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293

297

NH

Peptide

Polyketide



Cover The cover illustrates enzymatic macrocyclization in natural product biosynthesis. It shows schematically how the thioesterase (TE) domain can constrain natural product structures into their bioactive conformations.

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FOCUS ARTICLE

Enantioselective catalysis in fine chemicals production

Hans-Ulrich Blaser



The application of enantioselective catalysis to the fine chemicals industry has great potential both from economic and ecological points of view, but to date has not been widely implemented on a technical scale. The author hopes that the award of the 2001 Chemistry Nobel Prize in this field will give the necessary impetus to future applications.

EATURE ARTICLI

Enzymology of acyl chain macrocyclization in natural product biosynthesis

Rahul M. Kohli and Christopher T. Walsh

A myriad of natural products are brought to their bioactive macrocyclic conformations by versatile enzymatic catalysts.

COMMUNICATIONS

Anchor chain length alters the apparent mechanism of chloride channel function in SCMTR derivatives

Paul H. Schlesinger,* Natasha K. Djedovič, Riccardo Ferdani, Jolanta Pajewska, Robert Pajewski and George W. Gokel*

Two membrane-anchored heptapeptides have been prepared and their poreformation behavior in phospholipid bilayer membranes has been found to differ profoundly as a result only of alkyl chain length.



Peptide-NH₂

Polyketide-OH



312

Low molecular weight organogelators for water

Guijun Wang and Andrew D. Hamilton*



A new class of mono-urea serine derivatives with low molecular weights was prepared in one synthetic step and certain members show remarkable selfassembling and gelation properties in water.

Helical templating of polyiodide networks at a binuclear metallo complex

Caitlin J. Horn, Alexander J. Blake, Neil R. Champness, Alessandra Garau, Vito Lippolis, Claire Wilson and Martin Schröder*

A homobimetallic supramolecular helicate has been used to template the formation of two polyiodide networks, one containing an unprecedented figure-of-eight polyiodide helix, the other dominated by peripheral C-H…I polyiodide chain interactions.





Synthesis and characterization of hyperbranched mesoporous silica SBA-15

Zoltán Kónya, Ji Zhu, Agnes Szegedi, Imre Kiricsi, Paul Alivisatos and Gabor A. Somorjai*

Branched mesoporous silica SBA-15 materials have been prepared in a simple process using non-ionic surfactant in acidic conditions in the presence of metal ions.

Binding of an inhibitor of the p53/MDM2 Interaction to MDM2

Sara J. Duncan, Matthew A. Cooper and Dudley H. Williams*



Efficient function of the protein p53 is important in the prevention of human cancer. Its action is down-regulated by interaction with the protein MDM2. The secondary metabolite chlorofusin inhibits the interaction between p53 and MDM2, specifically by binding to MDM2.



Indium nitride crystals with flower-like structure

Naoyuki Takahashi,* Arei Niwa, Haruka Sugiura and Takato Nakamura

Preparation of indium nitride at atmospheric pressure has been examined by means of halide CVD. Crystals deposited onto a Si(100) substrate showed flowerlike structure.

ii



320

= Cu(II)

= Na(l)

322

Synthesis, structure and magnetism of a unique dodecanuclearcopper(II) 'picture frame' held in a 4×4 grid-like assembly

Craig J. Matthews,* Stuart T. Onions, Gérald Morata, Margarita Bosch Salvia, Mark R. J. Elsegood and Daniel J. Price

The reaction of a flexible multidentate ligand with Cu^{II} leads to a unique self-assembled dodecanuclearcopper(II) 'picture frame', held in a 4 × 4 square grid-like array of organic ligands; it represents the largest structurally characterised Cu(II) assembly formed exclusively by a linear single stranded ligand.

New method for attachment of biomolecules to porous silicon

Bradley R. Hart, Sonia E. Létant, Staci R. Kane, Masood Z. Hadi, Sharon J. Shields and John G. Reynolds*



Biomolecules have been attached to porous silicon by a new linking method that forms a direct Si–C bond on the surface and retains the photoluminescence of the porous silicon.

The consequences of an interstitial N atom in the FeMo cofactor of nitrogenase

Ian Dance

The atom-centred FeMo cofactor of nitrogenase most likely contains N, resting at the $[NMoFe_7]^{18+}$ redox level, inserted from N₂, and subsequently restricting the modes of binding of substrate to the NFe₆ core.



CS₂

N-N = bpy

2

[м —он

N-N = phen

1

[M]—OH :

Pure silica BETA colloidal zeolite assembled in thin films

S. Mintova,* M. Reinelt, T. H. Metzger, J. Senker and T. Bein*

The synthesis of pure silica nanoscale zeolite BETA with monomodal particle size distribution is reported.

A new reactivity pattern of low-valent transition-metal hydroxo complexes: straightforward synthesis of hydrosulfido complexes *via* reaction with carbon disulfide

Darío C. Gerbino, Eva Hevia, Dolores Morales, M. Elena Navarro Clemente, Julio Pérez,* Lucía Riera, Víctor Riera and Daniel Miguel

A new basic transformation linking two important classes of transition metal compounds; namely, hydroxo and hydrosulfido complexes has been discovered.



328

[м]—зн

[M] = [Mo] 3

[Re] 4

iii







336

COMMUNICATIONS

Equilibrium potentials and charge transport of an I^-/I_3^- redox couple in an ionic liquid

Ryuji Kawano and Masayoshi Watanabe*

Equilibrium potentials and charge transport of an I^-/I_3^- redox couple in an ionic liquid are revealed by using a microelectrode technique, where the anomaly of the charge transport at high concentrations of the redox couple with comparable $[I^-]$ and $[I_3^-]$ can be attributed to the exchange reaction of $I^- + I_3^- \rightarrow I_3^- + I^-$.

Direct Zn-diamine promoted reduction of C=O and C=N bonds by polymethylhydrosiloxane in methanol

Virginie Bette, André Mortreux, Christian W. Lehmann and Jean-François Carpentier*

Ketones and imines are chemoselectively reduced in methanol to the corresponding alcohols and amines in a one-step procedure using polymethylhydrosiloxane (PMHS) and a simple zinc–diamine catalyst (19 examples, 50–99% yield).

Novel Cr-PNP complexes as catalysts for the trimerisation of ethylene

David S. McGuinness,* Peter Wasserscheid,* Wilhelm Keim, Chunhua Hu, Ulli Englert, John T. Dixon and Cronje Grove

Cr(III) complexes of tridentate PNP ligands have been prepared and evaluated as catalysts for ethylene trimerisation, with several giving high activity and excellent selectivity towards 1-hexene when activated with methylaluminoxane.

A rigid cavity containing tetra-cobalt(III) [2 \times 2] grid complex

Jeffrey P. Plante, Paul D. Jones, Douglas R. Powell and Timothy E. Glass*

The synthesis and structure of a rigid, cavity containing tetra-cobalt(III) $[2 \times 2]$ grid complex using an unusual bis(bipyridine)dimethoxynaphthyridine ligand is described.



A reagentless electrochemical biosensor based on a protein scaffold

Sulay D. Jhaveri, J. Matthew Mauro, Harold M. Goldston, Caroline L. Schauer, Leonard M. Tender* and Scott A. Trammell*

Apo-myoglobin, labeled with the environmentally sensitive redox probe $Ru^{II}(NH_3)_4(1,10$ -phenanthroline-5-maleimide)²⁺, was immobilized onto modified gold electrodes and subsequently labeled with biotin. The resulting protein scaffold transduced the molecular recognition of avidin into a measurable electrochemical signal.

iv



v

Enantioselective total synthesis of (-)-heliannuol A

Hidetoshi Kishuku, Mitsuru Shindo and Kozo Shishido*



350

352

356

358

An efficient and enantiocontrolled total synthesis of (–)-heliannuol A has been accomplished by employing ring closing metathesis and sequential diastereoselective epoxidation and regioselective reductive cleavage of the epoxide ring.

Growth of individual hydrogen-bonded nanostructures on gold monolayers



Juan J. Garcia-Lopez, Szczepan Zapotoczny, Peter Timmerman, Frank C. J. M. van Veggel, G. Julius Vancso, Mercedes Crego-Calama* and David N. Reinhoudt*

The growth of the nanostructures was achieved through an exchange reaction between single isolated species of calix[4]arene dimelamine $(1.1 \pm 0.2 \text{ nm})$ embedded in hexanethiol monolayers and double rosette hydrogen bonded assemblies in solution.

Highly selective Friedel-Crafts monoalkylation using micromixing

Seiji Suga, Aiichiro Nagaki and Jun-icih Yoshida*

Highly selective Friedel–Crafts monoalkylation of aromatic compounds with *N*-acyliminium ions has been achieved by efficient 1:1 mixing using a multilamination-type micromixer.

Engineering redox functions in a nucleic acid binding protein

Jon R. Wilson, Daren J. Caruana and Gianfranco Gilardi*

Proteins with diverse functions are often found within the same superfamily. An interesting challenge for protein engineering is to enable the combination of functions for proteins of the same superfamily. Here we show how a RNA-binding protein can be engineered to bind haem, exhibiting the electrochemistry typical of cytochromes of the same superfamily.

 $(\eta^3-L)PtMe_2H^+ + C_6H_6 \xrightarrow{-CH_4} (\eta^3-L)PtMe(Ph)H^+ \longrightarrow LPt(Ph)(\sigma-CH_4)^+$



 $(\eta^3-L)PtH()^+ + 2PhH$

$N-Pt^{IV}-H/N-H\ldots Pt^{II}$ intramolecular redox equilibrium in a product of $H-C(sp^2)$ cleavage and unusual alkane/arene C-H bond selectivity of ([2.1.1]pyridinophane) $Pt^{II}(CH_3)^+$

Andrei N. Vedernikov* and Kenneth G. Caulton*

The stability of an olefin coordinated to Pt(II) makes possible the use of a phenyl complex of Pt(IV) for cyclopentane dehydrogenation to coordinated cyclopentene.

360

COMMUNICATIONS

Silver nanoparticle growth in 3D-hexagonal mesoporous silica films

Sophie Besson, Thierry Gacoin, Christian Ricolleau and Jean-Pierre Boilot*

The 3D-hexagonal mesoporous films are used as templates to grow uniform silver nanoparticles. The grafting of hydrophobic groups at the pore surface, significantly slows down the silver ion diffusion, anchoring small silver clusters in micropores and leading to organized domains of silver particles in mesopores with a narrow size distribution.

5nm

hv (355 nm)

pyrylium salts

362

364

366

368

MeC

 $\lambda = 380$ and 600 nm (LFP)

Sidewall functionalization of single-walled carbon nanotubes with organic peroxides

Haiqing Peng, Paul Reverdy, Valery N. Khabashesku* and John L. Margrave

The single-wall carbon nanotubes (SWNTs), covalently functionalized by a bulky long-chain group, *e.g.*, undecyl, shown here on a TEM image, are prepared by reactions of SWNT materials with organic peroxides.

Chemical and transient spectroscopic evidence for C₂–C₃ cleavage of 2,3diaryloxetane radical cations

Methoxy substitution at the 3-aryl group of 2,3diphenyloxetanes induces C_2-C_3 bond cleavage. The reaction mechanism is supported by detection of *trans*anethole radical cation as transient intermediate.

Understanding the building-up process of three dimensional openframework metal phosphates: Acid degradation of the 3D structures to lower dimensional structures

Amitava Choudhury and C. N. R. Rao*

A zinc phosphate with a three-dimensional channel structure is shown to transform to lower dimensional structures on treatment with acid.

Me

Highly proton conductive polyimide electrolytes containing fluorenyl groups

Kenji Miyatake, Hua Zhou, Hiroyuki Uchida and Masahiro Watanabe*



SPIH-x (x = 0 - 60)

Novel sulfonated polyimides have been synthesized as a potential electrolyte for polymer electrolyte fuel cells operationable at high temperature.



O-TMS

α

€

372

374

376

378

Synthesis and characterization of highly ordered mesoporous thin films with –COOH terminated pore surfaces

Nanguo Liu, Roger A. Assink and C. Jeffrey Brinker*

Highly ordered mesoporous thin films with negatively chargeable –COOH terminated surfaces, useful for mimicking biological ion channels, were synthesized by evaporation induced self-assembly.

Phosphazenium chloride catalysts immobilized on SBA-15 mesoporous material and silica gel: new exceptionally active catalysts for the chlorination of organic acids

Keun-Sik Kim, Jong-Ho Kim and Gon Seo*

Phosphazenium chloride catalysts immobilized on SBA-15 mesoporous material and silica gel show exceptional activities and selectivities even in the continuous chlorination reaction of organic acids with thionyl chloride or phosgene.

Secondary nucleation of the $\beta\text{-polymorph}$ of L-glutamic acid on the surface of $\alpha\text{-form}$ crystals

C. Cashell,* D. Corcoran and B. K. Hodnett

Secondary nucleation of β -L-glutamic acid at the surface of α -form crystals is observed for the first time by SEM and confirmed by Raman spectroscopy. Nucleation takes place at certain crystallographic faces, the absence of which prevents this transition. There is only one other report in the literature of formation of a stable polymorph of an organic compound on the surface of its metastable form.

'Nucleo-nanocages': designed ternary oligodeoxyribonucleotides spontaneously form nanosized DNA cages

Self-complementary sticky end Nucleo-nanocage K Hybridization Selforganization DNA-three-way junction CC

Kazunori Matsuura, Taro Yamashita, Yuuko Igami and Nobuo Kimizuka*

'Nucleo-nanocages' are spontaneously self-assembled from the suitably designed DNA three-way junctions that possess self-complementary sticky ends.

Gas phase oxidation of alcohols to aldehydes or ketones catalysed by supported gold

Serena Biella and Michele Rossi*



Gold supported on silica shows remarkable selectivity in the clean catalytic oxidation of aliphatic alcohols to carbonyl derivatives.



1 n=1 2 n=2 3 n=3

ix





Robert J. Baker and Cameron Jones*

Reaction of 'GaI' with a 1,3-diyne, Me₃SiC \equiv CC \equiv CSiMe₃, leads to C–C coupling reactions and the isolation of the novel organogallium species, [Ga₄I₈{C₈(SiMe₃)₄}], as two isomeric forms.

Molecular recognition. Electrostatic effects in supramolecular selfassembly

James D. Crowley, Andrew J. Goshe and B. Bosnich*



392

394

396

398

It is found that formation of 2:1 adducts between rigid, linear, di-site linkers and positively charged molecular cleft receptors is controlled by electrostatic repulsion which can overcome the binding energy.

Novel synthesis of highly active Pt/C cathode electrocatalyst for direct methanol fuel cell

Zhenhua Zhou, Suli Wang, Weijiang Zhou, Guoxiong Wang, Luhua Jiang, Wenzhen Li, Shuqin Song, Jianguo Liu, Gongquan Sun and Qin Xin*

A 40 wt% Pt/C cathode electrocatalyst with controlled Pt particle size of \sim 2.9 nm showing better performance than commercial catalyst for direct methanol fuel cell was prepared by a polyol process with water but without using stabilizing agent.

Direct preparation of polyfunctional amino-substituted arylmagnesium reagents *via* an iodine–magnesium exchange reaction



Greta Varchi, Christiane Kofink, David M. Lindsay, Alfredo Ricci and Paul Knochel*

The preparation of polyfunctional unprotected amino-substituted arylmagnesium reagents using a selective iodine–magnesium exchange and their reaction with a broad range of electrophiles is described.

Preparation, structure, and some coordination properties of 2-chloro-3,3diphenyl-3-thioxo-1-(2,4,6-tri-*t*-butylphenyl)- 1,3-diphosphapropene



 $Mes^* = 2,4,6-t-Bu_3C_6H_2$ cod = cycloocta-1,5-diene Shigekazu Ito, Hongze Liang and Masaaki Yoshifuji*

A kinetically stabilised compound with the P=C–P=S skeleton bearing the 2,4,6-*t*-butylphenyl group was prepared and its coordination properties were studied with carbonyltungsten(0) reagents or iodine.

х

50 nm



C₆₀, DBU, (I₂)

toluene

Х

-CI

-Br

-H/l₂

utensity 0.5

0.0 400

KHMDS

No reaction

Main Adduct

equatorial

trans-4

trans-4

800

406

500 600 700 Wavelength (nm) 400

402

OMe



Marcel A. Duin, Nicolas D. Clement, Kingsley J. Cavell* and Cornelis J. Elsevier*

Hydridoplatinum(II) bis(carbene) compounds have been synthesised for the first time from a Pt(0)(carbene) complex with two monoalkene ligands. Due to the strong donor capacity of the carbene ligand, the Pt(0) complex is able to intermolecularity activate C–H bonds at the 2-position of imidazolium salts at room temperature.

Unexpected switch in regioselectivity of tether-directed Bingel-type biscyclopropanations depending on the leaving groups at tethered active methylene moieties

Tetsuo Hino and Kazuhiko Saigo*

The reaction of C_{60} with unhalogenated tethered bis(active methylene) derivatives/I₂ and with brominated derivatives in the presence of DBU gave *trans*-4-adducts predominantly, while the reactions with chlorinated derivatives afforded *equatorial*-adducts almost exclusively.

The colourful fluorescence from readily-synthesised 3,4-diarylsubstituted maleimide fluorophores

Hsiu-Chih Yeh, Wei-Ching Wu and Chin-Ti Chen*

We have developed a one-step synthesis of 3,4-diaryl-substituted maleimides directly from commercially available acetonitrile derivatives. These maleimides exhibit a large variation of emission spectra spanning the entire visible range.

Potassium-zinc induced synergic enhancement of the basicity of hexamethyldisilazide (HMDS) towards methylbenzene molecules

William Clegg, Glenn C. Forbes, Alan R. Kennedy, Robert E. Mulvey* and Stephen T. Liddle

Following the old adage that two heads are better than one, when the base HMDS is supported by both K and Zn then it can readily metallate toluene to form a novel mixed-metal mixed amido-benzyl product, but in the company of either metal alone no such deprotonation occurs.



Hydrothermal synthesis of perovskite nanotubes

Yuanbing Mao, Sarbajit Banerjee and Stanislaus S. Wong*

Titania nanotubes as precursors in the hydrothermal synthesis of $BaTiO_3$ and $SrTiO_3$ nanotubes under ambient temperature and strong alkaline conditions.



Dioxygen activation by a dinuclear nickel thiolate complex: structural characterization of the ligand oxidized product

Todd C. Harrop, Marilyn M. Olmstead and Pradip K. Mascharak*

A dinuclear nickel(II) complex with carboxamido N and thiolato S donors reacts with oxygen to give the ligand oxidized product with Ni-bound phenolates.



Three-fold interpenetrating three-dimensional networks based on *C*-methylcalix[4]resorcinarene incorporating benzophenone guest molecules

Bao-Qing Ma and Philip Coppens*

Two benzophenone molecules are entrapped within the cavity left after the formation of three-fold interpenetrating hydrogen-bonded frameworks based on *C*-methylcalix[4]resorcinarene and the spacer bis(4-pyridylmethylidyne)hydrazine.

An electrogenerated poly(pyrrole-benzophenone) film for the photografting of proteins

Serge Cosnier* and Anne Senillou



A new electropolymerisable photoreactive pyrrole-benzophenone was synthesized and polymerized, the resulting polymer, upon irradiation, gives rise to protein attachment.



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Fluorophore-capped cyclodextrins as efficient chemical-to-light energy converters

De-Qi Yuan,* Naoya Kishikawa, Cheng Yang, Kazutaka Koga, Naotaka Kuroda and Kahee Fujita*

The conjugated binding site greatly contributes to the efficiency of the fluorophore cap in converting chemical energy to light.



Electrochemistry of $P450_{\text{cin}}$: new insights into P450 electron transfer

Kondo-François Aguey-Zinsou, Paul V. Bernhardt,* James J. De Voss and Kate E. Slessor

Electrochemistry of bacterial cytochrome $P450_{cin}$ (CYP176A) reveals that, unusually, substrate binding does not affect the heme redox potential, although a marked pH dependence is consistent with a coupled single electron/single proton transfer reaction in the range 6 < pH < 10.





The contrasting behaviour of bridged amido-cyclopentadienyl (constrained geometry) group 15 chlorides and cations derived therefrom

Robert J. Wiacek, Charles L. B. Macdonald, Jamie N. Jones, Jeffrey M. Pietryga and Alan H. Cowley*

The first amido-cyclopentadienyl (constrained geometry) phosphenium, arsenium and stibenium cations have been isolated as their tetrachloroaluminate salts.

A simple strategy for preparation of sensor arrays: molecularly structured monolayers as recognition elements



432

434

438

Thomas Hirsch, Hubert Kettenberger, Otto S. Wolfbeis and Vladimir M. Mirsky*

The spreader-bar approach is a simple method for producing a huge variety of receptors with different selectivities. A sensor-array consisting of five such receptors is presented. A pattern recognition provides selective detection of different purines and pyrimidines.

Acoustic wave sensor for barium based on poly[Ni(salen)(crown)] recognition chemistry

Magda Martins, Cristina Freire* and A. Robert Hillman*

Interfacial recognition of barium ions by a polymeric crown ether receptor is quantified using an acoustic wave sensor, and the isotherm rationalised on the basis of solution complexation chemistry and polymer viscoelastic properties.



1.0x10⁵ 1.5x10⁵ 2.0x10⁵

 $(c_{\text{Re}^{2^*}})^{-1}$ / mol⁻¹ dm³

Charge transfers influence on the spin ground state of manganese and iron superoxide dismutases: a DFT study on a model of the reduced active site interacting with O_2^-

Rosa Carrasco, Irène Morgenstern-Badarau and Joan Cano*

From DFT and time-dependent DFT calculations on Mn^{II}SOD and Fe^{II}SOD active site models interacting with O_2^- we have determined that metal-ligand charge transfers stabilise the S = 2 and S = 5/2 spin states as ground spin states for the $[Mn^{II}SOD-O_2^-]$ and $[Fe^{II}SOD-O_2^-]$ model complexes, respectively.

The redox thermodynamics of microperoxidase are dependent on the solvent medium

David O' Donoghue and Edmond Magner*

The redox thermodynamics of the heme undecapeptide, microperoxidase have been examined in aqueous buffer and in glycerol. The change in $E^{\circ\prime}$ on transition from water to glycerol is dominated by the change in $\Delta S^{\circ\prime}$.



1.4x10 പ 1.2x10

1.0x10 $\Delta m_{_{\rm B}}$

8 0x10 6.0x10

 5.0×10^{4}

dsorbec



$Rh_2(OAc)_4$ -catalyzed reactions of α -diazoimides: a simple and novel synthesis of mono- and bis(2,3-fused perhydrooxazol-4-one) systems

Sengodagounder Muthusamy* and Chidambaram Gunanathan

The unstable isomünchnones generated from the α -diazoimides have efficiently been trapped using the oxygen nucleophiles. This forms a facile entry into the new family of 2,3-fused perhydrooxazol-4-one and bis(2,3fused perhydrooxazol-4-one) systems.

Organoruthenium(II) and (III) amidinates, $(\eta^5 - C_5 Me_5)Ru(\eta$ -amidinate) and $(\eta^5 - C_5 Me_5) RuCl(\eta$ -amidinate), as unique redox catalysts for the intramolecular Kharasch reactions: Facile access to a pyrrolizidine alkaloid skeleton under mild conditions



 $(\eta^5-C_5Me_5)Ru(\eta-amidinate)$ or $(\eta^{5-}C_{5}Me_{5})RuCl(\eta-amidinate)$

benzene, r.t.



xviii

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Hideo Nagashima,* Mitsuru Gondo, Satoshi Masuda, Hideo Kondo, Yoshitaka Yamaguchi and Kouki Matsubara

Both of the Ru(II) and Ru(III) amidinate complexes are good catalysts for construction of a pyrrolizidine alkaloid skeleton under mild conditions.



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