa, Yves Journaux and Ally Aukauloo*

The locus of oxidation in square-planar nickel(II) oxamato complexes can be continuously shifted from the metal to the ligand by an appropriate choice of electron-donating substituents on the aromatic moiety of the ligand.

electron-poor ligand

Influence of substrate on self-assembled photonic crystal

Sachiko I. Matsushita and Masatsugu Shimomura*

Freestanding monolayers of self-assembled photonic crystals were prepared. The spectrum of the freestanding monolayer has less noise than that of the monolayer on the substrate, and besides, agrees with the theoretical result particularly in the near-field discussion.

Highly substituted pyridines via tethered imine-enamine (TIE) methodology

Steven A. Raw* and Richard J. K. Taylor*

Tethered imine–enamine methodology has been developed for the direct conversion of 1,2,4-triazines into highly substituted pyridines via the inverse electron demand Diels-Alder reaction which avoids the need for a discrete aromatisation step.

Toluene, reflux, 21 h,

4A mol. sieves

508

5 mm





NHR₁R₁' +

electron-rich ligand Ni^{II}-L



502



v





$V^{III}(OH){O_2C-C_6H_4-CO_2}.(HO_2C-C_6H_4-CO_2H)_x(DMF)_y(H_2O)_z$ (or MIL-68), a new vanadocarboxylate with a large pore hybrid topology : reticular synthesis with infinite inorganic building blocks?

K. Barthelet, J. Marrot, G. Férey and D. Riou*

MIL-68 is a vanado(III)terephthalate compound whose porous framework is described as an 'augmented' hexagonal bronze structure. This allows the extension of the reticular synthesis concept to edifices built from infinite inorganic building blocks.

Electrospray mass spectrometry of undiluted ionic liquids

Glen P. Jackson and Douglas C. Duckworth*

An *undiluted* ionic liquid Taylor cone forms during an electrospray mass spectrometric (ESMS) analysis. ESMS of neat ionic liquids provides a new approach for analyzing reactive ionic liquids or dissolved catalysts.

Surface energy and surface area measurements by ¹⁹F MAS NMR of adsorbed trifluoroacetic acid

Vitaliy L. Budarin, James H. Clark and Stewart J. Tavener*

Trifluoroacetic acid, when adsorbed on the surface of inorganic materials, is a useful ¹⁹F NMR probe molecule for studying surface properties including surface energy and surface area.

Carbon nanotube conducting arrays by consecutive amidation reactions

Dae-Hwan Jung, Myung Sup Jung, Young Koan Ko, Seung Joo Seo and Hee-Tae Jung*

Carbon nanotube conducting arrays with high-density multilayers were constructed *via* consecutive amidation reactions with the aid of a linker molecule and a condensation agent on a patterned amine-terminated glass substrate.

Preparation of highly accessible mordenite coatings on ceramic monoliths at loadings exceeding 50% by weight

(001) 12MR channel 7.0 x 6.5 Å 8 MR channel 5.7 x 2.6 Å 7 (010) 8 MR channel 9 4 8 x 3.4 Å

528

M. A. Ulla, E. Miro, R. Mallada, J. Coronas and J. Santamaría*

Mordenite coatings with a high accessibility have been synthesised on cordierite monolith supports at loadings exceeding 50% by weight.





524









iх



New magnetically responsive polydicarbazole-magnetite nanoparticles

Jean-Paul Lellouche,* Nurit Perlman, Augustine Joseph, Senthil Govindaraji, Ludmila Buzhansky, Aline Yakir and Ian Bruce

Magnetically responsive COOH-polydicarbazole-magnetite nanocomposites have been prepared by chemical oxidation of COOH-dicarbazole monomers in the presence of magnetite nanoparticles. Resulting nanoparticles have been tested for DNA hybridization experiments.



564

Ar-R

Mesocellular polymer foams with unprecedented uniform large mesopores and high surface areas

Jinwoo Lee, Jaeyun Kim, Sang-Wook Kim, Chae-Ho Shin* and Taeghwan Hyeon*

Mesocellular polymer foams with uniform ~17 nm cellular pores were fabricated using mesocellular silica foams as inorganic templates. The mesocellular polymer foams have high surface areas up to ~600 m^2g^{-1} and pore volumes of 1.6 cm^3g^{-1}

Microwave-assisted Negishi and Kumada cross-coupling reactions of aryl chlorides

Ar-Cl + XM R M = Zn, Mg X = Cl, Br, l R = aryl, alkyl $Pd_2(dba)_3/Bu_3P.HBF_4$ 0.015 - 2.5 mol% MW, THF/NMP, 175°C, 10 min Peter Walla and C. Oliver Kappe*

Fast and efficient microwave protocols for C–C coupling reactions of the Negishi and Kumada type involving aryl chlorides are described. The application to solid-phase synthesis is also reported.



$\rm C_1$ Coupling via bromine activation and tandem catalytic condensation and neutralization over CaO/zeolite composites

Ivan Lorkovic,* Maria Noy, Mike Weiss, Jeff Sherman, Eric McFarland, Galen D. Stucky and Peter C. Ford

Methane is partially oxidized by O_2 to olefins using Br_2 as a mediator for methane activation. Intermediate CH_xBr_{4-x} are condensed to higher hydrocarbons and HBr is neutralized over a regenerable CaO/ZSM-5 composite. Br_2 is released upon treating the spent composite with O_2 .



Spontaneous template-free assembly of ordered macroporous titania

Andrew Collins, Daniel Carriazo, Sean A. Davis* and Stephen Mann

Titania powders and fibres with ordered macroporosity have been produced by the facile reaction of titanium alkoxides with aqueous ammonia solution.

х

35 nm

(SiO₂)_{1-x}(LSiO_{1.5})_x L= organo group, x=0.05-0.50

576

578



Brij 56,

acetic acid

LSiY₃

sodium

silicate

Synthesis and structural characterisation of a novel phosphineborane-stabilised dicarbanion and an unusual bis(phosphineborane)

Keith Izod,* William McFarlane, Brent V. Tyson, William Clegg and Ross W. Harrington

The X-ray crystal structure of the phosphine-borane-stabilised dicarbanion **2b** reveals that the lithium is bound by the BH_3 hydrogens rather than the carbanion centre.

A versatile pathway for the direct assembly of organofunctional mesostructures from sodium silicate

Jainisha Shah, Seong-Su Kim and Thomas J. Pinnavaia

Silica mesostructures with well-expressed hexagonal and wormhole framework structures and up to 50% organo-functionalization of the framework silicon sites have been directly assembled from sodium silicate.

Protein–DNA interaction: impedance study of MutS binding to a DNA mismatch

Chen-Zhong Li, Yi-Tao Long, Jeremy S. Lee* and Heinz-Bernhard Kraatz*

Electrochemical impedance spectroscopy is a highly sensitive tool for the detection of single base-pair mismatches in double stranded DNA by monitoring the binding of MutS to a diluted DNA film.



Alexander Kulak, Simon R. Hall and Stephen Mann*

Drug-loaded porous silica or titania microspheres with complex morphologies and storage/release properties can be prepared by sonication of nanoparticle suspensions confined within aqueous droplets of drug solutions dispersed in toluene.

Electrochromic tetrathiafulvalene derivatives functionalised with 2,5diaryl-1,3,4-oxadiazole chromophores

Changsheng Wang, Andrei S. Batsanov and Martin R. Bryce*

Electrochromic switching of 2,5-diaryl-1,3,4-oxadiazole extended tetrathiafulvalene derivatives, between orange (neutral) and dark-green (oxidised) states, has been demonstrated.

хi







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COMMUNICATIONS

Modelling catalytic turnover frequencies in ionic liquids: the determination of the bimolecular rate constant for solvent displacement from $[(C_6H_6)Cr(CO)_2Solv]$

in 1-n-butyl-3-methylimidazolium hexafluorophosphate

Konrad Swiderski, Andrew McLean,* Charles M. Gordon* and D. H. Vaughan

The bimolecular rate constant for solvent displacement from $[(C_6H_6)Cr(CO)_2Solv]$ by an incoming ligand has been determined in the room temperature ionic liquid, [bmim][PF₆], and is compared to that in cyclohexane and dichoroethane.

Identification of 5-fluoro-5-deoxy-D-ribose-1-phosphate as an intermediate in fluorometabolite biosynthesis in *Streptomyces cattleya*

Steven L. Cobb, Hai Deng, John T. G. Hamilton, Ryan P. McGlinchey and David O'Hagan*

5'-Fluoro-5'-deoxy-D-ribose-1-phosphate (FDRP) has been identified as a biochemical intermediate during fluorometabolite biosynthesis in *Streptomyces cattleya*.

The first general method for α -trifluoromethylation of carboxylic acids using BrF₃

Aviv Hagooly and Shlomo Rozen*

2-Carbomethoxy-1,1-bis(methylsulfide)-1-alkenes, treated with BrF_3 , produce the desired α -trifluoromethyl carboxylate derivatives – $RCH(CF_3)COOR'$ – in good yields.

used for labelling the $\rm CF_3$ moiety with the short living [18]F isotope for PET purposes.





The first fullerene-heterofullerene dyad

Frank Hauke, M. Ángeles Herranz, Luis Echegoyen,* Dirk Guldi,* Andreas Hirsch* and Stefan Atalick

In this paper we present the first example of a fullerene–heterofullerene dyad, synthesized *via* a highly regioselective addition reaction. This molecule represents a very interesting functional architecture that allows for a unidirectional transduction of energy.

Well-defined self-assembling supramolecular structures in water containing a small amount of C_{60}



602

604

606

ŌН

ŌΗ

97-99%ee (R,R) 97->99%ee (R,S)

up to 4:1 dr (*syn:anti*)

Jingcheng Hao,* Hongguang Li, Weimin Liu and Andreas Hirsch*

The well-defined spherical bilayer vesicles of the dendritic C_{60} amphiphile–surfactant hybrids were prepared in aqueous solutions containing a small amount of C_{60} .

Poly(ethylene glycol) as solvent for transition metal mediated living radical polymerisation

Sébastien Perrier,* Hesna Gemici and Song Li

The use of low molecular weight poly(ethylene glycol) (PEG) as novel solvent for the copper mediated living radical polymerisation of methyl methacrylate and styrene leads to well-defined polymers with low residual copper content.

1.2 eq

MMA

Styrene



Guofu Zhong*

Enantiopure mono-substituted 1,2-diols were easily made by a rapid one-pot aminoxylation–allylation reaction. The discovery of *in situ* allylation extends the utility and importance far beyond the initial discovery of the asymmetric α -aminoxylation.



Copper mediated

nitrosobenzene (1.0 eq)

roline (20 mol%)/DMSŐ 10-20 min

then, allyl bromide (1.5 eq) In (1.5 eq), Nal (1.5 eq), rt 5 min

65 - 82% yield

Living Radical Polymerisation

An efficient approach for aromatic epoxidation using hydrogen peroxide and $Mn({\rm III})$ porphyrins

Susana L. H. Rebelo, Mário M. Q. Simões, M. Graça P. M. S. Neves, Artur M. S. Silva and José A. S. Cavaleiro*

In the presence of Mn(III) porphyrins and H_2O_2 , naphthalene and anthracene afford the *anti*-1,2:3,4-arene dioxides whereas phenanthrene gives rise to the 9,10-oxide, all in very high conversions and selectivities.

$\begin{array}{c} 610 \\ \hline \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ $	y of phosphates to zinc(II) complexes can be increased with bond donors Juan C. Mareque-Rivas,* Rafael Torres Martín de Rosales and Simon Parsons Non-coordinating amino H-bond donors adjacent to a zinc(II)-centre enhance the affinity of phosphates to the zinc(II) centre
612 Products scCO ₂ CO _{H2} scCO ₂ (ionic) catalyst product mixture R OHC	Continuous flow homogeneous catalysis using supercritical fluids Paul B. Webb and David J. Cole-Hamilton* 1-Octene is hydroformylated in a continuous flow process by retaining the catalyst within a reactor dissolved in the steady state mixture of reactants and products and transporting the substrates and products into and out of the reactor dissolved in scCO ₂ .
A novel scandium fluoride, [C ₂ N ₂ H ₁₀] _{0.5} [ScF ₄], with an unprecedented tungsten bronze-related layer structure. Nicholas F. Stephens, Alexandra M. Z. Slawin and Philip Lightfoot* [C ₂ N ₂ H ₁₀] _{0.5} [ScF ₄] exhibits a novel structure that consists of single octahedrally connected scandium fluoride layers and has features similar to those of the tungsten bronzes and layered perovskites. It may represent the first in a family of new layered structure types based on these general structural and compositional features.	
616 Bi be be biotin-X-NHS Biotin-X-NHS Biotin-X-NHS Biotin-X-NHS HHC H H H H H H H H H H H H H H H H H	otinylated and enzyme-immobilized carrier prepared by tero-bifunctional latex beads ong-Zhong Du, Takenori Tomohiro, Gao Zhang, Kazuhiko ikamura and Masato Kodaka* otinylated and pyruvate kinase immobilized nano-bio element have en prepared using hetero-bifunctional latex beads, where the enzyme ivity is roughly half of the free enzyme.
$R = (CH_2)_nOH \xrightarrow{R^1-B(OH)_2 (2)} \xrightarrow{R^1}_{R} \xrightarrow{(CH_2)_nOH} \xrightarrow{R^1}_{R} \xrightarrow{(CH_2)_nOH} \xrightarrow{R^1}_{A} \xrightarrow{(CH_2)_nO} \xrightarrow{R^1}_{A} \xrightarrow{(CH_2)_nOH} \xrightarrow{R^1}_{A} \xrightarrow{(CH_2)_nO} \xrightarrow{(CH_2)_{A} \xrightarrow{(CH_2)_nO} \xrightarrow{R^1}_{A} \xrightarrow{(CH_2)_{A} \xrightarrow{CH_2}_{A} (CH_$	ioselectivity of Pd-catalyzed additions of organoboronic acids netrical alkynes Nakjoon Kim, Ki Seong Kim, Aruna Kumar Gupta and Chang Ho Oh* The Pd-catalyzed reaction of unsymmetrical alkynes 1 with organoboronic acids 2 gave a mixture of products 3 and 4, whose ratios were controlled by the electronic as well as steric effects of the substrates 1.



Cubane-like structure of a silanethiol – primary amine assembly – a novel, unusual hydrogen bond pattern

Barbara Becker,* Katarzyna Baranowska, Jarosław Chojnacki and Wiesław Wojnowski

Four molecules of a silanethiol RSH and four molecules of a primary amine $R'NH_2$ combine giving a 'cubane-like' supermolecule, where a somewhat idealized picture shows how its twelve $N-H\cdots S$ hydrogen bonds form a novel, previously unknown pattern.

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